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## PERMIT ATTACHMENT

### APPENDIX XX

#### SUBPART CC COMPLIANCE PLAN

This document was altered from the April 2018 permit application since it was missing pages. The Region copied the missing pages from the April 2012 application submittal.

September 2018

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APPENDIX XX

SUBPART CC COMPLIANCE PLAN

FOR

EVOQUA WATER TECHNOLOGIES

PARKER REACTIVATION FACILITY

PARKER, ARIZONA

Revision 6  
July 2014

# **Subpart CC Compliance Plan**

Hazardous Waste Treatment, Storage and Disposal Facilities  
and Hazardous Waste Generators; Organic Air Emission  
Standards for Tanks, Surface Impoundments and Containers

## **Evoqua Water Technologies**

**Parker, Arizona**

Revision 6  
July 2014

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# Evoqua Water Technologies

## Subpart CC Compliance Plan

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### 1. Introduction

This document summarizes the applicable air emission standards that apply to tanks, surface impoundments and containers used to manage hazardous waste relative to the Evoqua Water Technologies, Parker, Arizona facility under the U.S. Environmental Protection Agency (EPA) final Subpart CC regulations, and provides the plan to assure compliance with these standards. As discussed below, the Subpart CC regulations specifically exempt waste management operations performed in tanks and containers that comply with the National Emission Standards for Benzene Waste Operations promulgated by the EPA under the Section 112 of the Clean Air Act - National Emission Standards for Hazardous Air Pollutants (NESHAP), codified at 40 CFR Part 61, Subpart FF.

### 2. Facility Description

A detailed description of the facility operations is provided in Section D of the facility's Part B application dated April 2012.

### 3. Management Summary of Rule Requirements

Under Section 3004(n) of the authority of the Resource Conservation and Recovery Act (RCRA), the EPA has established standards to control air emissions from hazardous waste treatment, storage and disposal facilities as may be necessary to protect human health and the environment. Briefly, the EPA has established air emission standards for the following hazardous waste management units:

- **Process Vents** - referred to as Subpart AA regulations (codified at 40 CFR §264.1030, *et seq.* For permitted Treatment Storage and Disposal Facilities (TSDF) and 40 CFR §265.1030, *et seq.* for TSDFs allowed to manage hazardous waste under interim status.)
- **Equipment Leaks from Pumps, Valves and Compressors** - referred to as Subpart BB regulations (codified at 40 CFR §264.1050, *et seq.* or permitted Treatment Storage and Disposal Facilities (TSDF) and 40 CFR §265.1050, *et seq.* for TSDFs allowed to manage hazardous waste under interim status.

- **Tanks, Surface Impounds and Containers** - referred to as Subpart CC regulations (codified at 40 CFR §264.1080, *et seq.* or permitted Treatment Storage and Disposal Facilities (TSDF) and 40 CFR §265.1080, *et seq.* for TSDFs allowed to manage hazardous waste under interim status.)<sup>1</sup>

None of the waste management units at the facility are subject to Subpart AA or BB. Briefly, the facility is not subject to Subpart AA as there are no process vents associated with distillation, fractionation, thin-film evaporation, solvent extraction, or air or steam stripping (§265.130). Evoqua has conducted a waste determination and has determined that the organic loading of materials currently being managed at the facility is less than 10% by weight. Therefore the RCRA Subpart BB standards do not apply. Information regarding the waste determination is presented in Appendix XIX of the Part B Application. (§265.1050(b)). This compliance plan deals only with EPA Subpart CC standards that apply to tanks, surface impounds and containers that manage hazardous waste at the facility.

Relative to the Parker facility, the facility manages wastes in tanks and containers but not in surface impoundments. The tanks at the facility that are used for waste management are exempt from *all* Subpart CC emission control, monitoring, sampling, testing, reporting and record keeping requirements *provided* the facility certifies that these waste management units are equipped and operated with air emission controls in accordance with the Benzene Waste Operations NESHAP (40 CFR §61.340, *et seq.*). The final standards, as amended on November 25, 1996 provide in pertinent part that:

***(b) The requirements of this subpart [Subpart CC] do not apply to the following waste management units at the facility:***

\* \* \* \* \*

***(7) A hazardous waste management unit that the owner or operator certifies is equipped with and operating air emission controls in accordance with the requirements of an applicable Clean Air Act regulation codified under 40 CFR 60, 61 or part 63. For the purpose of complying with this paragraph, a tank for***

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<sup>1</sup> Subpart CC regulations have a lengthy regulatory history. Briefly, the final standards were originally promulgated on December 6, 1994 (59 FR 69826). The final rule caused much confusion and met with substantial opposition from the regulated community. The effective date of the rule was extended on three separate occasions (60 FR 26828, May 19, 1995; 60 FR 56952, November 13, 1995 and 61 FR 28508, June 5, 1996); EPA issued three subsequent final interpretive ruling to clarify Subpart CC requirements and to request additional public comment (60 FR 41870, August 14, 1995 and 61 FR 4903, February 9, 1996 and 61 FR 59932, November 25, 1996).

***which the air emission control includes an enclosure, as opposed to a cover, must be in compliance with the enclosure and control device requirements of §265.1085(l), except as provided in §265.1080(c)(5). [40 CFR §265.1080(b)(7)]***

A tank or container for which all hazardous waste entering the unit has an average volatile organic (VO) concentration at the point of waste origination of less than 500 parts per million by weight (ppmw) is subject to Subpart CC, but is exempt from air emissions control requirements ( §265.1082(c)). The average VO concentration is to be determined either by sampling and testing as directed by Subpart CC or by operator knowledge of the waste (§264.1084). If test data are used as the basis for knowledge, then the operator must document the test method, sampling protocol, and the means by which sampling and analytic variability are accounted for in determination of average VO concentration (§265.1084(b)(4)(ii)). Operators that rely on average VO concentration in a hazardous waste to exempt a unit from air emission controls must review and update, as necessary, this determination at least once every 12 months following the initial determination ((§265.1082(c)(1)).

For containers at the facility that are used for waste management, those that contain hazardous waste with an average VO concentration greater than 500 ppmw (and are not subject to the Benzene Waste NESHAP) must comply with prescribed air emission control requirements, testing, monitoring and reporting provisions of Subpart CC (§§265.1085-1088). Currently, only certain containers at the facility meet these prerequisites and are subject to air control requirements of Subpart CC. This plan applies to those containers as described below.

#### **4. Evoqua Water Technologies Subpart CC Compliance Plan**

The Subpart CC compliance plan for the Evoqua Water Technologies facility identifies three types of waste management units:

- Waste management units (containers) that are regulated per Subpart CC requirements:
- Waste management units that are exempt from Subpart CC requirements because they are otherwise regulated under the Benzene Waste Operation NESHAP; and
- Waste management units that have a volatile organic (VO) concentration less than 500 ppmw, and are therefore exempt from the Subpart CC air emissions control requirements (( §§265.1085-1087). Record keeping and monitoring requirements under Subpart CC apply to these exempt units §265.1082(c)(1 and 1090(f)).

Compliance requirements for each of these categories of waste management units is

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discussed below.

## **4.1 Subpart CC Applicability**

### **4.1.1 Tanks Standards**

Under the final Subpart CC regulations, tanks that are equipped with and comply with the Benzene Waste Operations NESHAP (Subpart FF) are exempt from all Subpart CC requirements (see 40 CFR §265.1080(b)(7)). Therefore, the facility will demonstrate compliance with Subpart CC regulations by assuring that all tanks used to manage hazardous waste are equipped with appropriate air emission controls as required by, and operate in compliance with, Subpart FF.

### **4.1.2 Container Standards 40 CFR 264.1086 (c )**

For containers that are not regulated under Subpart FF, a variety of container types are used to store and manage spent carbon at the Facility. All of these containers, including bulk bags, drums, various types of spent carbon adsorption vessels, and other portable vessels, must meet the performance standards for containers in 40 CFR §264.1086 when managing Subpart CC regulated hazardous waste. This section applies only to those containers not regulated under Subpart FF.

#### **1. Container Requirements for Subpart CC**

- A. Containers with a design capacity less than or equal to 26.4 gallons are exempt from Subpart CC requirements. 40 CFR §264.1080(b)(2).
- B. Containers with a design capacity greater than 26.4 gallons and no more than 119 gallons are subject to Level 1 controls.
- C. Containers with a design capacity of greater than 119 gallons that are not in light material service are subject to Level 1 controls.
- D. Containers with a design capacity of greater than 119 gallons that are in light material service are subject to Level 2 controls.
- E. Containers with a design capacity of greater than 26.4 gallons that are used for treatment of a hazardous waste by a waste stabilization process are subject to Level 3 controls. The Facility does not treat hazardous waste in containers using a waste stabilization process and therefore Level 3 controls are not further discussed.



*In light material service* is defined as managing a material for which both of the following conditions apply: (i) the vapor pressure of one or more of the organic constituents in the material is greater than 0.3 kPa at 20° C; and (ii) the total concentration of the pure organic constituents having a vapor pressure great than 0.3 kPa at 20° C is equal to or greater than 20 percent by weight. 40 CFR §265.1081.

#### **4.1.2.1 Containers - Level 1 Controls.**

For containers subject to Level 1 controls, the Facility complies with Subpart CC as follows:

- A. All Level 1 containers are subject to the following requirements:
  - (i) Level 1 containers must be compliant with US DOT hazardous material packaging requirements - *i.e.*, these containers meet the requirements of 49 CFR Parts 178 and 179, and waste is managed in accordance with 49 CFR Parts 107 (subpart B), 172, 173 and 180 (no exceptions to 178 or 179 are allowed for this purpose) (see 40 CFR §264.1086(f)); or
  - (ii) Level 1 containers must be equipped with a cover and closure devices that form a continuous barrier such that there are no visible holes, gaps or other open spaces.
- B. All containers are inspected upon receipt (on or before the date of acceptance at the Facility), and repairs are conducted where defects are observed, as follows:
  - (i) visual inspections are conducted to ensure the containers are equipped with a cover and closure devices that form a continuous barrier over the container openings such that when the cover and closure devices are secured in a closed position there are no visible cracks, holes, gaps or other open spaces into the interior of the containers. 40 CFR §264.1086(c)(1)(ii) and 264.1086(c)(4)(i); and
  - (ii) visual inspections also confirm that the containers meet the applicable US DOT requirements on packaging hazardous materials for transportation in 49 CFR Parts 107, 172, 173, 178, and 180. 40 CFR §264.1086(d)(i) and 1086(f) see 40 CFR §264.1086(c)(1)(i)); and
  - (iii) where defects in containers are detected, the Facility makes first

attempts to repair no later than 24 hours from detection and completes repair with as soon as possible and in any event within 5 calendar days, or alternatively transfers the waste to an intact container or tank.

- C. Where containers are initially placed in service at the Facility, the inspection occurs when the cover is applied to the container. If containers were to remain in use at the Facility for a period of one year or more, the Facility would conduct a visual inspection at least once every 12 months as discussed in item A above. (Since storage of hazardous waste is currently prohibited for more than one year, these inspections are not anticipated.) See 40 CFR §264.1086(c)(4)(ii).
- D. The Facility's operating practice is to only open containers subject to Level 1 controls for the following reasons:
  - (i) to remove wastes in a continuous process until the container is RCRA empty (40 CFR §261.7(b)); or to remove waste in batches, in which case containers are closed upon completion of the batch removal for 15 minutes or if the operator leaves the immediate vicinity;
  - (ii) to perform routine activities other than waste transfer, provided that the containers are promptly closed; and
  - (iii) to open a safety device to avoid an unsafe condition.

#### **4.1.2.2 Containers - Level 2 Controls**

For containers subject to Level 2 controls, the Facility complies with Subpart CC as follows:

- A. All Level 2 containers are subject to the following requirements:
  - (i) Level 2 containers must be compliant with US DOT hazardous material packaging requirements - *i.e.*, containers meet the requirements of 49 CFR Parts 178 and 179, and waste is managed in accordance with 49 CFR Parts 107 (subpart B), 172, 173 and 180 (no exceptions to 178 or 179 are allowed for this purpose) (see 40 CFR §264.1086(f)); or
  - (ii) Level 2 containers must be tested upon receipt to confirm that they operate with no detectable organic emissions as determined through Method 21.

- B. All containers are inspected upon receipt (on or before the date of acceptance at the Facility), and repairs are conducted where defects are observed, as follows:
- (i) visual inspections confirm that the containers are equipped with a cover and closure devices that form a continuous barrier over the container openings such that when the cover and closure devices are secured in a closed position there are no visible cracks, holes, gaps or other open spaces into the interior of the containers. 40 CFR §264.1086(d)(4)(i);
  - (ii) visual inspections also confirm that the containers meet the applicable US DOT requirements on packaging hazardous materials for transportation in 49 CFR Parts 107, 172, 173, 178, and 180. 40 CFR §264.1086(d)(i) and 1086(f); and
  - (iii) where defects in containers are detected, the Facility makes first attempts to repair no later than 24 hours from detection and completes repair with as soon as possible and in any event within 5 calendar days, or alternatively transfers the waste to an intact container or tank. 40 CFR §264.1086(d)(4)(iii).
- C. Where a container is initially placed in service at the Facility, the inspection occurs when the cover is applied to the container. 61 Fed. Reg. 59947. If containers remain in use at the Facility for a period of one year or more, the Facility conducts a visual inspection at least once every 12 months as discussed in item B above. (Since storage of hazardous waste is currently prohibited for more than one year, these annual inspections are not anticipated.) 40 CFR §264.1086(d)(4)(ii).[
- D. All transfers of hazardous waste subject to Subpart CC out of a container is conducted to minimize exposure of the waste to the atmosphere, to the extent practical, considering the physical properties of the waste and good engineering and safety practices for handling the wastes.

#### **4.1.2.3 Container Recordkeeping**

Level 1 and Level 2 containers are subject to very limited recordkeeping requirements under Subpart CC. See 61 Fed. Reg. 59947 (Nov. 25, 1996). The facility's Waste Tally sheet is used to document that the containers meet USDOT and visual inspection requirements.

Listed below, in Table 1, are the waste management units at the facility that are potentially

subject to Subpart CC requirements, together with the applicable Benzene NESHAP Subpart FF requirement:

**Table 1**

<b>I.D. NO.</b>	<b>DESCRIPTION</b>	<b>APPLICABLE SUBPART FF STANDARD (40 CFR §)</b>	<b>COMMENTS</b>
<b>N/A</b>	Spent Carbon Containers Subject to Subpart FF	§61.345	Subpart FF wastes are stored in drums and other containers. All RCRA drums and containers received at the facility that are also regulated under Subpart FF are managed as Subpart FF-affected wastes.
<b>N/A</b>	Debris Bin and Associated Drums.	§61.345 §61.34(f)	All RCRA waste drums at the facility are managed as Subpart FF-affected wastes. Benzene wastes shipped offsite must meet container and offsite shipment requirements.
<b>H-1 H-2</b>	Spent carbon unloading hoppers, Nos. 1 and 2.	§61.348	Both hoppers H-1 and H-2 receive spent carbon from containers and are managed as Subpart FF-affected units. Emissions from these hoppers are directed to WS-2.
<b>T-1</b>	Spent Carbon Storage Tank	§61.343	Tank T-1 is managed as a Subpart FF-affected unit. Tank vapors are controlled by carbon adsorber (WS-1).
<b>I.D. NO.</b>	<b>DESCRIPTION</b>	<b>APPLICABLE SUBPART FF STANDARD (40 CFR §)</b>	<b>COMMENTS</b>
	Spent Carbon Storage		Tank T-2 is managed as a Subpart

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<b>T-2</b>	Tank	§61.343	FF-affected unit. Tank vapors are controlled by carbon adsorber (WS-1).
<b>T-5</b>	Spent Carbon Storage Tank	§61.343	Tank T-5 is managed as a Subpart FF-affected unit. Tank vapors are controlled by carbon adsorber (WS-1).
<b>T-6</b>	Spent Carbon Storage Tank	§61.343	Tank T-6 is managed as a Subpart FF-affected unit. Tank vapors are controlled by carbon adsorber (WS-1).
<b>T-11</b>	Scrubber/Recycle/Boiler and Cooling Tower Blow- Down Water Storage Tank	§61.342(c)	Exempt from treatment since benzene concentration is less than 10 ppmw.
<b>T-19</b>	Packed Bed Scrubber Recirculation Tank	§61.342(c)	Exempt from treatment since benzene concentration is less than 10 ppmw.
<b>RF-2 AB-2</b>	Reactivation Furnace No.2 and Afterburner No. 2	§61.348	Regenerated carbon must contain less than 10 ppmw benzene and the unit must meet 99+% benzene destruction efficiency.
<b>WS-1</b>	Carbon Adsorber No.1	§61.349	Carbon canister, used to control volatile emissions from Tanks T-1, T-2, T-5, T-6, and T-9.
<b>WS-2</b>	Carbon Adsorber No.2	§61.349	Carbon canister, used to control volatile emissions from Hoppers H-1 and H-2.
<b>WS-3</b>	Carbon Adsorber No.3	§61.349	Carbon canister, used to control volatile emissions from Tank T-18 (T-18 is not a RCRA-regulated tank).

Listed below, in Table 2, are the waste management units at the facility that are subject to Subpart CC requirements as they are not regulated under Subpart FF.

**Table 2**

I.D. NO.	DESCRIPTION	APPLICABLE SUBPART CC STANDARD (40 CFR §)	COMMENTS
N/A	Spent Carbon Containers	40 CFR 264.1086 (c)	All non-FF RCRA containers received at the facility are managed in accordance with the applicable portions of the US DOT hazardous material packaging requirements - <i>i.e.</i> , for containers, 49 CFR Parts 178 and for waste, 49 CFR Parts 107 (subpart B), 172, 173 and 180 (no exceptions to 178 or 179 are allowed for this purpose) (see 40 CFR §264.1086(f)). Each container is equipped with a cover and/or closure devices that form a continuous barrier such that there are no visible holes, gaps or other open spaces.

As summarized above, all process units, debris bins and waste management containers at the facility are subject to the Benzene Waste NESHAPs with the exception Tanks T-19 and T-11. Tank T-19 recirculates water to the packed bed scrubber and is the introduction point for makeup water added to the scrubber system. City water is used for makeup. Tank T-11 collects scrubber water blow down, cooling water blow down, boiler blow down and excess recycle water. T-11 is therefore potentially subject to regulation under Subpart CC, as it is not regulated under the Benzene Waste Operations NESHAP.

#### 4.2 Waste Management Units Exempt From Subpart CC Control Requirements

As summarized in Section 4.1, Tank T-11 is subject to Subpart CC because it is not is not regulated under the Benzene Waste Operations NESHAP, and not otherwise exempt under §265.1080. As demonstrated below, Tank T-11 is not subject to the Subpart CC air emission control requirements because the average VO concentration in the waste entering the unit is less than 500 ppmw (§264.1082(c)(1)). Tank T-11 is, however, subject to

monitoring and record keeping requirements (§§265.1082(c)(1) and 1090(f)(1)), which are discussed below.

### **4.3 VO Concentration Determination Procedures**

#### **4.3.1 Initial VO Concentration Determination**

Operator knowledge provides the basis to conclude that the average VO concentration of hazardous waste entering Tank T-11 is less than 500 ppmw from T-9. The following test data from sampling previously conducted confirm that the average VO concentration of waste entering Tank T-11 is less than 500 ppmw:

- On November 30, 1994, the facility sampled the recycle water that drains from Tank T-12 to Tank T-11. Samples were collected from the process line that connects these two tanks. This sampling point was selected to assure that the sample will be representative of the VO concentration at the point of generation. A sample cannot be obtained directly from T-11, as the tank also receives scrubber water blow down, boiler blow down and cooling tower blow down. Further, obtaining a sample from the process line assures that there will be no gravitational or phase separation of VO constituents, which may bias the sample.
- On February 22, 1994, December 19, 1994 and October 12, 1995, the facility sampled the water discharged from Tank T-11 prior to discharge, under permit, to the sanitary sewer. This sampling point was selected as representative of the average VO concentration at the point of discharge.

The sample collection and handling procedures were in accordance with EPA Publication No. SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", as amended by Update I, November 15, 1992. Briefly, screw cap VOA vials with Teflon lined silicone septa were used to collect the sample. Care was taken to assure that no air bubbles were entrained in the vial prior to closure. Samples were preserved as required in the applicable methods, stored at 4° C, and analyzed within holding times. Concentration of VO constituents were determined by EPA Methods 8260 (volatile organics), 8270 (semi-volatile organics), 8080 (organochlorine pesticides and PCBs) and 8015 (GC/FID Alcohol Screen).

Total VO concentration reported in each of these four sampling events are summarized in Table 2, below:

**Table 3**

#### **Summary of VO Concentration Reported in Prior Sampling of Tank T-11**

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Sampling Event	Reported Total VO Concentration
November 30, 1994	519 µg/l
February 22, 1994	Below Detection Limits
December 19, 1994	22 µg/l
October 12, 1995	23 µg/l

#### 4.3.2 Future VO Concentration Determinations

The facility must review, and as necessary, update this VO determination every 12 months (§265.1082(c)(1)). The annual confirmatory analyses of the annual samples can be found in Appendix B. If the Facility staff determines that the initial VO concentration determination defined in Section 4.2.1 is no longer valid, additional waste determination sampling must be performed. Such sampling must comply with requirements outlined in (§§265.1084(a)(2) and (3), which are briefly summarized below:

- Must identify and record the point of origination for the hazardous waste (§265.1084(a)(3)(I)).
- Sampling must be performed pursuant to a sampling plan, and must meet the following requirements:
  - Identification of where in the process the samples are to be taken.
  - The appropriate averaging period to be used to determine the average VO concentration in the sample. The averaging period cannot exceed one (1) year. Record the date, time and location that each sample is collected and maintain these data in the Subpart CC sampling plan.
  - The sample collection method used to minimize volatilization of organic compounds contained in the sample. At least four (4) samples are required to calculate the average volatile organic concentration.
  - The analytic methods used to determine concentrations of volatile organic compounds. Acceptable analytic methods include: Method 25D, Methods 8260(latest version) and 8270(latest version) as defined in SW-846, Methods 624, 625, 1624 and 1625 as defined in 40 CFR Part 136. If any method aside from Method 25D is used, the facility must demonstrate that all target



compounds in the sample are included amount those compounds listed by the EPA as ones for which the method is considered appropriate. If target compounds are not on this list, additional requirements will apply to the analytic methods (see §265.1084(a)(3)(iii)). The sampling plan must include a quality assurance plan to document the specific procedures used to minimize loss of VO compounds due to volatilization, reaction, biodegradation, or sorption during the sample collection, storage, and preparation steps, and a measurement of the overall accuracy and precision of the specific procedures. Further, if analytic methods other than Method 25D are used, the facility may exclude those organics with a Henry's Law constant values less than 0.1 mole-fraction-in-the-gas-phase/mole-fraction-in-the-liquid-phase. A list of all such compounds is included in Appendix VI to the final Subpart CC regulations.

Sampling locations are shown in Figure 1. A sampling plan is provided in Appendix C. EPA reference analytical methods are shown in Appendix D. Laboratory SOPs for the analytical methods are presented in Appendix E.

#### **4.4 Monitoring Requirements**

Provided the VO concentration of liquids contained in Tank T-11 remains below 500 ppmw, the facility must perform the following monitoring of its operations:

- Assure that the facility complies with all applicable requirements as defined in the Benzene Waste Operations NESHAP - Subpart FF.
- Review the waste determination for Tank T-11 on an annual basis, no later than December 4 of each calendar year (see §265.1083(c)(1)).

This section must be updated should either the exemption status of T-11 change or if the applicability determinations for Subpart FF are modified.

#### **4.5 Record Keeping Requirements**

The Facility must maintain the following records as part of the Subpart CC Plan:

- Maintain the sampling data attached in Appendix B ((see §265.1090(f)(1)).

This section must be updated should either the exemption status of T-11 change or if the applicability determinations for Subpart FF be modified.

#### **4.6 Reporting Requirements**

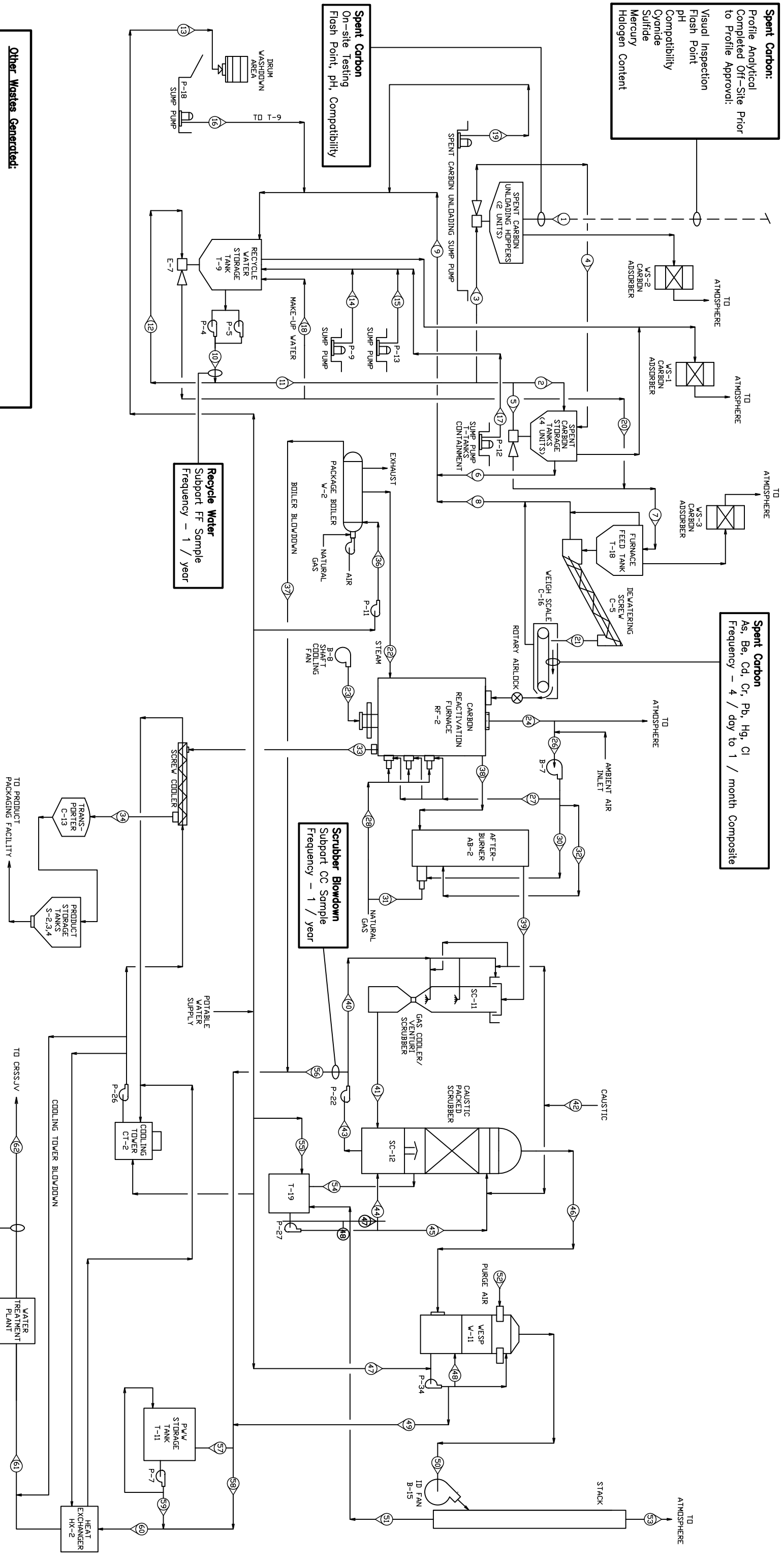
Under the final regulations promulgated on November 25, 1996, Subpart CC applicability was amended to exempt any hazardous waste management unit that the facility certifies is equipped with and operating air emission controls in accordance with the Benzene Waste Operations NESHAP (Subpart FF). The notification and reporting provisions included in the final Subpart CC regulations do not specifically require that the facility send such a certification to the U.S. EPA (see §265.1090). However, to assure compliance with this revised applicability standard, the facility has made this certification in letter to the U.S. EPA, Region IX. A copy of this letter is attached at Appendix A.

Under 40 CFR Part 270, facilities that manage hazardous waste under interim status must amend their Part B permit application to define the inspection and control systems employed at the facility to comply with Subpart CC requirements. As discussed above, no such inspection or control requirements apply to the facility, as all waste units are either exempt under §265.1080(b)(7), or are exempt from control requirements under §265.1083(c). Thus there is no requirement to modify the Part B permit application for the facility. The letter certifying compliance with Subpart CC advises EPA Region IX that no such modification to the Part B permit application is required for the facility.

**Spent Carbon:**  
 Profile Analytical Completed Off-Site Prior to Profile Approval:  
 Visual Inspection  
 Flash Point  
 pH  
 Compatibility  
 Cyanide  
 Sulfide  
 Mercury  
 Halogen Content

**Spent Carbon**  
 On-site Testing  
 Flash Point, pH, Compatibility

**Spent Carbon**  
 As, Be, Cd, Cr, Pb, Hg, Cl  
 Frequency - 4 / day to 1 / month Composite



**Other Wastes Generated:**

- Misc. Debris - Weekly - All Waste Codes Facility has received. Not Currently Tested Put in 90 day bin for incineration
- WWTP Filter Press Cake - All Waste Codes Facility has received. Not Currently Tested Put in 90 day bin for incineration

**NOTES:**

- THIS DRAWING INCLUDES COMPONENTS OF THE FACILITY THAT ARE EXCEPT FROM PERMITTING UNDER VARIOUS PROVISIONS OF RCRA. DATA RELATED TO THESE COMPONENTS IS PROVIDED FOR INFORMATIONAL PURPOSES AND EASE OF REVIEW ONLY, AND THEY ARE NOT INTENDED TO BECOME REGULATED COMPONENTS OF THE HAZARDOUS WASTE FACILITY.

**Wastewater Discharge**  
 POTW TSS Sample  
 Frequency - 2 / month

**Wastewater Discharge**  
 CWT Sample  
 Frequency - 2 / year

**Wastewater Discharge**  
 POTW TTO Sample  
 Frequency - 1 / year

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NO.	DATE	APP.	REVISIONS	DATE
1	7/6/11	JBE	DATE	7/7/11
2	7/7/11	KEM	DATE	

**CBE CHAVOND-BARRY ENGINEERING CORP.**  
 400 Route 518 • P.O. Box 205 • Bloomerburg, New Jersey 08504

**SIEMENS INDUSTRY, INC.**  
 2523 MITAHAR STREET, PARKER, AZ 85344

**SAMPLE LOCATIONS**

DRAWN	CHECKED	APPROVED
JBE	7/6/11	KEM
SCALE	DWG. NO.	WAP-001
NDNF		REV. 0

# **APPENDIX A**

December 3, 1996 Letter/Certification

VIA FEDERAL EXPRESS

December 3, 1996

Mr. John McCarroll  
Permits Section Chief  
Hazardous Waste Management Division  
Mail Code WST-4 - 10th Floor  
U.S. Environmental Protection Agency  
75 Hawthorne Street  
San Francisco, CA 94105

**FILE COPY**

**RE: WESTATES CARBON-ARIZONA, INC.  
EPA No. AZD 982 441 263  
CERTIFICATION OF COMPLIANCE - SUBPART CC**

Dear Mr. Fox:

Westates Carbon-Arizona, Inc. (WCAI) operates a carbon reactivation facility in Parker, Arizona. A detailed description of the facility is provided in the Part B application dated November 1995. As discussed below, all waste management units at WCAI are either exempt from Subpart CC under §265.1080(b)(7), or exempt from the control requirements of Subpart CC under §265.1083(c). Therefore, based on our review of 40 CFR Part 270 - EPA Administered Permit Programs: The Hazardous Waste Management Program, there is no need to modify the Part B permit application for the WCAI facility to comply with the requirements of Subpart CC.

On November 25, 1996 the United States Environmental Protection Agency modified the air emission control standards that apply to certain waste management units, known colloquially as the Subpart CC standards (see 61 FR 59932). In part, the Subpart CC applicability was amended to exempt any hazardous waste management unit that the owner or operator certifies is equipped with and operating air emission controls in accordance with an applicable Clean Air Act regulation, including the Benzene Waste Operations National Emission Standard for Hazardous Air Pollutants (40 CFR Part 61, Subpart FF).

Pursuant to §265.1080(b)(7), WCAI hereby certifies that all waste management units potentially subject to Subpart CC, with the exception of Tank T-11, are equipped with and operate air emission controls in accordance with Subpart FF. EPA Region IX previously approved these air emission control systems as meeting the requirements of Subpart FF in its approval of an application to modify the facility under 40 CFR §61.07. The approval was granted by EPA Region IX on August 4, 1995 pursuant to 40 CFR §61.08 of the NESHAPs program.

As described in the Part B permit application, Tank T-11 collects scrubber water blow down, cooling water blow down, boiler water blow down and recycle water from Tank T-12, that has been filtered through activated carbon. Tank T-11 is not subject to the Benzene Waste NESHAPs because the benzene concentration in the water is less than 10 ppmw. Based on operator knowledge, Tank T-11 is exempt from the air emission control and inspection requirements pursuant to 40 CFR §265.1083(c)(1) because the average VO concentration of the wastes entering the unit is less than 500 ppmw (§264.1082(c)(1)). WCAI, will however, comply with the Subpart CC record keeping and monitoring requirements that are applicable to Tank T-11.

Please call me at (520) 669-5758 should you have any questions regarding the foregoing.

Sincerely,



Monte McCue  
Plant Manager

cc: William Carlson (WESI)  
Ray Fox (USEPA Region IX) (via fax)  
Matt Killeen (WESI)  
Steve Richmond (WESI)

State of ARIZONA

County of LA PAZ

On this 3 day of Dec, 1996, M. McCue personally appeared before me,

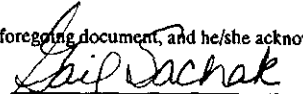
whose identity I verified on the basis of \_\_\_\_\_,

who is personally known to me,

whose identity I verified on the oath/affirmation of \_\_\_\_\_,

a credible witness,

to be the signer of the foregoing document, and he/she acknowledge that he/she signed it.

  
Notary Public  
My Commission expires: 2/22/99



# **APPENDIX B**

## Confirmatory Sample Analysis

## LABORATORY REPORT

Prepared For: Siemens Water Technologies  
P.O. Box 3308 (2523 Mutahar St.)  
Parker, AZ 85344  
Attention: Todd Guimond

Project: Subpart CC

Sampled: 12/04/09  
Received: 12/05/09  
Issued: 12/16/09 10:34

NELAP #01108CA California ELAP#2706 CSDLAC #10256 AZ #AZ0671 NV #CA01531

*The results listed within this Laboratory Report pertain only to the samples tested in the laboratory. The analyses contained in this report were performed in accordance with the applicable certifications as noted. All soil samples are reported on a wet weight basis unless otherwise noted in the report. This Laboratory Report is confidential and is intended for the sole use of TestAmerica and its client. This report shall not be reproduced, except in full, without written permission from TestAmerica.  
This entire report was reviewed and approved for release.*

## SAMPLE CROSS REFERENCE

SUBCONTRACTED: Refer to the last page for specific subcontract laboratory information included in this report.

LABORATORY ID	CLIENT ID	MATRIX
ISL0598-01	Subpart CC (A)	Water
ISL0598-02	Subpart CC (B)	Water
ISL0598-03	Subpart CC (C)	Water
ISL0598-04	Subpart CC (D)	Water

Reviewed By:



**TestAmerica Irvine**

Lena Davidkova  
Project Manager



Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromochloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromodichloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromoform	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromomethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
n-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
sec-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
tert-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Carbon tetrachloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chloroform	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
2-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
4-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromo-3-chloropropane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Dibromochloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromoethane (EDB)	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dibromomethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dichlorodifluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
cis-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
trans-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
2,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
cis-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	L, M7
trans-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Ethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Hexachlorobutadiene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Isopropylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
p-Isopropyltoluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Methylene chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Naphthalene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Styrene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	M2
1,1,1,2-Tetrachloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,2,2-Tetrachloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Tetrachloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Toluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,1-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,2-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichlorofluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichloropropane	EPA 8260B	9L11040	10	ND	1	12/11/2009	12/11/2009	
1,2,4-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3,5-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Vinyl chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
m,p-Xylenes	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
o-Xylene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Surrogate: 4-Bromofluorobenzene (80-120%)				93 %				
Surrogate: Dibromofluoromethane (80-120%)				85 %				
Surrogate: Toluene-d8 (80-120%)				101 %				

TestAmerica Irvine

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromochloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromodichloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromoform	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromomethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
n-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
sec-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
tert-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Carbon tetrachloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chloroform	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
2-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
4-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromo-3-chloropropane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Dibromochloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromoethane (EDB)	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dibromomethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dichlorodifluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
cis-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
trans-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
2,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
cis-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	L
trans-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Ethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Hexachlorobutadiene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Isopropylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
p-Isopropyltoluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Methylene chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Naphthalene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Styrene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,1,2-Tetrachloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,2,2-Tetrachloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Tetrachloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Toluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,1-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,2-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichlorofluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichloropropane	EPA 8260B	9L11040	10	ND	1	12/11/2009	12/11/2009	
1,2,4-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3,5-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Vinyl chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
m,p-Xylenes	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
o-Xylene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Surrogate: 4-Bromofluorobenzene (80-120%)				94 %				
Surrogate: Dibromofluoromethane (80-120%)				84 %				
Surrogate: Toluene-d8 (80-120%)				101 %				

TestAmerica Irvine

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromochloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromodichloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromoform	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromomethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
n-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
sec-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
tert-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Carbon tetrachloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chloroform	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
2-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
4-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromo-3-chloropropane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Dibromochloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromoethane (EDB)	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dibromomethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dichlorodifluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
cis-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
trans-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
2,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
cis-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	L
trans-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Ethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Hexachlorobutadiene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Isopropylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
p-Isopropyltoluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Methylene chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Naphthalene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	

**TestAmerica Irvine**

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Styrene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,1,2-Tetrachloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,2,2-Tetrachloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Tetrachloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Toluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,1-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,2-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichlorofluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichloropropane	EPA 8260B	9L11040	10	ND	1	12/11/2009	12/11/2009	
1,2,4-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3,5-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Vinyl chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
m,p-Xylenes	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
o-Xylene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Surrogate: 4-Bromofluorobenzene (80-120%)				94 %				
Surrogate: Dibromofluoromethane (80-120%)				86 %				
Surrogate: Toluene-d8 (80-120%)				101 %				

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Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromochloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromodichloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Bromoform	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Bromomethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
n-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
sec-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
tert-Butylbenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Carbon tetrachloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Chloroform	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Chloromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
2-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
4-Chlorotoluene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromo-3-chloropropane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Dibromochloromethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dibromoethane (EDB)	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dibromomethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Dichlorodifluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloroethene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
cis-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
trans-1,2-Dichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
2,2-Dichloropropane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
cis-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	L
trans-1,3-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1-Dichloropropene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Ethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Hexachlorobutadiene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Isopropylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
p-Isopropyltoluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Methylene chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
Naphthalene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	

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Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Styrene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,1,2-Tetrachloroethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,2,2-Tetrachloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Tetrachloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Toluene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,1,1-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,1,2-Trichloroethane	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichloroethene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Trichlorofluoromethane	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
1,2,3-Trichloropropane	EPA 8260B	9L11040	10	ND	1	12/11/2009	12/11/2009	
1,2,4-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
1,3,5-Trimethylbenzene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Vinyl chloride	EPA 8260B	9L11040	5.0	ND	1	12/11/2009	12/11/2009	
m,p-Xylenes	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
o-Xylene	EPA 8260B	9L11040	2.0	ND	1	12/11/2009	12/11/2009	
Surrogate: 4-Bromofluorobenzene (80-120%)				94 %				
Surrogate: Dibromofluoromethane (80-120%)				88 %				
Surrogate: Toluene-d8 (80-120%)				101 %				

TestAmerica Irvine

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 Project Manager



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Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Acenaphthylene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Aniline	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	C-2
Anthracene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzidine	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Benzo(a)anthracene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(a)pyrene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(b)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(g,h,i)perylene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(k)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzoic acid	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Benzyl alcohol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Bromophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Butyl benzyl phthalate	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Chloro-3-methylphenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Chloroaniline	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-chloroethoxy)methane	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-chloroethyl)ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-chloroisopropyl)ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-ethylhexyl)phthalate	EPA 8270C	9L08085	48	ND	0.966	12/8/2009	12/11/2009	
2-Chloronaphthalene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Chlorophenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4-Chlorophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Chrysene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Dibenz(a,h)anthracene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Dibenzofuran	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Di-n-butyl phthalate	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
3,3'-Dichlorobenzidine	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4-Dichlorophenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Diethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2,4-Dimethylphenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Dimethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4,6-Dinitro-2-methylphenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4-Dinitrophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2,6-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Di-n-octyl phthalate	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	

**TestAmerica Irvine**

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Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Fluorene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Hexachlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Hexachlorobutadiene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Hexachlorocyclopentadiene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	C, L
Hexachloroethane	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Indeno(1,2,3-cd)pyrene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Isophorone	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Methylnaphthalene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Naphthalene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Nitroaniline	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
3-Nitroaniline	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Nitroaniline	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Nitrobenzene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2-Nitrophenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4-Nitrophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
N-Nitroso-di-n-propylamine	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
N-Nitrosodiphenylamine	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Pentachlorophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Phenanthrene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Phenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Pyrene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2,4,5-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4,6-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Surrogate: 2,4,6-Tribromophenol (40-120%)								88 %
Surrogate: 2-Fluorobiphenyl (50-120%)								73 %
Surrogate: 2-Fluorophenol (30-120%)								58 %
Surrogate: Nitrobenzene-d5 (45-120%)								73 %
Surrogate: Phenol-d6 (35-120%)								58 %
Surrogate: Terphenyl-d14 (50-125%)								87 %

TestAmerica Irvine

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Acenaphthylene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Aniline	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	C-2
Anthracene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzidine	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Benzo(a)anthracene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(a)pyrene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(b)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(g,h,i)perylene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(k)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzoic acid	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Benzyl alcohol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Bromophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Butyl benzyl phthalate	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Chloro-3-methylphenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Chloroaniline	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-chloroethoxy)methane	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-chloroethyl)ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-chloroisopropyl)ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-ethylhexyl)phthalate	EPA 8270C	9L08085	49	ND	0.971	12/8/2009	12/11/2009	
2-Chloronaphthalene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Chlorophenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4-Chlorophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Chrysene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Dibenz(a,h)anthracene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Dibenzofuran	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Di-n-butyl phthalate	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
3,3'-Dichlorobenzidine	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4-Dichlorophenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Diethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2,4-Dimethylphenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Dimethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4,6-Dinitro-2-methylphenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4-Dinitrophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2,6-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Di-n-octyl phthalate	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Fluorene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Hexachlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Hexachlorobutadiene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Hexachlorocyclopentadiene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	C, L
Hexachloroethane	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Indeno(1,2,3-cd)pyrene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Isophorone	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Methylnaphthalene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Naphthalene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Nitroaniline	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
3-Nitroaniline	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Nitroaniline	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Nitrobenzene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2-Nitrophenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4-Nitrophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
N-Nitroso-di-n-propylamine	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
N-Nitrosodiphenylamine	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Pentachlorophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Phenanthrene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Phenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Pyrene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2,4,5-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4,6-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Surrogate: 2,4,6-Tribromophenol (40-120%)								81 %
Surrogate: 2-Fluorobiphenyl (50-120%)								70 %
Surrogate: 2-Fluorophenol (30-120%)								56 %
Surrogate: Nitrobenzene-d5 (45-120%)								70 %
Surrogate: Phenol-d6 (35-120%)								55 %
Surrogate: Terphenyl-d14 (50-125%)								82 %

TestAmerica Irvine

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
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 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Acenaphthylene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Aniline	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	C-2
Anthracene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzidine	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Benzo(a)anthracene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(a)pyrene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(b)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(g,h,i)perylene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzo(k)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Benzoic acid	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Benzyl alcohol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Bromophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Butyl benzyl phthalate	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Chloro-3-methylphenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Chloroaniline	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-chloroethoxy)methane	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-chloroethyl)ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-chloroisopropyl)ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Bis(2-ethylhexyl)phthalate	EPA 8270C	9L08085	49	ND	0.971	12/8/2009	12/11/2009	
2-Chloronaphthalene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Chlorophenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4-Chlorophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Chrysene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Dibenz(a,h)anthracene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Dibenzofuran	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Di-n-butyl phthalate	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
3,3'-Dichlorobenzidine	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4-Dichlorophenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Diethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2,4-Dimethylphenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Dimethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4,6-Dinitro-2-methylphenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4-Dinitrophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2,6-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Di-n-octyl phthalate	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Fluorene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Hexachlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Hexachlorobutadiene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Hexachlorocyclopentadiene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	C, L
Hexachloroethane	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Indeno(1,2,3-cd)pyrene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Isophorone	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Methylnaphthalene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Naphthalene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2-Nitroaniline	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
3-Nitroaniline	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
4-Nitroaniline	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Nitrobenzene	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2-Nitrophenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
4-Nitrophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
N-Nitroso-di-n-propylamine	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
N-Nitrosodiphenylamine	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Pentachlorophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Phenanthrene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Phenol	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
Pyrene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.971	12/8/2009	12/11/2009	
2,4,5-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
2,4,6-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.971	12/8/2009	12/11/2009	
Surrogate: 2,4,6-Tribromophenol (40-120%)								76 %
Surrogate: 2-Fluorobiphenyl (50-120%)								73 %
Surrogate: 2-Fluorophenol (30-120%)								56 %
Surrogate: Nitrobenzene-d5 (45-120%)								74 %
Surrogate: Phenol-d6 (35-120%)								56 %
Surrogate: Terphenyl-d14 (50-125%)								91 %

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager

The results pertain only to the samples tested in the laboratory. This report shall not be reproduced, except in full, without written permission from TestAmerica.

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Acenaphthylene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Aniline	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	C-2
Anthracene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzidine	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Benzo(a)anthracene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(a)pyrene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(b)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(g,h,i)perylene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzo(k)fluoranthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Benzoic acid	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Benzyl alcohol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Bromophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Butyl benzyl phthalate	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Chloro-3-methylphenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Chloroaniline	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-chloroethoxy)methane	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-chloroethyl)ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-chloroisopropyl)ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Bis(2-ethylhexyl)phthalate	EPA 8270C	9L08085	48	ND	0.966	12/8/2009	12/11/2009	
2-Chloronaphthalene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Chlorophenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4-Chlorophenyl phenyl ether	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Chrysene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Dibenz(a,h)anthracene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Dibenzofuran	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Di-n-butyl phthalate	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
1,2-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
1,3-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
1,4-Dichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
3,3'-Dichlorobenzidine	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4-Dichlorophenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Diethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2,4-Dimethylphenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Dimethyl phthalate	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4,6-Dinitro-2-methylphenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4-Dinitrophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2,6-Dinitrotoluene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Di-n-octyl phthalate	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	

### TestAmerica Irvine

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Fluorene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Hexachlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Hexachlorobutadiene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Hexachlorocyclopentadiene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	C, L
Hexachloroethane	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Indeno(1,2,3-cd)pyrene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Isophorone	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Methylnaphthalene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4-Methylphenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Naphthalene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2-Nitroaniline	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
3-Nitroaniline	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
4-Nitroaniline	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Nitrobenzene	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2-Nitrophenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
4-Nitrophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
N-Nitroso-di-n-propylamine	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
N-Nitrosodiphenylamine	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Pentachlorophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Phenanthrene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Phenol	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
Pyrene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
1,2,4-Trichlorobenzene	EPA 8270C	9L08085	9.7	ND	0.966	12/8/2009	12/11/2009	
2,4,5-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
2,4,6-Trichlorophenol	EPA 8270C	9L08085	19	ND	0.966	12/8/2009	12/11/2009	
Surrogate: 2,4,6-Tribromophenol (40-120%)								103 %
Surrogate: 2-Fluorobiphenyl (50-120%)								82 %
Surrogate: 2-Fluorophenol (30-120%)								66 %
Surrogate: Nitrobenzene-d5 (45-120%)								82 %
Surrogate: Phenol-d6 (35-120%)								70 %
Surrogate: Terphenyl-d14 (50-125%)								97 %

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 Project Manager

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Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
4,4'-DDE	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
4,4'-DDT	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Aldrin	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
alpha-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
beta-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
delta-BHC	EPA 3510C/8081A	9L09036	0.20	ND	1.01	12/9/2009	12/10/2009	C-1
Dieldrin	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endosulfan I	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endosulfan II	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endosulfan sulfate	EPA 3510C/8081A	9L09036	0.20	ND	1.01	12/9/2009	12/10/2009	C-1
Endrin	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endrin aldehyde	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endrin ketone	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
gamma-BHC (Lindane)	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Heptachlor	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Heptachlor epoxide	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Methoxychlor	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Chlordane	EPA 3510C/8081A	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Toxaphene	EPA 3510C/8081A	9L09036	5.1	ND	1.01	12/9/2009	12/10/2009	
Surrogate: Decachlorobiphenyl (45-120%)				100 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				84 %				

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Sampled: 12/04/09  
 Received: 12/05/09

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
4,4'-DDE	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
4,4'-DDT	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Aldrin	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
alpha-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
beta-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
delta-BHC	EPA 3510C/8081A	9L09036	0.20	ND	1.01	12/9/2009	12/10/2009	C-1
Dieldrin	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endosulfan I	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endosulfan II	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endosulfan sulfate	EPA 3510C/8081A	9L09036	0.20	ND	1.01	12/9/2009	12/10/2009	C-1
Endrin	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endrin aldehyde	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Endrin ketone	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
gamma-BHC (Lindane)	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Heptachlor	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Heptachlor epoxide	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Methoxychlor	EPA 3510C/8081A	9L09036	0.10	ND	1.01	12/9/2009	12/10/2009	
Chlordane	EPA 3510C/8081A	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Toxaphene	EPA 3510C/8081A	9L09036	5.1	ND	1.01	12/9/2009	12/10/2009	
Surrogate: Decachlorobiphenyl (45-120%)				99 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				80 %				

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Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
4,4'-DDE	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
4,4'-DDT	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Aldrin	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
alpha-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
beta-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
delta-BHC	EPA 3510C/8081A	9L09036	0.20	ND	1	12/9/2009	12/10/2009	C-1
Dieldrin	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endosulfan I	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endosulfan II	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endosulfan sulfate	EPA 3510C/8081A	9L09036	0.20	ND	1	12/9/2009	12/10/2009	C-1
Endrin	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endrin aldehyde	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endrin ketone	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
gamma-BHC (Lindane)	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Heptachlor	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Heptachlor epoxide	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Methoxychlor	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Chlordane	EPA 3510C/8081A	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Toxaphene	EPA 3510C/8081A	9L09036	5.0	ND	1	12/9/2009	12/10/2009	
Surrogate: Decachlorobiphenyl (45-120%)				95 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				80 %				

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Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
4,4'-DDE	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
4,4'-DDT	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Aldrin	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
alpha-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
beta-BHC	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
delta-BHC	EPA 3510C/8081A	9L09036	0.20	ND	1	12/9/2009	12/10/2009	C-1
Dieldrin	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endosulfan I	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endosulfan II	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endosulfan sulfate	EPA 3510C/8081A	9L09036	0.20	ND	1	12/9/2009	12/10/2009	C-1
Endrin	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endrin aldehyde	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Endrin ketone	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
gamma-BHC (Lindane)	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Heptachlor	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Heptachlor epoxide	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Methoxychlor	EPA 3510C/8081A	9L09036	0.10	ND	1	12/9/2009	12/10/2009	
Chlordane	EPA 3510C/8081A	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Toxaphene	EPA 3510C/8081A	9L09036	5.0	ND	1	12/9/2009	12/10/2009	
Surrogate: Decachlorobiphenyl (45-120%)				101 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				65 %				

TestAmerica Irvine

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1221	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1232	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1242	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1248	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1254	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1260	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				82 %				
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1221	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1232	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1242	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1248	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1254	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
Aroclor 1260	EPA 8082	9L09036	1.0	ND	1.01	12/9/2009	12/10/2009	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				81 %				
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1221	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1232	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1242	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1248	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1254	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1260	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				87 %				

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Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water)</b>								
<b>Reporting Units: ug/l</b>								
Aroclor 1016	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1221	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1232	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1242	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1248	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1254	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
Aroclor 1260	EPA 8082	9L09036	1.0	ND	1	12/9/2009	12/10/2009	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				85 %				

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Sampled: 12/04/09  
 Received: 12/05/09

## Alcohols by EPA Method 8015 modified

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ISL0598-01 (Subpart CC (A) - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Methanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
1-Propanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Surrogate: Isopropyl Acetate (39-128%)				50 %				
<b>Sample ID: ISL0598-02 (Subpart CC (B) - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Methanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
1-Propanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Surrogate: Isopropyl Acetate (39-128%)				50 %				
<b>Sample ID: ISL0598-03 (Subpart CC (C) - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Methanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
1-Propanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Surrogate: Isopropyl Acetate (39-128%)				49 %				
<b>Sample ID: ISL0598-04 (Subpart CC (D) - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Methanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
1-Propanol	SW846 8015B	9121636	10.0	ND	1	12/9/2009	12/9/2009	
Surrogate: Isopropyl Acetate (39-128%)				50 %				

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Sampled: 12/04/09  
 Received: 12/05/09

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>Blank Analyzed: 12/11/2009 (9L11040-BLK1)</b>										
Benzene	ND	2.0	ug/l							
Bromobenzene	ND	5.0	ug/l							
Bromochloromethane	ND	5.0	ug/l							
Bromodichloromethane	ND	2.0	ug/l							
Bromoform	ND	5.0	ug/l							
Bromomethane	ND	5.0	ug/l							
n-Butylbenzene	ND	5.0	ug/l							
sec-Butylbenzene	ND	5.0	ug/l							
tert-Butylbenzene	ND	5.0	ug/l							
Carbon tetrachloride	ND	5.0	ug/l							
Chlorobenzene	ND	2.0	ug/l							
Chloroethane	ND	5.0	ug/l							
Chloroform	ND	2.0	ug/l							
Chloromethane	ND	5.0	ug/l							
2-Chlorotoluene	ND	5.0	ug/l							
4-Chlorotoluene	ND	5.0	ug/l							
1,2-Dibromo-3-chloropropane	ND	5.0	ug/l							
Dibromochloromethane	ND	2.0	ug/l							
1,2-Dibromoethane (EDB)	ND	2.0	ug/l							
Dibromomethane	ND	2.0	ug/l							
1,2-Dichlorobenzene	ND	2.0	ug/l							
1,3-Dichlorobenzene	ND	2.0	ug/l							
1,4-Dichlorobenzene	ND	2.0	ug/l							
Dichlorodifluoromethane	ND	5.0	ug/l							
1,1-Dichloroethane	ND	2.0	ug/l							
1,2-Dichloroethane	ND	2.0	ug/l							
1,1-Dichloroethene	ND	5.0	ug/l							
cis-1,2-Dichloroethene	ND	2.0	ug/l							
trans-1,2-Dichloroethene	ND	2.0	ug/l							
1,2-Dichloropropane	ND	2.0	ug/l							
1,3-Dichloropropane	ND	2.0	ug/l							
2,2-Dichloropropane	ND	2.0	ug/l							
cis-1,3-Dichloropropene	ND	2.0	ug/l							
trans-1,3-Dichloropropene	ND	2.0	ug/l							
1,1-Dichloropropene	ND	2.0	ug/l							
Ethylbenzene	ND	2.0	ug/l							

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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>Blank Analyzed: 12/11/2009 (9L11040-BLK1)</b>										
Hexachlorobutadiene	ND	5.0	ug/l							
Isopropylbenzene	ND	2.0	ug/l							
p-Isopropyltoluene	ND	2.0	ug/l							
Methylene chloride	ND	5.0	ug/l							
Naphthalene	ND	5.0	ug/l							
n-Propylbenzene	ND	2.0	ug/l							
Styrene	ND	2.0	ug/l							
1,1,1,2-Tetrachloroethane	ND	5.0	ug/l							
1,1,2,2-Tetrachloroethane	ND	2.0	ug/l							
Tetrachloroethene	ND	2.0	ug/l							
Toluene	ND	2.0	ug/l							
1,2,3-Trichlorobenzene	ND	5.0	ug/l							
1,2,4-Trichlorobenzene	ND	5.0	ug/l							
1,1,1-Trichloroethane	ND	2.0	ug/l							
1,1,2-Trichloroethane	ND	2.0	ug/l							
Trichloroethene	ND	2.0	ug/l							
Trichlorofluoromethane	ND	5.0	ug/l							
1,2,3-Trichloropropane	ND	10	ug/l							
1,2,4-Trimethylbenzene	ND	2.0	ug/l							
1,3,5-Trimethylbenzene	ND	2.0	ug/l							
Vinyl chloride	ND	5.0	ug/l							
m,p-Xylenes	ND	2.0	ug/l							
o-Xylene	ND	2.0	ug/l							
Surrogate: 4-Bromofluorobenzene	23.2		ug/l	25.0		93	80-120			
Surrogate: Dibromofluoromethane	20.6		ug/l	25.0		82	80-120			
Surrogate: Toluene-d8	25.2		ug/l	25.0		101	80-120			
<b>LCS Analyzed: 12/11/2009 (9L11040-BS1)</b>										
Benzene	26.0	2.0	ug/l	25.0		104	70-120			
Bromobenzene	26.9	5.0	ug/l	25.0		108	75-120			
Bromochloromethane	22.2	5.0	ug/l	25.0		89	70-130			
Bromodichloromethane	28.4	2.0	ug/l	25.0		114	70-135			
Bromoform	24.8	5.0	ug/l	25.0		99	55-130			
Bromomethane	23.9	5.0	ug/l	25.0		96	65-140			
n-Butylbenzene	25.6	5.0	ug/l	25.0		102	70-130			
sec-Butylbenzene	29.5	5.0	ug/l	25.0		118	70-125			

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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>LCS Analyzed: 12/11/2009 (9L11040-BS1)</b>										
tert-Butylbenzene	25.5	5.0	ug/l	25.0		102	70-125			
Carbon tetrachloride	27.4	5.0	ug/l	25.0		110	65-140			
Chlorobenzene	27.2	2.0	ug/l	25.0		109	75-120			
Chloroethane	22.7	5.0	ug/l	25.0		91	60-140			
Chloroform	23.2	2.0	ug/l	25.0		93	70-130			
Chloromethane	17.7	5.0	ug/l	25.0		71	50-140			
2-Chlorotoluene	26.8	5.0	ug/l	25.0		107	70-125			
4-Chlorotoluene	27.5	5.0	ug/l	25.0		110	75-125			
1,2-Dibromo-3-chloropropane	26.9	5.0	ug/l	25.0		108	50-135			
Dibromochloromethane	28.5	2.0	ug/l	25.0		114	70-140			
1,2-Dibromoethane (EDB)	27.9	2.0	ug/l	25.0		112	75-125			
Dibromomethane	25.1	2.0	ug/l	25.0		101	70-125			
1,2-Dichlorobenzene	28.2	2.0	ug/l	25.0		113	75-120			
1,3-Dichlorobenzene	28.3	2.0	ug/l	25.0		113	75-120			
1,4-Dichlorobenzene	25.5	2.0	ug/l	25.0		102	75-120			
Dichlorodifluoromethane	16.5	5.0	ug/l	25.0		66	35-155			
1,1-Dichloroethane	23.3	2.0	ug/l	25.0		93	70-125			
1,2-Dichloroethane	26.0	2.0	ug/l	25.0		104	60-140			
1,1-Dichloroethene	24.0	5.0	ug/l	25.0		96	70-125			
cis-1,2-Dichloroethene	24.6	2.0	ug/l	25.0		98	70-125			
trans-1,2-Dichloroethene	23.6	2.0	ug/l	25.0		94	70-125			
1,2-Dichloropropane	25.1	2.0	ug/l	25.0		100	70-125			
1,3-Dichloropropane	27.7	2.0	ug/l	25.0		111	70-120			
2,2-Dichloropropane	26.3	2.0	ug/l	25.0		105	65-140			
cis-1,3-Dichloropropene	34.2	2.0	ug/l	25.0		137	75-125			L
trans-1,3-Dichloropropene	25.6	2.0	ug/l	25.0		102	70-125			
1,1-Dichloropropene	30.2	2.0	ug/l	25.0		121	75-130			
Ethylbenzene	29.9	2.0	ug/l	25.0		120	75-125			
Hexachlorobutadiene	31.6	5.0	ug/l	25.0		126	65-135			
Isopropylbenzene	25.5	2.0	ug/l	25.0		102	75-130			
p-Isopropyltoluene	27.8	2.0	ug/l	25.0		111	75-125			
Methylene chloride	22.8	5.0	ug/l	25.0		91	55-130			
Naphthalene	25.7	5.0	ug/l	25.0		103	55-135			
n-Propylbenzene	28.6	2.0	ug/l	25.0		114	75-130			
Styrene	30.5	2.0	ug/l	25.0		122	75-130			
1,1,1,2-Tetrachloroethane	27.4	5.0	ug/l	25.0		110	70-130			

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 Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>LCS Analyzed: 12/11/2009 (9L11040-BS1)</b>										
1,1,2,2-Tetrachloroethane	27.0	2.0	ug/l	25.0		108	55-130			
Tetrachloroethene	30.8	2.0	ug/l	25.0		123	70-125			
Toluene	25.1	2.0	ug/l	25.0		100	70-120			
1,2,3-Trichlorobenzene	30.0	5.0	ug/l	25.0		120	65-125			
1,2,4-Trichlorobenzene	30.7	5.0	ug/l	25.0		123	70-135			
1,1,1-Trichloroethane	25.3	2.0	ug/l	25.0		101	65-135			
1,1,2-Trichloroethane	26.9	2.0	ug/l	25.0		108	70-125			
Trichloroethene	25.7	2.0	ug/l	25.0		103	70-125			
Trichlorofluoromethane	23.4	5.0	ug/l	25.0		94	65-145			
1,2,3-Trichloropropane	25.4	10	ug/l	25.0		102	60-130			
1,2,4-Trimethylbenzene	26.2	2.0	ug/l	25.0		105	75-125			
1,3,5-Trimethylbenzene	26.3	2.0	ug/l	25.0		105	75-125			
Vinyl chloride	19.4	5.0	ug/l	25.0		78	55-135			
m,p-Xylenes	62.6	2.0	ug/l	50.0		125	75-125			
o-Xylene	30.1	2.0	ug/l	25.0		121	75-125			
Surrogate: 4-Bromofluorobenzene	25.6		ug/l	25.0		102	80-120			
Surrogate: Dibromofluoromethane	21.7		ug/l	25.0		87	80-120			
Surrogate: Toluene-d8	25.3		ug/l	25.0		101	80-120			
<b>Matrix Spike Analyzed: 12/11/2009 (9L11040-MS1)</b>					<b>Source: ISL0598-01</b>					
Benzene	24.7	2.0	ug/l	25.0	ND	99	65-125			
Bromobenzene	26.4	5.0	ug/l	25.0	ND	106	70-125			
Bromochloromethane	21.8	5.0	ug/l	25.0	ND	87	65-135			
Bromodichloromethane	28.1	2.0	ug/l	25.0	ND	112	70-135			
Bromoform	24.2	5.0	ug/l	25.0	ND	97	55-135			
Bromomethane	23.8	5.0	ug/l	25.0	ND	95	55-145			
n-Butylbenzene	25.0	5.0	ug/l	25.0	ND	100	65-135			
sec-Butylbenzene	27.8	5.0	ug/l	25.0	ND	111	70-125			
tert-Butylbenzene	24.1	5.0	ug/l	25.0	ND	96	65-130			
Carbon tetrachloride	27.1	5.0	ug/l	25.0	ND	109	65-140			
Chlorobenzene	25.5	2.0	ug/l	25.0	ND	102	75-125			
Chloroethane	22.4	5.0	ug/l	25.0	ND	90	55-140			
Chloroform	22.7	2.0	ug/l	25.0	ND	91	65-135			
Chloromethane	17.2	5.0	ug/l	25.0	ND	69	45-145			
2-Chlorotoluene	25.9	5.0	ug/l	25.0	ND	104	65-135			
4-Chlorotoluene	26.7	5.0	ug/l	25.0	ND	107	70-135			

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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>Matrix Spike Analyzed: 12/11/2009 (9L11040-MS1)</b>					<b>Source: ISL0598-01</b>					
1,2-Dibromo-3-chloropropane	25.7	5.0	ug/l	25.0	ND	103	45-145			
Dibromochloromethane	27.7	2.0	ug/l	25.0	ND	111	65-140			
1,2-Dibromoethane (EDB)	26.8	2.0	ug/l	25.0	ND	107	70-130			
Dibromomethane	24.9	2.0	ug/l	25.0	ND	100	65-135			
1,2-Dichlorobenzene	27.2	2.0	ug/l	25.0	ND	109	75-125			
1,3-Dichlorobenzene	27.2	2.0	ug/l	25.0	ND	109	75-125			
1,4-Dichlorobenzene	24.9	2.0	ug/l	25.0	ND	99	75-125			
Dichlorodifluoromethane	17.4	5.0	ug/l	25.0	ND	70	25-155			
1,1-Dichloroethane	22.6	2.0	ug/l	25.0	ND	90	65-130			
1,2-Dichloroethane	25.5	2.0	ug/l	25.0	ND	102	60-140			
1,1-Dichloroethene	23.3	5.0	ug/l	25.0	ND	93	60-130			
cis-1,2-Dichloroethene	23.8	2.0	ug/l	25.0	ND	95	65-130			
trans-1,2-Dichloroethene	23.0	2.0	ug/l	25.0	ND	92	65-130			
1,2-Dichloropropane	24.4	2.0	ug/l	25.0	ND	98	65-130			
1,3-Dichloropropane	26.0	2.0	ug/l	25.0	ND	104	65-135			
2,2-Dichloropropane	25.3	2.0	ug/l	25.0	ND	101	60-145			
cis-1,3-Dichloropropene	32.6	2.0	ug/l	25.0	ND	130	70-130			
trans-1,3-Dichloropropene	24.6	2.0	ug/l	25.0	ND	98	65-135			
1,1-Dichloropropene	28.8	2.0	ug/l	25.0	ND	115	70-135			
Ethylbenzene	28.2	2.0	ug/l	25.0	ND	113	65-130			
Hexachlorobutadiene	31.0	5.0	ug/l	25.0	ND	124	60-135			
Isopropylbenzene	25.0	2.0	ug/l	25.0	ND	100	70-135			
p-Isopropyltoluene	26.8	2.0	ug/l	25.0	ND	107	65-130			
Methylene chloride	21.2	5.0	ug/l	25.0	ND	85	50-135			
Naphthalene	24.4	5.0	ug/l	25.0	ND	98	50-140			
n-Propylbenzene	27.7	2.0	ug/l	25.0	ND	111	70-135			
Styrene	9.38	2.0	ug/l	25.0	ND	38	50-145			M2
1,1,1,2-Tetrachloroethane	26.1	5.0	ug/l	25.0	ND	104	65-140			
1,1,2,2-Tetrachloroethane	26.6	2.0	ug/l	25.0	ND	106	55-135			
Tetrachloroethene	29.2	2.0	ug/l	25.0	ND	117	65-130			
Toluene	24.1	2.0	ug/l	25.0	ND	96	70-125			
1,2,3-Trichlorobenzene	28.7	5.0	ug/l	25.0	ND	115	60-135			
1,2,4-Trichlorobenzene	29.8	5.0	ug/l	25.0	ND	119	65-135			
1,1,1-Trichloroethane	24.8	2.0	ug/l	25.0	ND	99	65-140			
1,1,2-Trichloroethane	25.8	2.0	ug/l	25.0	ND	103	65-130			
Trichloroethene	24.8	2.0	ug/l	25.0	ND	99	65-125			

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 Received: 12/05/09

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>Matrix Spike Analyzed: 12/11/2009 (9L11040-MS1)</b>					<b>Source: ISL0598-01</b>					
Trichlorofluoromethane	23.6	5.0	ug/l	25.0	ND	95	60-145			
1,2,3-Trichloropropane	25.2	10	ug/l	25.0	ND	101	55-135			
1,2,4-Trimethylbenzene	25.1	2.0	ug/l	25.0	ND	101	55-135			
1,3,5-Trimethylbenzene	25.1	2.0	ug/l	25.0	ND	100	70-130			
Vinyl chloride	19.2	5.0	ug/l	25.0	ND	77	45-140			
m,p-Xylenes	59.6	2.0	ug/l	50.0	ND	119	65-130			
o-Xylene	28.3	2.0	ug/l	25.0	ND	113	65-125			
<i>Surrogate: 4-Bromofluorobenzene</i>	<i>25.1</i>		<i>ug/l</i>	<i>25.0</i>		<i>100</i>	<i>80-120</i>			
<i>Surrogate: Dibromofluoromethane</i>	<i>21.9</i>		<i>ug/l</i>	<i>25.0</i>		<i>88</i>	<i>80-120</i>			
<i>Surrogate: Toluene-d8</i>	<i>25.2</i>		<i>ug/l</i>	<i>25.0</i>		<i>101</i>	<i>80-120</i>			
<b>Matrix Spike Dup Analyzed: 12/11/2009 (9L11040-MSD1)</b>					<b>Source: ISL0598-01</b>					
Benzene	25.1	2.0	ug/l	25.0	ND	100	65-125	2	20	
Bromobenzene	26.9	5.0	ug/l	25.0	ND	108	70-125	2	20	
Bromochloromethane	21.7	5.0	ug/l	25.0	ND	87	65-135	1	25	
Bromodichloromethane	27.9	2.0	ug/l	25.0	ND	112	70-135	1	20	
Bromoform	23.6	5.0	ug/l	25.0	ND	95	55-135	2	25	
Bromomethane	24.4	5.0	ug/l	25.0	ND	98	55-145	3	25	
n-Butylbenzene	25.6	5.0	ug/l	25.0	ND	103	65-135	3	20	
sec-Butylbenzene	28.8	5.0	ug/l	25.0	ND	115	70-125	3	20	
tert-Butylbenzene	24.9	5.0	ug/l	25.0	ND	100	65-130	3	20	
Carbon tetrachloride	27.3	5.0	ug/l	25.0	ND	109	65-140	1	25	
Chlorobenzene	26.3	2.0	ug/l	25.0	ND	105	75-125	3	20	
Chloroethane	22.9	5.0	ug/l	25.0	ND	92	55-140	2	25	
Chloroform	22.7	2.0	ug/l	25.0	ND	91	65-135	0	20	
Chloromethane	18.2	5.0	ug/l	25.0	ND	73	45-145	6	25	
2-Chlorotoluene	26.5	5.0	ug/l	25.0	ND	106	65-135	2	20	
4-Chlorotoluene	27.4	5.0	ug/l	25.0	ND	110	70-135	3	20	
1,2-Dibromo-3-chloropropane	25.5	5.0	ug/l	25.0	ND	102	45-145	1	30	
Dibromochloromethane	27.7	2.0	ug/l	25.0	ND	111	65-140	0	25	
1,2-Dibromoethane (EDB)	26.8	2.0	ug/l	25.0	ND	107	70-130	0	25	
Dibromomethane	23.4	2.0	ug/l	25.0	ND	94	65-135	6	25	
1,2-Dichlorobenzene	27.2	2.0	ug/l	25.0	ND	109	75-125	0	20	
1,3-Dichlorobenzene	27.4	2.0	ug/l	25.0	ND	110	75-125	1	20	
1,4-Dichlorobenzene	25.1	2.0	ug/l	25.0	ND	101	75-125	1	20	
Dichlorodifluoromethane	17.8	5.0	ug/l	25.0	ND	71	25-155	2	30	

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Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>Matrix Spike Dup Analyzed: 12/11/2009 (9L11040-MSD1)</b>					<b>Source: ISL0598-01</b>					
1,1-Dichloroethane	22.9	2.0	ug/l	25.0	ND	92	65-130	1	20	
1,2-Dichloroethane	24.4	2.0	ug/l	25.0	ND	98	60-140	4	20	
1,1-Dichloroethene	24.2	5.0	ug/l	25.0	ND	97	60-130	4	20	
cis-1,2-Dichloroethene	23.8	2.0	ug/l	25.0	ND	95	65-130	0	20	
trans-1,2-Dichloroethene	23.2	2.0	ug/l	25.0	ND	93	65-130	1	20	
1,2-Dichloropropane	24.1	2.0	ug/l	25.0	ND	97	65-130	1	20	
1,3-Dichloropropane	26.3	2.0	ug/l	25.0	ND	105	65-135	1	25	
2,2-Dichloropropane	24.7	2.0	ug/l	25.0	ND	99	60-145	2	25	
cis-1,3-Dichloropropene	33.1	2.0	ug/l	25.0	ND	132	70-130	2	20	M7
trans-1,3-Dichloropropene	24.8	2.0	ug/l	25.0	ND	99	65-135	1	25	
1,1-Dichloropropene	29.6	2.0	ug/l	25.0	ND	118	70-135	3	20	
Ethylbenzene	29.1	2.0	ug/l	25.0	ND	116	65-130	3	20	
Hexachlorobutadiene	32.4	5.0	ug/l	25.0	ND	130	60-135	4	20	
Isopropylbenzene	26.2	2.0	ug/l	25.0	ND	105	70-135	5	20	
p-Isopropyltoluene	27.5	2.0	ug/l	25.0	ND	110	65-130	3	20	
Methylene chloride	21.5	5.0	ug/l	25.0	ND	86	50-135	2	20	
Naphthalene	24.2	5.0	ug/l	25.0	ND	97	50-140	1	30	
n-Propylbenzene	28.8	2.0	ug/l	25.0	ND	115	70-135	4	20	
Styrene	7.11	2.0	ug/l	25.0	ND	28	50-145	28	30	M2
1,1,1,2-Tetrachloroethane	26.2	5.0	ug/l	25.0	ND	105	65-140	0	20	
1,1,2,2-Tetrachloroethane	26.4	2.0	ug/l	25.0	ND	106	55-135	1	30	
Tetrachloroethene	30.4	2.0	ug/l	25.0	ND	121	65-130	4	20	
Toluene	24.3	2.0	ug/l	25.0	ND	97	70-125	1	20	
1,2,3-Trichlorobenzene	29.1	5.0	ug/l	25.0	ND	116	60-135	1	20	
1,2,4-Trichlorobenzene	29.8	5.0	ug/l	25.0	ND	119	65-135	0	20	
1,1,1-Trichloroethane	24.7	2.0	ug/l	25.0	ND	99	65-140	0	20	
1,1,2-Trichloroethane	25.9	2.0	ug/l	25.0	ND	104	65-130	0	25	
Trichloroethene	25.2	2.0	ug/l	25.0	ND	101	65-125	2	20	
Trichlorofluoromethane	23.6	5.0	ug/l	25.0	ND	95	60-145	0	25	
1,2,3-Trichloropropane	25.1	10	ug/l	25.0	ND	100	55-135	0	30	
1,2,4-Trimethylbenzene	25.8	2.0	ug/l	25.0	ND	103	55-135	3	25	
1,3,5-Trimethylbenzene	25.6	2.0	ug/l	25.0	ND	102	70-130	2	20	
Vinyl chloride	20.2	5.0	ug/l	25.0	ND	81	45-140	5	30	
m,p-Xylenes	61.2	2.0	ug/l	50.0	ND	122	65-130	3	25	
o-Xylene	28.9	2.0	ug/l	25.0	ND	116	65-125	2	20	
Surrogate: 4-Bromofluorobenzene	24.6		ug/l	25.0		98	80-120			

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Project ID: Subpart CC

Report Number: ISL0598

Sampled: 12/04/09  
Received: 12/05/09

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L11040 Extracted: 12/11/09</b>										
<b>Matrix Spike Dup Analyzed: 12/11/2009 (9L11040-MSD1)</b>										
Surrogate: Dibromofluoromethane	22.0		ug/l	25.0		88	80-120			
Surrogate: Toluene-d8	25.1		ug/l	25.0		100	80-120			

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Siemens Water Technologies  
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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>Blank Analyzed: 12/10/2009 (9L08085-BLK1)</b>										
Acenaphthene	ND	10	ug/l							
Acenaphthylene	ND	10	ug/l							
Aniline	ND	10	ug/l							
Anthracene	ND	10	ug/l							
Benzidine	ND	20	ug/l							
Benzo(a)anthracene	ND	10	ug/l							
Benzo(a)pyrene	ND	10	ug/l							
Benzo(b)fluoranthene	ND	10	ug/l							
Benzo(g,h,i)perylene	ND	10	ug/l							
Benzo(k)fluoranthene	ND	10	ug/l							
Benzoic acid	ND	20	ug/l							
Benzyl alcohol	ND	20	ug/l							
4-Bromophenyl phenyl ether	ND	10	ug/l							
Butyl benzyl phthalate	ND	20	ug/l							
4-Chloro-3-methylphenol	ND	20	ug/l							
4-Chloroaniline	ND	10	ug/l							
Bis(2-chloroethoxy)methane	ND	10	ug/l							
Bis(2-chloroethyl)ether	ND	10	ug/l							
Bis(2-chloroisopropyl)ether	ND	10	ug/l							
Bis(2-ethylhexyl)phthalate	ND	50	ug/l							
2-Chloronaphthalene	ND	10	ug/l							
2-Chlorophenol	ND	10	ug/l							
4-Chlorophenyl phenyl ether	ND	10	ug/l							
Chrysene	ND	10	ug/l							
Dibenz(a,h)anthracene	ND	20	ug/l							
Dibenzofuran	ND	10	ug/l							
Di-n-butyl phthalate	ND	20	ug/l							
1,2-Dichlorobenzene	ND	10	ug/l							
1,3-Dichlorobenzene	ND	10	ug/l							
1,4-Dichlorobenzene	ND	10	ug/l							
3,3'-Dichlorobenzidine	ND	20	ug/l							
2,4-Dichlorophenol	ND	10	ug/l							
Diethyl phthalate	ND	10	ug/l							
2,4-Dimethylphenol	ND	20	ug/l							
Dimethyl phthalate	ND	10	ug/l							
4,6-Dinitro-2-methylphenol	ND	20	ug/l							

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>Blank Analyzed: 12/10/2009 (9L08085-BLK1)</b>										
2,4-Dinitrophenol	ND	20	ug/l							
2,4-Dinitrotoluene	ND	10	ug/l							
2,6-Dinitrotoluene	ND	10	ug/l							
Di-n-octyl phthalate	ND	20	ug/l							
1,2-Diphenylhydrazine/Azobenzene	ND	20	ug/l							
Fluoranthene	ND	10	ug/l							
Fluorene	ND	10	ug/l							
Hexachlorobenzene	ND	10	ug/l							
Hexachlorobutadiene	ND	10	ug/l							
Hexachlorocyclopentadiene	ND	20	ug/l							
Hexachloroethane	ND	10	ug/l							
Indeno(1,2,3-cd)pyrene	ND	20	ug/l							
Isophorone	ND	10	ug/l							
2-Methylnaphthalene	ND	10	ug/l							
2-Methylphenol	ND	10	ug/l							
4-Methylphenol	ND	10	ug/l							
Naphthalene	ND	10	ug/l							
2-Nitroaniline	ND	20	ug/l							
3-Nitroaniline	ND	20	ug/l							
4-Nitroaniline	ND	20	ug/l							
Nitrobenzene	ND	20	ug/l							
2-Nitrophenol	ND	10	ug/l							
4-Nitrophenol	ND	20	ug/l							
N-Nitroso-di-n-propylamine	ND	10	ug/l							
N-Nitrosodiphenylamine	ND	10	ug/l							
Pentachlorophenol	ND	20	ug/l							
Phenanthrene	ND	10	ug/l							
Phenol	ND	10	ug/l							
Pyrene	ND	10	ug/l							
1,2,4-Trichlorobenzene	ND	10	ug/l							
2,4,5-Trichlorophenol	ND	20	ug/l							
2,4,6-Trichlorophenol	ND	20	ug/l							
Surrogate: 2,4,6-Tribromophenol	187		ug/l	200		93	40-120			
Surrogate: 2-Fluorobiphenyl	86.2		ug/l	100		86	50-120			
Surrogate: 2-Fluorophenol	142		ug/l	200		71	30-120			
Surrogate: Nitrobenzene-d5	83.2		ug/l	100		83	45-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>Blank Analyzed: 12/10/2009 (9L08085-BLK1)</b>										
Surrogate: Phenol-d6	151		ug/l	200		75	35-120			
Surrogate: Terphenyl-d14	94.5		ug/l	100		95	50-125			
<b>LCS Analyzed: 12/10/2009 (9L08085-BS1)</b>										
Acenaphthene	86.0	10	ug/l	100		86	60-120			MNR1
Acenaphthylene	85.6	10	ug/l	100		86	60-120			
Aniline	77.2	10	ug/l	100		77	35-120			
Anthracene	88.3	10	ug/l	100		88	65-120			
Benzidine	109	20	ug/l	100		109	30-160			
Benzo(a)anthracene	86.7	10	ug/l	100		87	65-120			
Benzo(a)pyrene	92.4	10	ug/l	100		92	55-130			
Benzo(b)fluoranthene	89.4	10	ug/l	100		89	55-125			
Benzo(g,h,i)perylene	95.4	10	ug/l	100		95	45-135			
Benzo(k)fluoranthene	98.8	10	ug/l	100		99	50-125			
Benzoic acid	77.0	20	ug/l	100		77	25-120			
Benzyl alcohol	84.3	20	ug/l	100		84	50-120			
4-Bromophenyl phenyl ether	87.4	10	ug/l	100		87	60-120			
Butyl benzyl phthalate	90.5	20	ug/l	100		90	55-130			
4-Chloro-3-methylphenol	86.6	20	ug/l	100		87	60-120			
4-Chloroaniline	82.6	10	ug/l	100		83	55-120			
Bis(2-chloroethoxy)methane	82.0	10	ug/l	100		82	55-120			
Bis(2-chloroethyl)ether	80.0	10	ug/l	100		80	50-120			
Bis(2-chloroisopropyl)ether	78.5	10	ug/l	100		79	45-120			
Bis(2-ethylhexyl)phthalate	94.1	50	ug/l	100		94	65-130			
2-Chloronaphthalene	80.6	10	ug/l	100		81	60-120			
2-Chlorophenol	75.6	10	ug/l	100		76	45-120			
4-Chlorophenyl phenyl ether	86.5	10	ug/l	100		87	65-120			
Chrysene	89.2	10	ug/l	100		89	65-120			
Dibenz(a,h)anthracene	91.2	20	ug/l	100		91	50-135			
Dibenzofuran	85.3	10	ug/l	100		85	65-120			
Di-n-butyl phthalate	97.2	20	ug/l	100		97	60-125			
1,2-Dichlorobenzene	66.4	10	ug/l	100		66	40-120			
1,3-Dichlorobenzene	62.5	10	ug/l	100		63	35-120			
1,4-Dichlorobenzene	64.3	10	ug/l	100		64	35-120			
3,3'-Dichlorobenzidine	69.6	20	ug/l	100		70	45-135			
2,4-Dichlorophenol	80.8	10	ug/l	100		81	55-120			

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### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>LCS Analyzed: 12/10/2009 (9L08085-BS1)</b>										
Diethyl phthalate	88.7	10	ug/l	100		89	55-120			MNR1
2,4-Dimethylphenol	77.2	20	ug/l	100		77	40-120			
Dimethyl phthalate	86.9	10	ug/l	100		87	30-120			
4,6-Dinitro-2-methylphenol	92.3	20	ug/l	100		92	45-120			
2,4-Dinitrophenol	88.6	20	ug/l	100		89	40-120			
2,4-Dinitrotoluene	92.5	10	ug/l	100		92	65-120			
2,6-Dinitrotoluene	88.2	10	ug/l	100		88	65-120			
Di-n-octyl phthalate	96.1	20	ug/l	100		96	65-135			
1,2-Diphenylhydrazine/Azobenzene	87.3	20	ug/l	100		87	60-120			
Fluoranthene	95.8	10	ug/l	100		96	60-120			
Fluorene	85.9	10	ug/l	100		86	65-120			
Hexachlorobenzene	88.8	10	ug/l	100		89	60-120			
Hexachlorobutadiene	69.8	10	ug/l	100		70	40-120			
Hexachlorocyclopentadiene	125	20	ug/l	100		125	25-120			L
Hexachloroethane	59.2	10	ug/l	100		59	35-120			
Indeno(1,2,3-cd)pyrene	93.1	20	ug/l	100		93	45-135			
Isophorone	86.6	10	ug/l	100		87	50-120			
2-Methylnaphthalene	83.0	10	ug/l	100		83	55-120			
2-Methylphenol	80.8	10	ug/l	100		81	50-120			
4-Methylphenol	82.6	10	ug/l	100		83	50-120			
Naphthalene	77.8	10	ug/l	100		78	55-120			
2-Nitroaniline	86.1	20	ug/l	100		86	65-120			
3-Nitroaniline	90.3	20	ug/l	100		90	60-120			
4-Nitroaniline	93.4	20	ug/l	100		93	55-125			
Nitrobenzene	79.0	20	ug/l	100		79	55-120			
2-Nitrophenol	79.4	10	ug/l	100		79	50-120			
4-Nitrophenol	90.8	20	ug/l	100		91	45-120			
N-Nitroso-di-n-propylamine	87.5	10	ug/l	100		87	45-120			
N-Nitrosodiphenylamine	91.8	10	ug/l	100		92	60-120			
Pentachlorophenol	91.9	20	ug/l	100		92	50-120			
Phenanthrene	86.9	10	ug/l	100		87	65-120			
Phenol	73.0	10	ug/l	100		73	40-120			
Pyrene	87.6	10	ug/l	100		88	55-125			
1,2,4-Trichlorobenzene	72.4	10	ug/l	100		72	45-120			
2,4,5-Trichlorophenol	82.2	20	ug/l	100		82	55-120			
2,4,6-Trichlorophenol	82.6	20	ug/l	100		83	55-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>LCS Analyzed: 12/10/2009 (9L08085-BS1)</b>										
Surrogate: 2,4,6-Tribromophenol	187		ug/l	200		94	40-120			MNR1
Surrogate: 2-Fluorobiphenyl	81.0		ug/l	100		81	50-120			
Surrogate: 2-Fluorophenol	126		ug/l	200		63	30-120			
Surrogate: Nitrobenzene-d5	78.9		ug/l	100		79	45-120			
Surrogate: Phenol-d6	142		ug/l	200		71	35-120			
Surrogate: Terphenyl-d14	86.8		ug/l	100		87	50-125			
<b>LCS Dup Analyzed: 12/10/2009 (9L08085-BSD1)</b>										
Acenaphthene	88.2	10	ug/l	100		88	60-120	3	20	
Acenaphthylene	87.9	10	ug/l	100		88	60-120	3	20	
Aniline	79.3	10	ug/l	100		79	35-120	3	30	
Anthracene	88.6	10	ug/l	100		89	65-120	0	20	
Benzidine	100	20	ug/l	100		100	30-160	9	35	
Benzo(a)anthracene	87.8	10	ug/l	100		88	65-120	1	20	
Benzo(a)pyrene	93.1	10	ug/l	100		93	55-130	1	25	
Benzo(b)fluoranthene	92.1	10	ug/l	100		92	55-125	3	25	
Benzo(g,h,i)perylene	103	10	ug/l	100		103	45-135	8	25	
Benzo(k)fluoranthene	94.5	10	ug/l	100		94	50-125	4	20	
Benzoic acid	90.4	20	ug/l	100		90	25-120	16	30	
Benzyl alcohol	88.0	20	ug/l	100		88	50-120	4	20	
4-Bromophenyl phenyl ether	89.9	10	ug/l	100		90	60-120	3	25	
Butyl benzyl phthalate	92.6	20	ug/l	100		93	55-130	2	20	
4-Chloro-3-methylphenol	91.0	20	ug/l	100		91	60-120	5	25	
4-Chloroaniline	87.0	10	ug/l	100		87	55-120	5	25	
Bis(2-chloroethoxy)methane	85.2	10	ug/l	100		85	55-120	4	20	
Bis(2-chloroethyl)ether	81.2	10	ug/l	100		81	50-120	2	20	
Bis(2-chloroisopropyl)ether	79.1	10	ug/l	100		79	45-120	1	20	
Bis(2-ethylhexyl)phthalate	96.6	50	ug/l	100		97	65-130	3	20	
2-Chloronaphthalene	83.5	10	ug/l	100		84	60-120	4	20	
2-Chlorophenol	76.4	10	ug/l	100		76	45-120	1	25	
4-Chlorophenyl phenyl ether	87.5	10	ug/l	100		87	65-120	1	20	
Chrysene	90.2	10	ug/l	100		90	65-120	1	20	
Dibenz(a,h)anthracene	92.4	20	ug/l	100		92	50-135	1	25	
Dibenzofuran	87.8	10	ug/l	100		88	65-120	3	20	
Di-n-butyl phthalate	96.4	20	ug/l	100		96	60-125	1	20	
1,2-Dichlorobenzene	68.7	10	ug/l	100		69	40-120	3	25	

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Report Number: ISL0598

Sampled: 12/04/09  
 Received: 12/05/09

## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>LCS Dup Analyzed: 12/10/2009 (9L08085-BSD1)</b>										
1,3-Dichlorobenzene	65.5	10	ug/l	100		65	35-120	5	25	
1,4-Dichlorobenzene	66.3	10	ug/l	100		66	35-120	3	25	
3,3'-Dichlorobenzidine	76.6	20	ug/l	100		77	45-135	10	25	
2,4-Dichlorophenol	84.6	10	ug/l	100		85	55-120	5	20	
Diethyl phthalate	88.9	10	ug/l	100		89	55-120	0	30	
2,4-Dimethylphenol	77.9	20	ug/l	100		78	40-120	1	25	
Dimethyl phthalate	88.7	10	ug/l	100		89	30-120	2	30	
4,6-Dinitro-2-methylphenol	94.3	20	ug/l	100		94	45-120	2	25	
2,4-Dinitrophenol	87.6	20	ug/l	100		88	40-120	1	25	
2,4-Dinitrotoluene	93.8	10	ug/l	100		94	65-120	1	20	
2,6-Dinitrotoluene	90.7	10	ug/l	100		91	65-120	3	20	
Di-n-octyl phthalate	100	20	ug/l	100		100	65-135	4	20	
1,2-Diphenylhydrazine/Azobenzene	87.8	20	ug/l	100		88	60-120	1	25	
Fluoranthene	94.5	10	ug/l	100		94	60-120	1	20	
Fluorene	87.6	10	ug/l	100		88	65-120	2	20	
Hexachlorobenzene	90.5	10	ug/l	100		90	60-120	2	20	
Hexachlorobutadiene	71.7	10	ug/l	100		72	40-120	3	25	
Hexachlorocyclopentadiene	133	20	ug/l	100		133	25-120	7	30	L
Hexachloroethane	61.5	10	ug/l	100		61	35-120	4	25	
Indeno(1,2,3-cd)pyrene	101	20	ug/l	100		101	45-135	8	25	
Isophorone	90.6	10	ug/l	100		91	50-120	4	20	
2-Methylnaphthalene	87.2	10	ug/l	100		87	55-120	5	20	
2-Methylphenol	82.1	10	ug/l	100		82	50-120	2	20	
4-Methylphenol	84.4	10	ug/l	100		84	50-120	2	20	
Naphthalene	79.8	10	ug/l	100		80	55-120	3	20	
2-Nitroaniline	88.3	20	ug/l	100		88	65-120	3	20	
3-Nitroaniline	92.4	20	ug/l	100		92	60-120	2	25	
4-Nitroaniline	92.0	20	ug/l	100		92	55-125	1	20	
Nitrobenzene	81.8	20	ug/l	100		82	55-120	4	25	
2-Nitrophenol	84.1	10	ug/l	100		84	50-120	6	25	
4-Nitrophenol	93.9	20	ug/l	100		94	45-120	3	30	
N-Nitroso-di-n-propylamine	88.8	10	ug/l	100		89	45-120	2	20	
N-Nitrosodiphenylamine	93.8	10	ug/l	100		94	60-120	2	20	
Pentachlorophenol	92.7	20	ug/l	100		93	50-120	1	25	
Phenanthrene	87.2	10	ug/l	100		87	65-120	0	20	
Phenol	74.1	10	ug/l	100		74	40-120	2	25	

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L08085 Extracted: 12/08/09</b>										
<b>LCS Dup Analyzed: 12/10/2009 (9L08085-BSD1)</b>										
Pyrene	89.6	10	ug/l	100		90	55-125	2	25	
1,2,4-Trichlorobenzene	75.2	10	ug/l	100		75	45-120	4	20	
2,4,5-Trichlorophenol	86.2	20	ug/l	100		86	55-120	5	30	
2,4,6-Trichlorophenol	86.5	20	ug/l	100		86	55-120	5	30	
Surrogate: 2,4,6-Tribromophenol	194		ug/l	200		97	40-120			
Surrogate: 2-Fluorobiphenyl	83.9		ug/l	100		84	50-120			
Surrogate: 2-Fluorophenol	125		ug/l	200		63	30-120			
Surrogate: Nitrobenzene-d5	82.5		ug/l	100		82	45-120			
Surrogate: Phenol-d6	145		ug/l	200		73	35-120			
Surrogate: Terphenyl-d14	89.5		ug/l	100		89	50-125			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L09036 Extracted: 12/09/09</b>										
<b>Blank Analyzed: 12/09/2009 (9L09036-BLK1)</b>										
4,4'-DDD	ND	0.10	ug/l							
4,4'-DDE	ND	0.10	ug/l							
4,4'-DDT	ND	0.10	ug/l							
Aldrin	ND	0.10	ug/l							
alpha-BHC	ND	0.10	ug/l							
beta-BHC	ND	0.10	ug/l							
delta-BHC	ND	0.20	ug/l							
Dieldrin	ND	0.10	ug/l							
Endosulfan I	ND	0.10	ug/l							
Endosulfan II	ND	0.10	ug/l							
Endosulfan sulfate	ND	0.20	ug/l							
Endrin	ND	0.10	ug/l							
Endrin aldehyde	ND	0.10	ug/l							
Endrin ketone	ND	0.10	ug/l							
gamma-BHC (Lindane)	ND	0.10	ug/l							
Heptachlor	ND	0.10	ug/l							
Heptachlor epoxide	ND	0.10	ug/l							
Methoxychlor	ND	0.10	ug/l							
Chlordane	ND	1.0	ug/l							
Toxaphene	ND	5.0	ug/l							
Surrogate: Decachlorobiphenyl	0.425		ug/l	0.500		85	45-120			
Surrogate: Tetrachloro-m-xylene	0.378		ug/l	0.500		76	35-115			

### LCS Analyzed: 12/09/2009 (9L09036-BS1)

MNR1

4,4'-DDD	0.435	0.10	ug/l	0.500		87	55-120			
4,4'-DDE	0.435	0.10	ug/l	0.500		87	50-120			
4,4'-DDT	0.465	0.10	ug/l	0.500		93	55-120			
Aldrin	0.416	0.10	ug/l	0.500		83	40-115			
alpha-BHC	0.413	0.10	ug/l	0.500		83	45-115			
beta-BHC	0.437	0.10	ug/l	0.500		87	55-115			
delta-BHC	0.435	0.20	ug/l	0.500		87	55-115			
Dieldrin	0.444	0.10	ug/l	0.500		89	55-115			
Endosulfan I	0.438	0.10	ug/l	0.500		88	55-115			
Endosulfan II	0.441	0.10	ug/l	0.500		88	55-120			
Endosulfan sulfate	0.459	0.20	ug/l	0.500		92	60-120			
Endrin	0.460	0.10	ug/l	0.500		92	55-115			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L09036 Extracted: 12/09/09</b>										
<b>LCS Analyzed: 12/09/2009 (9L09036-BS1)</b>										
Endrin aldehyde	0.423	0.10	ug/l	0.500		85	50-120			MNR1
Endrin ketone	0.425	0.10	ug/l	0.500		85	55-120			
gamma-BHC (Lindane)	0.450	0.10	ug/l	0.500		90	45-115			
Heptachlor	0.423	0.10	ug/l	0.500		85	45-115			
Heptachlor epoxide	0.434	0.10	ug/l	0.500		87	55-115			
Methoxychlor	0.427	0.10	ug/l	0.500		85	60-120			
Surrogate: Decachlorobiphenyl	0.419		ug/l	0.500		84	45-120			
Surrogate: Tetrachloro-m-xylene	0.389		ug/l	0.500		78	35-115			
<b>LCS Dup Analyzed: 12/09/2009 (9L09036-BSD1)</b>										
4,4'-DDD	0.451	0.10	ug/l	0.500		90	55-120	4	30	
4,4'-DDE	0.439	0.10	ug/l	0.500		88	50-120	1	30	
4,4'-DDT	0.481	0.10	ug/l	0.500		96	55-120	3	30	
Aldrin	0.411	0.10	ug/l	0.500		82	40-115	1	30	
alpha-BHC	0.387	0.10	ug/l	0.500		77	45-115	6	30	
beta-BHC	0.431	0.10	ug/l	0.500		86	55-115	1	30	
delta-BHC	0.441	0.20	ug/l	0.500		88	55-115	1	30	
Dieldrin	0.451	0.10	ug/l	0.500		90	55-115	2	30	
Endosulfan I	0.438	0.10	ug/l	0.500		88	55-115	0	30	
Endosulfan II	0.456	0.10	ug/l	0.500		91	55-120	3	30	
Endosulfan sulfate	0.477	0.20	ug/l	0.500		95	60-120	4	30	
Endrin	0.470	0.10	ug/l	0.500		94	55-115	2	30	
Endrin aldehyde	0.428	0.10	ug/l	0.500		86	50-120	1	30	
Endrin ketone	0.441	0.10	ug/l	0.500		88	55-120	4	30	
gamma-BHC (Lindane)	0.425	0.10	ug/l	0.500		85	45-115	6	30	
Heptachlor	0.405	0.10	ug/l	0.500		81	45-115	4	30	
Heptachlor epoxide	0.430	0.10	ug/l	0.500		86	55-115	1	30	
Methoxychlor	0.446	0.10	ug/l	0.500		89	60-120	4	30	
Surrogate: Decachlorobiphenyl	0.426		ug/l	0.500		85	45-120			
Surrogate: Tetrachloro-m-xylene	0.363		ug/l	0.500		73	35-115			

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## METHOD BLANK/QC DATA

### POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9L09036 Extracted: 12/09/09</b>										
<b>Blank Analyzed: 12/09/2009 (9L09036-BLK1)</b>										
Aroclor 1016	ND	1.0	ug/l							
Aroclor 1221	ND	1.0	ug/l							
Aroclor 1232	ND	1.0	ug/l							
Aroclor 1242	ND	1.0	ug/l							
Aroclor 1248	ND	1.0	ug/l							
Aroclor 1254	ND	1.0	ug/l							
Aroclor 1260	ND	1.0	ug/l							
Surrogate: Decachlorobiphenyl	0.467		ug/l	0.500		93	45-120			
<b>LCS Analyzed: 12/09/2009 (9L09036-BS2)</b>										
Aroclor 1016	3.45	1.0	ug/l	4.00		86	50-115			MNR1
Aroclor 1260	3.81	1.0	ug/l	4.00		95	60-120			
Surrogate: Decachlorobiphenyl	0.469		ug/l	0.500		94	45-120			
<b>LCS Dup Analyzed: 12/09/2009 (9L09036-BSD2)</b>										
Aroclor 1016	3.44	1.0	ug/l	4.00		86	50-115	0	30	
Aroclor 1260	3.84	1.0	ug/l	4.00		96	60-120	1	25	
Surrogate: Decachlorobiphenyl	0.472		ug/l	0.500		94	45-120			

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## METHOD BLANK/QC DATA

### Alcohols by EPA Method 8015 modified

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 9121636 Extracted: 12/09/09</b>										
<b>Blank Analyzed: 12/09/2009 (9121636-BLK1)</b>										
n-Butanol	ND	10.0	mg/L							
Ethanol	ND	10.0	mg/L							
Isopropyl alcohol	ND	10.0	mg/L							
Methanol	ND	10.0	mg/L							
Isobutanol	ND	10.0	mg/L							
Tertiary Butyl Alcohol	ND	10.0	mg/L							
1-Propanol	ND	10.0	mg/L							
Surrogate: Isopropyl Acetate	25.7		mg/L	50.0		51	39-128			
<b>LCS Analyzed: 12/09/2009 (9121636-BS1)</b>										
n-Butanol	48.6	10.0	mg/L	50.0		97	76-124			MHA
Ethanol	48.3	10.0	mg/L	50.0		97	71-120			MHA
Isopropyl alcohol	50.2	10.0	mg/L	50.0		100	72-122			MHA
Methanol	51.9	10.0	mg/L	50.0		104	74-120			MHA
Isobutanol	49.6	10.0	mg/L	50.0		99	80-122			MHA
Tertiary Butyl Alcohol	47.4	10.0	mg/L	50.0		95	72-122			MHA
1-Propanol	48.0	10.0	mg/L	50.0		96	73-122			MHA
Surrogate: Isopropyl Acetate	48.8		mg/L	50.0		98	39-128			

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## GC CALIBRATION CHECK CRITERIA

Per Method 8000B of SW-846, the percent recovery of the calibration checks for GC analyses must be within  $\pm 15\%$  from the true value for each individual compound or the average % recovery of all compounds in the calibration check solution must be within  $\pm 15\%$  recovery. Per Method 8000B, the end user is to be notified if the latter situation occurs.

The % recovery for the following individual compounds fell outside the  $\pm 15\%$  criteria, however the average % recovery of all compounds in the calibration check solution was within  $\pm 15\%$ , thus meeting the overall calibration check criteria.

<u>Compound</u>	<u>Footnote</u>	<u>Calibration Check</u> <u>% Recovery</u>	<u>Lab Number</u>	<u>Batch</u>
delta-BHC	1	118	ISL0598-01	9L09036
delta-BHC	1	118	ISL0598-02	9L09036
delta-BHC	1	118	ISL0598-03	9L09036
delta-BHC	1	118	ISL0598-04	9L09036
Endosulfan sulfate	1	117	ISL0598-01	9L09036
Endosulfan sulfate	1	117	ISL0598-02	9L09036
Endosulfan sulfate	1	117	ISL0598-03	9L09036
Endosulfan sulfate	1	117	ISL0598-04	9L09036

Footnotes:

- 1 The calibration demonstrated a high bias for this compound. Samples were flagged to indicate a possible high bias in the result for this compound.
- 2 The calibration demonstrated a low bias for this compound. Samples were flagged to indicate a possible low bias in the result for this compound.

## GCMS CALIBRATION CHECK CRITERIA

The % recovery for the following individual compounds fell outside the  $\pm 15\%$  criteria, however the average % recovery of all compounds in the calibration check solution was within  $\pm 15\%$ , thus meeting the overall calibration check criteria.

<u>Compound</u>	<u>Footnote</u>	<u>Calibration Check</u> <u>% Recovery</u>	<u>Lab Number</u>	<u>Batch</u>
Aniline	2	75%	ISL0598-01	9L08085
Aniline	2	75%	ISL0598-02	9L08085
Aniline	2	75%	ISL0598-03	9L08085
Aniline	2	75%	ISL0598-04	9L08085

Footnotes:

- 1 The calibration demonstrated a high bias for this compound. Samples were flagged to indicate a possible high bias in the result for this compound.
- 2 The calibration demonstrated a low bias for this compound. Samples were flagged to indicate a possible low bias in the result for this compound.

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## DATA QUALIFIERS AND DEFINITIONS

- C** Calibration Verification recovery was above the method control limit for this analyte. Analyte not detected, data not impacted.
- C-1** Calibration Verification recovery was above the method control limit for this analyte, however the average % difference for all analytes met method criteria. See Calibration Summary form.
- C-2** Calibration Verification recovery was below the method control limit for this analyte, however the average % difference for all analytes met method criteria. See Calibration Summary form.
- L** Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was above the acceptance limits. Analyte not detected, data not impacted.
- M2** The MS and/or MSD were below the acceptance limits due to sample matrix interference. See Blank Spike (LCS).
- M7** The MS and/or MSD were above the acceptance limits. See Blank Spike (LCS).
- MHA** Due to high levels of analyte in the sample, the MS/MSD calculation does not provide useful spike recovery information. See Blank Spike (LCS).
- MNR1** There was no MS/MSD analyzed with this batch due to insufficient sample volume. See Blank Spike/Blank Spike Duplicate.
- ND** Analyte NOT DETECTED at or above the reporting limit or MDL, if MDL is specified.
- RPD** Relative Percent Difference

## ADDITIONAL COMMENTS

**For 1,2-Diphenylhydrazine:**

The result for 1,2-Diphenylhydrazine is based upon the reading of its breakdown product, Azobenzene.

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## Certification Summary

### TestAmerica Irvine

Method	Matrix	Nelac	California
EPA 3510C/8081A	Water	X	X
EPA 8082	Water	X	X
EPA 8260B	Water	X	X
EPA 8270C	Water	X	X

*Nevada and NELAP provide analyte specific accreditations. Analyte specific information for TestAmerica may be obtained by contacting the laboratory or visiting our website at [www.testamericainc.com](http://www.testamericainc.com)*

### Subcontracted Laboratories

#### TestAmerica - Nashville, TN

2960 Foster Creighton Drive - Nashville, TN 37204

Method Performed: SW846 8015B

Samples: ISL0598-01, ISL0598-02, ISL0598-03, ISL0598-04

### TestAmerica Irvine

Lena Davidkova  
Project Manager

## LABORATORY REPORT

Prepared For: Siemens Water Technologies  
P.O. Box 3308 (2523 Mutahar St.)  
Parker, AZ 85344  
Attention: Todd Guimond

Project: Subpart CC

Sampled: 11/16/10  
Received: 11/17/10  
Issued: 12/02/10 17:11

NELAP #01108CA California ELAP#2706 CSDLAC #10256 AZ #AZ0671 NV #CA01531

*The results listed within this Laboratory Report pertain only to the samples tested in the laboratory. The analyses contained in this report were performed in accordance with the applicable certifications as noted. All soil samples are reported on a wet weight basis unless otherwise noted in the report. This Laboratory Report is confidential and is intended for the sole use of TestAmerica and its client. This report shall not be reproduced, except in full, without written permission from TestAmerica. The Chain(s) of Custody, 4 pages, are included and are an integral part of this report.  
This entire report was reviewed and approved for release.*

## SAMPLE CROSS REFERENCE

SUBCONTRACTED: Refer to the last page for specific subcontract laboratory information included in this report.

### LABORATORY ID

ITK1839-01  
ITK1839-02  
ITK1839-03  
ITK1839-04

### CLIENT ID

Subpart CC-A  
Subpart CC-B  
Subpart CC-C  
Subpart CC-D

### MATRIX

Water  
Water  
Water  
Water

Reviewed By:



**TestAmerica Irvine**

Lena Davidkova  
Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Bromobenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Bromochloromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Bromodichloromethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Bromoform	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Bromomethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
n-Butylbenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
sec-Butylbenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
tert-Butylbenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Carbon tetrachloride	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Chlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Chloroethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Chloroform	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Chloromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
2-Chlorotoluene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
4-Chlorotoluene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromo-3-chloropropane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Dibromochloromethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromoethane (EDB)	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Dibromomethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,4-Dichlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Dichlorodifluoromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
cis-1,2-Dichloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,2-Dichloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloropropane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichloropropane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
2,2-Dichloropropane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
cis-1,3-Dichloropropene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,3-Dichloropropene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloropropene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Ethylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Hexachlorobutadiene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Isopropylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
p-Isopropyltoluene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Methylene chloride	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Naphthalene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Styrene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1,1,2-Tetrachloroethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,1,2,2-Tetrachloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Tetrachloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Toluene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichlorobenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,2,4-Trichlorobenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,1,1-Trichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1,2-Trichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Trichloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Trichlorofluoromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichloropropane	EPA 8260B	10K3087	10	ND	1	11/24/2010	11/24/2010	
1,2,4-Trimethylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,3,5-Trimethylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Vinyl chloride	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
m,p-Xylenes	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
o-Xylene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Surrogate: 4-Bromofluorobenzene (80-120%)				90 %				
Surrogate: Dibromofluoromethane (80-120%)				96 %				
Surrogate: Toluene-d8 (80-120%)				101 %				

TestAmerica Irvine

Lena Davidkova  
 Project Manager



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Sampled: 11/16/10  
 Received: 11/17/10

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Bromobenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Bromochloromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Bromodichloromethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Bromoform	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Bromomethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
n-Butylbenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
sec-Butylbenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
tert-Butylbenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Carbon tetrachloride	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Chlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Chloroethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Chloroform	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Chloromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
2-Chlorotoluene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
4-Chlorotoluene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromo-3-chloropropane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Dibromochloromethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromoethane (EDB)	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Dibromomethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,4-Dichlorobenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Dichlorodifluoromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
cis-1,2-Dichloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,2-Dichloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloropropane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichloropropane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
2,2-Dichloropropane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
cis-1,3-Dichloropropene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,3-Dichloropropene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloropropene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Ethylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Hexachlorobutadiene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Isopropylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
p-Isopropyltoluene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Methylene chloride	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
Naphthalene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	

### TestAmerica Irvine

Lena Davidkova  
 Project Manager

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Styrene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1,1,2-Tetrachloroethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,1,2,2-Tetrachloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Tetrachloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Toluene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichlorobenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,2,4-Trichlorobenzene	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,1,1-Trichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,1,2-Trichloroethane	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Trichloroethene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Trichlorofluoromethane	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichloropropane	EPA 8260B	10K3087	10	ND	1	11/24/2010	11/24/2010	
1,2,4-Trimethylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
1,3,5-Trimethylbenzene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Vinyl chloride	EPA 8260B	10K3087	5.0	ND	1	11/24/2010	11/24/2010	
m,p-Xylenes	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
o-Xylene	EPA 8260B	10K3087	2.0	ND	1	11/24/2010	11/24/2010	
Surrogate: 4-Bromofluorobenzene (80-120%)				90 %				
Surrogate: Dibromofluoromethane (80-120%)				97 %				
Surrogate: Toluene-d8 (80-120%)				99 %				

TestAmerica Irvine

Lena Davidkova  
 Project Manager

Siemens Water Technologies  
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 Parker, AZ 85344  
 Attention: Todd Guimond

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Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Bromobenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Bromochloromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Bromodichloromethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Bromoform	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Bromomethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
n-Butylbenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
sec-Butylbenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
tert-Butylbenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Carbon tetrachloride	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Chlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Chloroethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Chloroform	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Chloromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
2-Chlorotoluene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
4-Chlorotoluene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromo-3-chloropropane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Dibromochloromethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromoethane (EDB)	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Dibromomethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,4-Dichlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Dichlorodifluoromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
cis-1,2-Dichloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,2-Dichloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloropropane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichloropropane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
2,2-Dichloropropane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
cis-1,3-Dichloropropene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,3-Dichloropropene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloropropene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Ethylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Hexachlorobutadiene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Isopropylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
p-Isopropyltoluene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Methylene chloride	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Naphthalene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	

**TestAmerica Irvine**

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Styrene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1,1,2-Tetrachloroethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,1,2,2-Tetrachloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Tetrachloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Toluene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichlorobenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,2,4-Trichlorobenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,1,1-Trichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1,2-Trichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Trichloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Trichlorofluoromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichloropropane	EPA 8260B	10K3081	10	ND	1	11/24/2010	11/24/2010	
1,2,4-Trimethylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,3,5-Trimethylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Vinyl chloride	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
m,p-Xylenes	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
o-Xylene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Surrogate: 4-Bromofluorobenzene (80-120%)								93 %
Surrogate: Dibromofluoromethane (80-120%)								100 %
Surrogate: Toluene-d8 (80-120%)								100 %

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## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water)</b>								
<b>Reporting Units: ug/l</b>								
Benzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	P-HS
Bromobenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Bromochloromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Bromodichloromethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Bromoform	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Bromomethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
n-Butylbenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
sec-Butylbenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
tert-Butylbenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Carbon tetrachloride	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Chlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Chloroethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Chloroform	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Chloromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
2-Chlorotoluene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
4-Chlorotoluene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromo-3-chloropropane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Dibromochloromethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dibromoethane (EDB)	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Dibromomethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,4-Dichlorobenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Dichlorodifluoromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloroethene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
cis-1,2-Dichloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,2-Dichloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2-Dichloropropane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,3-Dichloropropane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
2,2-Dichloropropane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
cis-1,3-Dichloropropene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
trans-1,3-Dichloropropene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1-Dichloropropene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Ethylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Hexachlorobutadiene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Isopropylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
p-Isopropyltoluene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Methylene chloride	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
Naphthalene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	

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## VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water) - cont.</b>								<b>P-HS</b>
<b>Reporting Units: ug/l</b>								
n-Propylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Styrene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1,1,2-Tetrachloroethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,1,2,2-Tetrachloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Tetrachloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Toluene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichlorobenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,2,4-Trichlorobenzene	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,1,1-Trichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,1,2-Trichloroethane	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Trichloroethene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Trichlorofluoromethane	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
1,2,3-Trichloropropane	EPA 8260B	10K3081	10	ND	1	11/24/2010	11/24/2010	
1,2,4-Trimethylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
1,3,5-Trimethylbenzene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Vinyl chloride	EPA 8260B	10K3081	5.0	ND	1	11/24/2010	11/24/2010	
m,p-Xylenes	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
o-Xylene	EPA 8260B	10K3081	2.0	ND	1	11/24/2010	11/24/2010	
Surrogate: 4-Bromofluorobenzene (80-120%)				90 %				
Surrogate: Dibromofluoromethane (80-120%)				98 %				
Surrogate: Toluene-d8 (80-120%)				100 %				

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 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Acenaphthylene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Aniline	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Anthracene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Benzidine	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Benzo(a)anthracene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Benzo(a)pyrene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Benzo(b)fluoranthene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Benzo(g,h,i)perylene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Benzo(k)fluoranthene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Benzoic acid	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Benzyl alcohol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
4-Bromophenyl phenyl ether	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Butyl benzyl phthalate	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
4-Chloro-3-methylphenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
4-Chloroaniline	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Bis(2-chloroethoxy)methane	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Bis(2-chloroethyl)ether	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Bis(2-chloroisopropyl)ether	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Bis(2-ethylhexyl)phthalate	EPA 8270C	10K2349	48	ND	0.962	11/18/2010	11/23/2010	
2-Chloronaphthalene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2-Chlorophenol	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
4-Chlorophenyl phenyl ether	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Chrysene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Dibenz(a,h)anthracene	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Dibenzofuran	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Di-n-butyl phthalate	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
1,2-Dichlorobenzene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
1,3-Dichlorobenzene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
1,4-Dichlorobenzene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
3,3'-Dichlorobenzidine	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
2,4-Dichlorophenol	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Diethyl phthalate	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2,4-Dimethylphenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Dimethyl phthalate	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	C
4,6-Dinitro-2-methylphenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
2,4-Dinitrophenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
2,4-Dinitrotoluene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2,6-Dinitrotoluene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	C
Di-n-octyl phthalate	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	

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Sampled: 11/16/10  
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## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Fluorene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Hexachlorobenzene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Hexachlorobutadiene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Hexachlorocyclopentadiene	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Hexachloroethane	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Indeno(1,2,3-cd)pyrene	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Isophorone	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2-Methylnaphthalene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2-Methylphenol	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
4-Methylphenol	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Naphthalene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2-Nitroaniline	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	C
3-Nitroaniline	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
4-Nitroaniline	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Nitrobenzene	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
2-Nitrophenol	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
4-Nitrophenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
N-Nitroso-di-n-propylamine	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
N-Nitrosodiphenylamine	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Pentachlorophenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Phenanthrene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Phenol	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
Pyrene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
1,2,4-Trichlorobenzene	EPA 8270C	10K2349	9.6	ND	0.962	11/18/2010	11/23/2010	
2,4,5-Trichlorophenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
2,4,6-Trichlorophenol	EPA 8270C	10K2349	19	ND	0.962	11/18/2010	11/23/2010	
Surrogate: 2,4,6-Tribromophenol (40-120%)				76 %				
Surrogate: 2-Fluorobiphenyl (50-120%)				63 %				
Surrogate: 2-Fluorophenol (30-120%)				50 %				
Surrogate: Nitrobenzene-d5 (45-120%)				67 %				
Surrogate: Phenol-d6 (35-120%)				62 %				
Surrogate: Terphenyl-d14 (50-125%)				57 %				

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Sampled: 11/16/10  
 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Acenaphthylene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Aniline	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Anthracene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Benzidine	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Benzo(a)anthracene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Benzo(a)pyrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Benzo(b)fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Benzo(g,h,i)perylene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Benzo(k)fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Benzoic acid	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Benzyl alcohol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
4-Bromophenyl phenyl ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Butyl benzyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
4-Chloro-3-methylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
4-Chloroaniline	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Bis(2-chloroethoxy)methane	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Bis(2-chloroethyl)ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Bis(2-chloroisopropyl)ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Bis(2-ethylhexyl)phthalate	EPA 8270C	10K2798	48	ND	0.952	11/22/2010	11/24/2010	
2-Chloronaphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2-Chlorophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
4-Chlorophenyl phenyl ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Chrysene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Dibenz(a,h)anthracene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Dibenzofuran	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Di-n-butyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
1,2-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
1,3-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
1,4-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
3,3'-Dichlorobenzidine	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
2,4-Dichlorophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Diethyl phthalate	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2,4-Dimethylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Dimethyl phthalate	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
4,6-Dinitro-2-methylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
2,4-Dinitrophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
2,4-Dinitrotoluene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2,6-Dinitrotoluene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Di-n-octyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	

**TestAmerica Irvine**

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Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Fluorene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Hexachlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Hexachlorobutadiene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Hexachlorocyclopentadiene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Hexachloroethane	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Indeno(1,2,3-cd)pyrene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Isophorone	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2-Methylnaphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2-Methylphenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
4-Methylphenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Naphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
3-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
4-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Nitrobenzene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
2-Nitrophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
4-Nitrophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
N-Nitroso-di-n-propylamine	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
N-Nitrosodiphenylamine	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Pentachlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Phenanthrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Phenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
Pyrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
1,2,4-Trichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	11/24/2010	
2,4,5-Trichlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
2,4,6-Trichlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	11/24/2010	
Surrogate: 2,4,6-Tribromophenol (40-120%)				56 %				
Surrogate: 2-Fluorobiphenyl (50-120%)				48 %				Z
Surrogate: 2-Fluorophenol (30-120%)				37 %				
Surrogate: Nitrobenzene-d5 (45-120%)				45 %				
Surrogate: Phenol-d6 (35-120%)				40 %				
Surrogate: Terphenyl-d14 (50-125%)				55 %				

TestAmerica Irvine

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Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Acenaphthylene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Aniline	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Anthracene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzidine	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Benzo(a)anthracene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(a)pyrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(b)fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(g,h,i)perylene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(k)fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzoic acid	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Benzyl alcohol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Bromophenyl phenyl ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Butyl benzyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Chloro-3-methylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Chloroaniline	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-chloroethoxy)methane	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-chloroethyl)ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-chloroisopropyl)ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-ethylhexyl)phthalate	EPA 8270C	10K2798	48	ND	0.952	11/22/2010	12/1/2010	
2-Chloronaphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Chlorophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4-Chlorophenyl phenyl ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Chrysene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Dibenz(a,h)anthracene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Dibenzofuran	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Di-n-butyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
1,2-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
1,3-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
1,4-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
3,3'-Dichlorobenzidine	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4-Dichlorophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Diethyl phthalate	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2,4-Dimethylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Dimethyl phthalate	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4,6-Dinitro-2-methylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4-Dinitrophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4-Dinitrotoluene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2,6-Dinitrotoluene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Di-n-octyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	

**TestAmerica Irvine**

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Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Fluorene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Hexachlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Hexachlorobutadiene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Hexachlorocyclopentadiene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Hexachloroethane	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Indeno(1,2,3-cd)pyrene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Isophorone	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Methylnaphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Methylphenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4-Methylphenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Naphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
3-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Nitrobenzene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2-Nitrophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4-Nitrophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
N-Nitroso-di-n-propylamine	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
N-Nitrosodiphenylamine	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Pentachlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Phenanthrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Phenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Pyrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
1,2,4-Trichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2,4,5-Trichlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4,6-Trichlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Surrogate: 2,4,6-Tribromophenol (40-120%)				56 %				
Surrogate: 2-Fluorobiphenyl (50-120%)				59 %				
Surrogate: 2-Fluorophenol (30-120%)				42 %				
Surrogate: Nitrobenzene-d5 (45-120%)				53 %				
Surrogate: Phenol-d6 (35-120%)				44 %				
Surrogate: Terphenyl-d14 (50-125%)				65 %				

TestAmerica Irvine

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Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Acenaphthylene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Aniline	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Anthracene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzidine	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Benzo(a)anthracene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(a)pyrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(b)fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(g,h,i)perylene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzo(k)fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Benzoic acid	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Benzyl alcohol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Bromophenyl phenyl ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Butyl benzyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Chloro-3-methylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Chloroaniline	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-chloroethoxy)methane	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-chloroethyl)ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-chloroisopropyl)ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Bis(2-ethylhexyl)phthalate	EPA 8270C	10K2798	48	ND	0.952	11/22/2010	12/1/2010	
2-Chloronaphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Chlorophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4-Chlorophenyl phenyl ether	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Chrysene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Dibenz(a,h)anthracene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Dibenzofuran	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Di-n-butyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
1,2-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
1,3-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
1,4-Dichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
3,3'-Dichlorobenzidine	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4-Dichlorophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Diethyl phthalate	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2,4-Dimethylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Dimethyl phthalate	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4,6-Dinitro-2-methylphenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4-Dinitrophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4-Dinitrotoluene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2,6-Dinitrotoluene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Di-n-octyl phthalate	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	

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 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Fluorene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Hexachlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Hexachlorobutadiene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Hexachlorocyclopentadiene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Hexachloroethane	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Indeno(1,2,3-cd)pyrene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Isophorone	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Methylnaphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Methylphenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4-Methylphenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Naphthalene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
3-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
4-Nitroaniline	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Nitrobenzene	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2-Nitrophenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
4-Nitrophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
N-Nitroso-di-n-propylamine	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
N-Nitrosodiphenylamine	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Pentachlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Phenanthrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Phenol	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
Pyrene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
1,2,4-Trichlorobenzene	EPA 8270C	10K2798	9.5	ND	0.952	11/22/2010	12/1/2010	
2,4,5-Trichlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
2,4,6-Trichlorophenol	EPA 8270C	10K2798	19	ND	0.952	11/22/2010	12/1/2010	
Surrogate: 2,4,6-Tribromophenol (40-120%)				66 %				
Surrogate: 2-Fluorobiphenyl (50-120%)				70 %				
Surrogate: 2-Fluorophenol (30-120%)				52 %				
Surrogate: Nitrobenzene-d5 (45-120%)				63 %				
Surrogate: Phenol-d6 (35-120%)				52 %				
Surrogate: Terphenyl-d14 (50-125%)				74 %				

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Siemens Water Technologies  
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Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
4,4'-DDE	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
4,4'-DDT	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	C-1, L
Aldrin	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
alpha-BHC	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
beta-BHC	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
delta-BHC	EPA 3510C/8081A	10K2443	0.19	ND	0.943	11/19/2010	11/20/2010	
Dieldrin	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Endosulfan I	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Endosulfan II	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Endosulfan sulfate	EPA 3510C/8081A	10K2443	0.19	ND	0.943	11/19/2010	11/20/2010	
Endrin	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Endrin aldehyde	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Endrin ketone	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
gamma-BHC (Lindane)	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Heptachlor	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Heptachlor epoxide	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Methoxychlor	EPA 3510C/8081A	10K2443	0.094	ND	0.943	11/19/2010	11/20/2010	
Chlordane	EPA 3510C/8081A	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Toxaphene	EPA 3510C/8081A	10K2443	4.7	ND	0.943	11/19/2010	11/20/2010	
Surrogate: Decachlorobiphenyl (45-120%)				93 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				77 %				

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## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
4,4'-DDE	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
4,4'-DDT	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Aldrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
alpha-BHC	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
beta-BHC	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
delta-BHC	EPA 3510C/8081A	10K2972	0.19	ND	0.962	11/23/2010	11/23/2010	
Dieldrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan I	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan II	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan sulfate	EPA 3510C/8081A	10K2972	0.19	ND	0.962	11/23/2010	11/23/2010	
Endrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endrin aldehyde	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endrin ketone	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	C-2
gamma-BHC (Lindane)	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Heptachlor	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Heptachlor epoxide	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Methoxychlor	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Chlordane	EPA 3510C/8081A	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Toxaphene	EPA 3510C/8081A	10K2972	4.8	ND	0.962	11/23/2010	11/23/2010	
Surrogate: Decachlorobiphenyl (45-120%)				72 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				79 %				

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 Received: 11/17/10

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
4,4'-DDE	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
4,4'-DDT	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Aldrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
alpha-BHC	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
beta-BHC	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
delta-BHC	EPA 3510C/8081A	10K2972	0.19	ND	0.962	11/23/2010	11/23/2010	
Dieldrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan I	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan II	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan sulfate	EPA 3510C/8081A	10K2972	0.19	ND	0.962	11/23/2010	11/23/2010	
Endrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endrin aldehyde	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endrin ketone	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	C-2
gamma-BHC (Lindane)	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Heptachlor	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Heptachlor epoxide	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Methoxychlor	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Chlordane	EPA 3510C/8081A	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Toxaphene	EPA 3510C/8081A	10K2972	4.8	ND	0.962	11/23/2010	11/23/2010	
Surrogate: Decachlorobiphenyl (45-120%)				75 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				81 %				

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 Received: 11/17/10

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
4,4'-DDE	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
4,4'-DDT	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Aldrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
alpha-BHC	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
beta-BHC	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
delta-BHC	EPA 3510C/8081A	10K2972	0.19	ND	0.962	11/23/2010	11/23/2010	
Dieldrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan I	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan II	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endosulfan sulfate	EPA 3510C/8081A	10K2972	0.19	ND	0.962	11/23/2010	11/23/2010	
Endrin	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endrin aldehyde	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Endrin ketone	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	C-2
gamma-BHC (Lindane)	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Heptachlor	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Heptachlor epoxide	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Methoxychlor	EPA 3510C/8081A	10K2972	0.096	ND	0.962	11/23/2010	11/23/2010	
Chlordane	EPA 3510C/8081A	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Toxaphene	EPA 3510C/8081A	10K2972	4.8	ND	0.962	11/23/2010	11/23/2010	
Surrogate: Decachlorobiphenyl (45-120%)				78 %				
Surrogate: Tetrachloro-m-xylene (35-115%)				84 %				

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Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Aroclor 1221	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Aroclor 1232	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Aroclor 1242	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Aroclor 1248	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Aroclor 1254	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
Aroclor 1260	EPA 8082	10K2443	0.94	ND	0.943	11/19/2010	11/20/2010	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				90 %				
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1221	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1232	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1242	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1248	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1254	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1260	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				73 %				
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1221	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1232	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1242	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1248	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1254	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1260	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				84 %				

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## POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water)</b>								
<b>Reporting Units: ug/l</b>								
Aroclor 1016	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1221	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1232	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1242	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1248	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1254	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
Aroclor 1260	EPA 8082	10K2972	0.96	ND	0.962	11/23/2010	11/23/2010	
<i>Surrogate: Decachlorobiphenyl (45-120%)</i>				86 %				

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Lena Davidkova  
Project Manager

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**ITK1839 <Page 23 of 66>**

Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## Alcohols/Gcols by GC

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: ITK1839-01 (Subpart CC-A - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Isopropyl alcohol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Methanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
1-Propanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
<b>Sample ID: ITK1839-02 (Subpart CC-B - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Isopropyl alcohol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Methanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
1-Propanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
<b>Sample ID: ITK1839-03 (Subpart CC-C - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Isopropyl alcohol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Methanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
1-Propanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
<b>Sample ID: ITK1839-04 (Subpart CC-D - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Isopropyl alcohol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
Methanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	
1-Propanol	SW 8015	10K1202	10.0	ND	1	12/1/2010	12/1/2010	

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 Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>Blank Analyzed: 11/24/2010 (10K3081-BLK1)</b>										
Benzene	ND	2.0	ug/l							
Bromobenzene	ND	5.0	ug/l							
Bromochloromethane	ND	5.0	ug/l							
Bromodichloromethane	ND	2.0	ug/l							
Bromoform	ND	5.0	ug/l							
Bromomethane	ND	5.0	ug/l							
n-Butylbenzene	ND	5.0	ug/l							
sec-Butylbenzene	ND	5.0	ug/l							
tert-Butylbenzene	ND	5.0	ug/l							
Carbon tetrachloride	ND	5.0	ug/l							
Chlorobenzene	ND	2.0	ug/l							
Chloroethane	ND	5.0	ug/l							
Chloroform	ND	2.0	ug/l							
Chloromethane	ND	5.0	ug/l							
2-Chlorotoluene	ND	5.0	ug/l							
4-Chlorotoluene	ND	5.0	ug/l							
1,2-Dibromo-3-chloropropane	ND	5.0	ug/l							
Dibromochloromethane	ND	2.0	ug/l							
1,2-Dibromoethane (EDB)	ND	2.0	ug/l							
Dibromomethane	ND	2.0	ug/l							
1,2-Dichlorobenzene	ND	2.0	ug/l							
1,3-Dichlorobenzene	ND	2.0	ug/l							
1,4-Dichlorobenzene	ND	2.0	ug/l							
Dichlorodifluoromethane	ND	5.0	ug/l							
1,1-Dichloroethane	ND	2.0	ug/l							
1,2-Dichloroethane	ND	2.0	ug/l							
1,1-Dichloroethene	ND	5.0	ug/l							
cis-1,2-Dichloroethene	ND	2.0	ug/l							
trans-1,2-Dichloroethene	ND	2.0	ug/l							
1,2-Dichloropropane	ND	2.0	ug/l							
1,3-Dichloropropane	ND	2.0	ug/l							
2,2-Dichloropropane	ND	2.0	ug/l							
cis-1,3-Dichloropropene	ND	2.0	ug/l							
trans-1,3-Dichloropropene	ND	2.0	ug/l							
1,1-Dichloropropene	ND	2.0	ug/l							
Ethylbenzene	ND	2.0	ug/l							

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 Project Manager

Siemens Water Technologies  
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Project ID: Subpart CC  
 Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>Blank Analyzed: 11/24/2010 (10K3081-BLK1)</b>										
Hexachlorobutadiene	ND	5.0	ug/l							
Isopropylbenzene	ND	2.0	ug/l							
p-Isopropyltoluene	ND	2.0	ug/l							
Methylene chloride	ND	5.0	ug/l							
Naphthalene	ND	5.0	ug/l							
n-Propylbenzene	ND	2.0	ug/l							
Styrene	ND	2.0	ug/l							
1,1,1,2-Tetrachloroethane	ND	5.0	ug/l							
1,1,2,2-Tetrachloroethane	ND	2.0	ug/l							
Tetrachloroethene	ND	2.0	ug/l							
Toluene	ND	2.0	ug/l							
1,2,3-Trichlorobenzene	ND	5.0	ug/l							
1,2,4-Trichlorobenzene	ND	5.0	ug/l							
1,1,1-Trichloroethane	ND	2.0	ug/l							
1,1,2-Trichloroethane	ND	2.0	ug/l							
Trichloroethene	ND	2.0	ug/l							
Trichlorofluoromethane	ND	5.0	ug/l							
1,2,3-Trichloropropane	ND	10	ug/l							
1,2,4-Trimethylbenzene	ND	2.0	ug/l							
1,3,5-Trimethylbenzene	ND	2.0	ug/l							
Vinyl chloride	ND	5.0	ug/l							
m,p-Xylenes	ND	2.0	ug/l							
o-Xylene	ND	2.0	ug/l							
Surrogate: 4-Bromofluorobenzene	23.1		ug/l	25.0		92	80-120			
Surrogate: Dibromofluoromethane	24.2		ug/l	25.0		97	80-120			
Surrogate: Toluene-d8	25.0		ug/l	25.0		100	80-120			
<b>LCS Analyzed: 11/24/2010 (10K3081-BS1)</b>										
Benzene	26.2	2.0	ug/l	25.0		105	70-120			
Bromobenzene	25.0	5.0	ug/l	25.0		100	75-120			
Bromochloromethane	24.3	5.0	ug/l	25.0		97	70-130			
Bromodichloromethane	24.8	2.0	ug/l	25.0		99	70-135			
Bromoform	18.3	5.0	ug/l	25.0		73	55-130			
Bromomethane	25.6	5.0	ug/l	25.0		103	65-140			
n-Butylbenzene	27.1	5.0	ug/l	25.0		108	70-130			
sec-Butylbenzene	27.0	5.0	ug/l	25.0		108	70-125			

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Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>LCS Analyzed: 11/24/2010 (10K3081-BS1)</b>										
tert-Butylbenzene	26.1	5.0	ug/l	25.0		105	70-125			
Carbon tetrachloride	21.8	5.0	ug/l	25.0		87	65-140			
Chlorobenzene	26.0	2.0	ug/l	25.0		104	75-120			
Chloroethane	25.4	5.0	ug/l	25.0		102	60-140			
Chloroform	24.4	2.0	ug/l	25.0		97	70-130			
Chloromethane	23.8	5.0	ug/l	25.0		95	50-140			
2-Chlorotoluene	26.0	5.0	ug/l	25.0		104	70-125			
4-Chlorotoluene	26.7	5.0	ug/l	25.0		107	75-125			
1,2-Dibromo-3-chloropropane	23.5	5.0	ug/l	25.0		94	50-135			
Dibromochloromethane	18.9	2.0	ug/l	25.0		76	70-140			
1,2-Dibromoethane (EDB)	25.5	2.0	ug/l	25.0		102	75-125			
Dibromomethane	26.0	2.0	ug/l	25.0		104	70-125			
1,2-Dichlorobenzene	25.6	2.0	ug/l	25.0		102	75-120			
1,3-Dichlorobenzene	26.3	2.0	ug/l	25.0		105	75-120			
1,4-Dichlorobenzene	26.1	2.0	ug/l	25.0		104	75-120			
Dichlorodifluoromethane	23.3	5.0	ug/l	25.0		93	35-155			
1,1-Dichloroethane	26.6	2.0	ug/l	25.0		107	70-125			
1,2-Dichloroethane	25.7	2.0	ug/l	25.0		103	60-140			
1,1-Dichloroethene	25.9	5.0	ug/l	25.0		104	70-125			
cis-1,2-Dichloroethene	26.1	2.0	ug/l	25.0		104	70-125			
trans-1,2-Dichloroethene	26.4	2.0	ug/l	25.0		105	70-125			
1,2-Dichloropropane	27.1	2.0	ug/l	25.0		109	70-125			
1,3-Dichloropropane	25.9	2.0	ug/l	25.0		103	70-120			
2,2-Dichloropropane	27.5	2.0	ug/l	25.0		110	65-140			
cis-1,3-Dichloropropene	26.5	2.0	ug/l	25.0		106	75-125			
trans-1,3-Dichloropropene	27.8	2.0	ug/l	25.0		111	70-125			
1,1-Dichloropropene	26.5	2.0	ug/l	25.0		106	75-130			
Ethylbenzene	26.9	2.0	ug/l	25.0		108	75-125			
Hexachlorobutadiene	24.5	5.0	ug/l	25.0		98	65-135			
Isopropylbenzene	25.1	2.0	ug/l	25.0		100	75-130			
p-Isopropyltoluene	27.6	2.0	ug/l	25.0		110	75-125			
Methylene chloride	24.3	5.0	ug/l	25.0		97	55-130			
Naphthalene	24.6	5.0	ug/l	25.0		98	55-135			
n-Propylbenzene	26.9	2.0	ug/l	25.0		107	75-130			
Styrene	28.0	2.0	ug/l	25.0		112	75-130			
1,1,1,2-Tetrachloroethane	23.7	5.0	ug/l	25.0		95	70-130			

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager



Siemens Water Technologies  
 P.O. Box 3308 (2523 Mutahar St.)  
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Project ID: Subpart CC  
 Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>LCS Analyzed: 11/24/2010 (10K3081-BS1)</b>										
1,1,2,2-Tetrachloroethane	27.4	2.0	ug/l	25.0		110	55-130			
Tetrachloroethene	24.5	2.0	ug/l	25.0		98	70-125			
Toluene	26.0	2.0	ug/l	25.0		104	70-120			
1,2,3-Trichlorobenzene	25.1	5.0	ug/l	25.0		100	65-125			
1,2,4-Trichlorobenzene	26.7	5.0	ug/l	25.0		107	70-135			
1,1,1-Trichloroethane	24.8	2.0	ug/l	25.0		99	65-135			
1,1,2-Trichloroethane	26.4	2.0	ug/l	25.0		105	70-125			
Trichloroethene	24.2	2.0	ug/l	25.0		97	70-125			
Trichlorofluoromethane	27.8	5.0	ug/l	25.0		111	65-145			
1,2,3-Trichloropropane	25.4	10	ug/l	25.0		102	60-130			
1,2,4-Trimethylbenzene	27.4	2.0	ug/l	25.0		110	75-125			
1,3,5-Trimethylbenzene	26.6	2.0	ug/l	25.0		107	75-125			
Vinyl chloride	22.8	5.0	ug/l	25.0		91	55-135			
m,p-Xylenes	54.7	2.0	ug/l	50.0		109	75-125			
o-Xylene	26.5	2.0	ug/l	25.0		106	75-125			
Surrogate: 4-Bromofluorobenzene	23.8		ug/l	25.0		95	80-120			
Surrogate: Dibromofluoromethane	24.0		ug/l	25.0		96	80-120			
Surrogate: Toluene-d8	24.9		ug/l	25.0		99	80-120			

### Matrix Spike Analyzed: 11/24/2010 (10K3081-MS1)

Source: ITK2255-07

Benzene	28.9	2.0	ug/l	25.0	4.04	100	65-125			
Bromobenzene	24.0	5.0	ug/l	25.0	ND	96	70-125			
Bromochloromethane	24.3	5.0	ug/l	25.0	ND	97	65-135			
Bromodichloromethane	23.9	2.0	ug/l	25.0	ND	96	70-135			
Bromoform	18.8	5.0	ug/l	25.0	ND	75	55-135			
Bromomethane	23.5	5.0	ug/l	25.0	ND	94	55-145			
n-Butylbenzene	26.0	5.0	ug/l	25.0	ND	104	65-135			
sec-Butylbenzene	25.6	5.0	ug/l	25.0	0.410	101	70-125			
tert-Butylbenzene	24.5	5.0	ug/l	25.0	ND	98	65-130			
Carbon tetrachloride	20.1	5.0	ug/l	25.0	ND	81	65-140			
Chlorobenzene	25.1	2.0	ug/l	25.0	ND	100	75-125			
Chloroethane	23.2	5.0	ug/l	25.0	ND	93	55-140			
Chloroform	23.4	2.0	ug/l	25.0	ND	94	65-135			
Chloromethane	21.5	5.0	ug/l	25.0	ND	86	45-145			
2-Chlorotoluene	25.0	5.0	ug/l	25.0	ND	100	65-135			
4-Chlorotoluene	25.9	5.0	ug/l	25.0	ND	104	70-135			

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Project ID: Subpart CC  
 Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>Matrix Spike Analyzed: 11/24/2010 (10K3081-MS1)</b>					<b>Source: ITK2255-07</b>					
1,2-Dibromo-3-chloropropane	26.5	5.0	ug/l	25.0	ND	106	45-145			
Dibromochloromethane	18.9	2.0	ug/l	25.0	ND	76	65-140			
1,2-Dibromoethane (EDB)	26.8	2.0	ug/l	25.0	ND	107	70-130			
Dibromomethane	26.2	2.0	ug/l	25.0	ND	105	65-135			
1,2-Dichlorobenzene	25.5	2.0	ug/l	25.0	ND	102	75-125			
1,3-Dichlorobenzene	25.2	2.0	ug/l	25.0	ND	101	75-125			
1,4-Dichlorobenzene	24.5	2.0	ug/l	25.0	ND	98	75-125			
Dichlorodifluoromethane	21.7	5.0	ug/l	25.0	ND	87	25-155			
1,1-Dichloroethane	25.0	2.0	ug/l	25.0	ND	100	65-130			
1,2-Dichloroethane	26.2	2.0	ug/l	25.0	ND	105	60-140			
1,1-Dichloroethene	24.4	5.0	ug/l	25.0	ND	97	60-130			
cis-1,2-Dichloroethene	25.3	2.0	ug/l	25.0	ND	101	65-130			
trans-1,2-Dichloroethene	25.2	2.0	ug/l	25.0	ND	101	65-130			
1,2-Dichloropropane	26.2	2.0	ug/l	25.0	ND	105	65-130			
1,3-Dichloropropane	26.9	2.0	ug/l	25.0	ND	108	65-135			
2,2-Dichloropropane	25.8	2.0	ug/l	25.0	ND	103	60-145			
cis-1,3-Dichloropropene	25.6	2.0	ug/l	25.0	ND	103	70-130			
trans-1,3-Dichloropropene	27.2	2.0	ug/l	25.0	ND	109	65-135			
1,1-Dichloropropene	25.1	2.0	ug/l	25.0	ND	100	70-135			
Ethylbenzene	30.9	2.0	ug/l	25.0	5.17	103	65-130			
Hexachlorobutadiene	23.2	5.0	ug/l	25.0	ND	93	60-135			
Isopropylbenzene	24.2	2.0	ug/l	25.0	0.870	93	70-135			
p-Isopropyltoluene	26.0	2.0	ug/l	25.0	0.350	103	65-130			
Methylene chloride	23.4	5.0	ug/l	25.0	ND	93	50-135			
Naphthalene	34.3	5.0	ug/l	25.0	6.52	111	50-140			
n-Propylbenzene	27.0	2.0	ug/l	25.0	1.62	101	70-135			
Styrene	26.3	2.0	ug/l	25.0	ND	105	50-145			
1,1,1,2-Tetrachloroethane	23.0	5.0	ug/l	25.0	ND	92	65-140			
1,1,1,2,2-Tetrachloroethane	29.4	2.0	ug/l	25.0	ND	118	55-135			
Tetrachloroethene	23.8	2.0	ug/l	25.0	ND	95	65-130			
Toluene	24.7	2.0	ug/l	25.0	ND	99	70-125			
1,2,3-Trichlorobenzene	24.9	5.0	ug/l	25.0	ND	100	60-135			
1,2,4-Trichlorobenzene	26.3	5.0	ug/l	25.0	ND	105	65-135			
1,1,1-Trichloroethane	23.4	2.0	ug/l	25.0	ND	94	65-140			
1,1,2-Trichloroethane	27.1	2.0	ug/l	25.0	ND	108	65-130			
Trichloroethene	23.3	2.0	ug/l	25.0	ND	93	65-125			

**TestAmerica Irvine**

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Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>Matrix Spike Analyzed: 11/24/2010 (10K3081-MS1)</b>					<b>Source: ITK2255-07</b>					
Trichlorofluoromethane	26.2	5.0	ug/l	25.0	ND	105	60-145			
1,2,3-Trichloropropane	27.3	10	ug/l	25.0	ND	109	55-135			
1,2,4-Trimethylbenzene	58.3	2.0	ug/l	25.0	33.8	98	55-135			
1,3,5-Trimethylbenzene	27.4	2.0	ug/l	25.0	1.96	102	70-130			
Vinyl chloride	20.2	5.0	ug/l	25.0	ND	81	45-140			
m,p-Xylenes	60.7	2.0	ug/l	50.0	7.81	106	65-130			
o-Xylene	27.1	2.0	ug/l	25.0	1.22	103	65-125			
<i>Surrogate: 4-Bromofluorobenzene</i>	<i>24.2</i>		<i>ug/l</i>	<i>25.0</i>		<i>97</i>	<i>80-120</i>			
<i>Surrogate: Dibromofluoromethane</i>	<i>24.1</i>		<i>ug/l</i>	<i>25.0</i>		<i>96</i>	<i>80-120</i>			
<i>Surrogate: Toluene-d8</i>	<i>24.9</i>		<i>ug/l</i>	<i>25.0</i>		<i>100</i>	<i>80-120</i>			
<b>Matrix Spike Dup Analyzed: 11/24/2010 (10K3081-MSD1)</b>					<b>Source: ITK2255-07</b>					
Benzene	28.7	2.0	ug/l	25.0	4.04	99	65-125	0.9	20	
Bromobenzene	24.0	5.0	ug/l	25.0	ND	96	70-125	0.4	20	
Bromochloromethane	23.6	5.0	ug/l	25.0	ND	95	65-135	3	25	
Bromodichloromethane	23.7	2.0	ug/l	25.0	ND	95	70-135	0.9	20	
Bromoform	17.7	5.0	ug/l	25.0	ND	71	55-135	7	25	
Bromomethane	23.4	5.0	ug/l	25.0	ND	93	55-145	0.7	25	
n-Butylbenzene	26.5	5.0	ug/l	25.0	ND	106	65-135	2	20	
sec-Butylbenzene	25.9	5.0	ug/l	25.0	0.410	102	70-125	1	20	
tert-Butylbenzene	25.1	5.0	ug/l	25.0	ND	100	65-130	2	20	
Carbon tetrachloride	20.7	5.0	ug/l	25.0	ND	83	65-140	3	25	
Chlorobenzene	24.8	2.0	ug/l	25.0	ND	99	75-125	1	20	
Chloroethane	23.0	5.0	ug/l	25.0	ND	92	55-140	1	25	
Chloroform	23.4	2.0	ug/l	25.0	ND	94	65-135	0.1	20	
Chloromethane	20.4	5.0	ug/l	25.0	ND	82	45-145	5	25	
2-Chlorotoluene	25.1	5.0	ug/l	25.0	ND	100	65-135	0.4	20	
4-Chlorotoluene	25.9	5.0	ug/l	25.0	ND	104	70-135	0.2	20	
1,2-Dibromo-3-chloropropane	23.5	5.0	ug/l	25.0	ND	94	45-145	12	30	
Dibromochloromethane	18.6	2.0	ug/l	25.0	ND	74	65-140	2	25	
1,2-Dibromoethane (EDB)	24.8	2.0	ug/l	25.0	ND	99	70-130	8	25	
Dibromomethane	24.3	2.0	ug/l	25.0	ND	97	65-135	7	25	
1,2-Dichlorobenzene	24.9	2.0	ug/l	25.0	ND	100	75-125	2	20	
1,3-Dichlorobenzene	25.3	2.0	ug/l	25.0	ND	101	75-125	0.5	20	
1,4-Dichlorobenzene	25.0	2.0	ug/l	25.0	ND	100	75-125	2	20	
Dichlorodifluoromethane	21.0	5.0	ug/l	25.0	ND	84	25-155	3	30	

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Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>Matrix Spike Dup Analyzed: 11/24/2010 (10K3081-MSD1)</b>					<b>Source: ITK2255-07</b>					
1,1-Dichloroethane	25.0	2.0	ug/l	25.0	ND	100	65-130	0	20	
1,2-Dichloroethane	24.2	2.0	ug/l	25.0	ND	97	60-140	8	20	
1,1-Dichloroethene	25.0	5.0	ug/l	25.0	ND	100	60-130	3	20	
cis-1,2-Dichloroethene	24.8	2.0	ug/l	25.0	ND	99	65-130	2	20	
trans-1,2-Dichloroethene	25.4	2.0	ug/l	25.0	ND	102	65-130	0.7	20	
1,2-Dichloropropane	25.6	2.0	ug/l	25.0	ND	102	65-130	2	20	
1,3-Dichloropropane	24.8	2.0	ug/l	25.0	ND	99	65-135	8	25	
2,2-Dichloropropane	25.5	2.0	ug/l	25.0	ND	102	60-145	1	25	
cis-1,3-Dichloropropene	24.7	2.0	ug/l	25.0	ND	99	70-130	4	20	
trans-1,3-Dichloropropene	25.7	2.0	ug/l	25.0	ND	103	65-135	6	25	
1,1-Dichloropropene	24.7	2.0	ug/l	25.0	ND	99	70-135	1	20	
Ethylbenzene	30.8	2.0	ug/l	25.0	5.17	103	65-130	0.2	20	
Hexachlorobutadiene	23.9	5.0	ug/l	25.0	ND	96	60-135	3	20	
Isopropylbenzene	24.8	2.0	ug/l	25.0	0.870	96	70-135	3	20	
p-Isopropyltoluene	26.4	2.0	ug/l	25.0	0.350	104	65-130	1	20	
Methylene chloride	23.0	5.0	ug/l	25.0	ND	92	50-135	2	20	
Naphthalene	32.1	5.0	ug/l	25.0	6.52	102	50-140	7	30	
n-Propylbenzene	27.0	2.0	ug/l	25.0	1.62	102	70-135	0.3	20	
Styrene	25.9	2.0	ug/l	25.0	ND	104	50-145	2	30	
1,1,1,2-Tetrachloroethane	23.0	5.0	ug/l	25.0	ND	92	65-140	0.04	20	
1,1,2,2-Tetrachloroethane	26.7	2.0	ug/l	25.0	ND	107	55-135	10	30	
Tetrachloroethene	23.5	2.0	ug/l	25.0	ND	94	65-130	1	20	
Toluene	24.6	2.0	ug/l	25.0	ND	99	70-125	0.2	20	
1,2,3-Trichlorobenzene	24.4	5.0	ug/l	25.0	ND	98	60-135	2	20	
1,2,4-Trichlorobenzene	26.3	5.0	ug/l	25.0	ND	105	65-135	0.3	20	
1,1,1-Trichloroethane	23.9	2.0	ug/l	25.0	ND	96	65-140	2	20	
1,1,2-Trichloroethane	25.3	2.0	ug/l	25.0	ND	101	65-130	7	25	
Trichloroethene	23.5	2.0	ug/l	25.0	ND	94	65-125	1	20	
Trichlorofluoromethane	26.2	5.0	ug/l	25.0	ND	105	60-145	0.08	25	
1,2,3-Trichloropropane	25.0	10	ug/l	25.0	ND	100	55-135	9	30	
1,2,4-Trimethylbenzene	59.2	2.0	ug/l	25.0	33.8	102	55-135	2	25	
1,3,5-Trimethylbenzene	27.8	2.0	ug/l	25.0	1.96	104	70-130	2	20	
Vinyl chloride	20.5	5.0	ug/l	25.0	ND	82	45-140	2	30	
m,p-Xylenes	60.5	2.0	ug/l	50.0	7.81	105	65-130	0.3	25	
o-Xylene	26.5	2.0	ug/l	25.0	1.22	101	65-125	2	20	
Surrogate: 4-Bromofluorobenzene	23.6		ug/l	25.0		94	80-120			

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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3081 Extracted: 11/24/10</b>										
<b>Matrix Spike Dup Analyzed: 11/24/2010 (10K3081-MSD1)</b>					<b>Source: ITK2255-07</b>					
Surrogate: Dibromofluoromethane	24.0		ug/l	25.0		96	80-120			
Surrogate: Toluene-d8	24.7		ug/l	25.0		99	80-120			
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>Blank Analyzed: 11/24/2010 (10K3087-BLK1)</b>										
Benzene	ND	2.0	ug/l							
Bromobenzene	ND	5.0	ug/l							
Bromochloromethane	ND	5.0	ug/l							
Bromodichloromethane	ND	2.0	ug/l							
Bromoform	ND	5.0	ug/l							
Bromomethane	ND	5.0	ug/l							
n-Butylbenzene	ND	5.0	ug/l							
sec-Butylbenzene	ND	5.0	ug/l							
tert-Butylbenzene	ND	5.0	ug/l							
Carbon tetrachloride	ND	5.0	ug/l							
Chlorobenzene	ND	2.0	ug/l							
Chloroethane	ND	5.0	ug/l							
Chloroform	ND	2.0	ug/l							
Chloromethane	ND	5.0	ug/l							
2-Chlorotoluene	ND	5.0	ug/l							
4-Chlorotoluene	ND	5.0	ug/l							
1,2-Dibromo-3-chloropropane	ND	5.0	ug/l							
Dibromochloromethane	ND	2.0	ug/l							
1,2-Dibromoethane (EDB)	ND	2.0	ug/l							
Dibromomethane	ND	2.0	ug/l							
1,2-Dichlorobenzene	ND	2.0	ug/l							
1,3-Dichlorobenzene	ND	2.0	ug/l							
1,4-Dichlorobenzene	ND	2.0	ug/l							
Dichlorodifluoromethane	ND	5.0	ug/l							
1,1-Dichloroethane	ND	2.0	ug/l							
1,2-Dichloroethane	ND	2.0	ug/l							
1,1-Dichloroethene	ND	5.0	ug/l							
cis-1,2-Dichloroethene	ND	2.0	ug/l							
trans-1,2-Dichloroethene	ND	2.0	ug/l							
1,2-Dichloropropane	ND	2.0	ug/l							

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Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>Blank Analyzed: 11/24/2010 (10K3087-BLK1)</b>										
1,3-Dichloropropane	ND	2.0	ug/l							
2,2-Dichloropropane	ND	2.0	ug/l							
cis-1,3-Dichloropropene	ND	2.0	ug/l							
trans-1,3-Dichloropropene	ND	2.0	ug/l							
1,1-Dichloropropene	ND	2.0	ug/l							
Ethylbenzene	ND	2.0	ug/l							
Hexachlorobutadiene	ND	5.0	ug/l							
Isopropylbenzene	ND	2.0	ug/l							
p-Isopropyltoluene	ND	2.0	ug/l							
Methylene chloride	ND	5.0	ug/l							
Naphthalene	ND	5.0	ug/l							
n-Propylbenzene	ND	2.0	ug/l							
Styrene	ND	2.0	ug/l							
1,1,1,2-Tetrachloroethane	ND	5.0	ug/l							
1,1,2,2-Tetrachloroethane	ND	2.0	ug/l							
Tetrachloroethene	ND	2.0	ug/l							
Toluene	ND	2.0	ug/l							
1,2,3-Trichlorobenzene	ND	5.0	ug/l							
1,2,4-Trichlorobenzene	ND	5.0	ug/l							
1,1,1-Trichloroethane	ND	2.0	ug/l							
1,1,2-Trichloroethane	ND	2.0	ug/l							
Trichloroethene	ND	2.0	ug/l							
Trichlorofluoromethane	ND	5.0	ug/l							
1,2,3-Trichloropropane	ND	10	ug/l							
1,2,4-Trimethylbenzene	ND	2.0	ug/l							
1,3,5-Trimethylbenzene	ND	2.0	ug/l							
Vinyl chloride	ND	5.0	ug/l							
m,p-Xylenes	ND	2.0	ug/l							
o-Xylene	ND	2.0	ug/l							
Surrogate: 4-Bromofluorobenzene	21.8		ug/l	25.0		87	80-120			
Surrogate: Dibromofluoromethane	24.6		ug/l	25.0		99	80-120			
Surrogate: Toluene-d8	25.2		ug/l	25.0		101	80-120			

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Siemens Water Technologies  
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Project ID: Subpart CC  
 Report Number: ITK1839

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 Received: 11/17/10

## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>LCS Analyzed: 11/24/2010 (10K3087-BS1)</b>										
Benzene	22.3	2.0	ug/l	25.0		89	70-120			
Bromobenzene	22.3	5.0	ug/l	25.0		89	75-120			
Bromochloromethane	21.1	5.0	ug/l	25.0		85	70-130			
Bromodichloromethane	20.4	2.0	ug/l	25.0		82	70-135			
Bromoform	18.9	5.0	ug/l	25.0		76	55-130			
Bromomethane	18.3	5.0	ug/l	25.0		73	65-140			
n-Butylbenzene	24.0	5.0	ug/l	25.0		96	70-130			
sec-Butylbenzene	24.2	5.0	ug/l	25.0		97	70-125			
tert-Butylbenzene	22.9	5.0	ug/l	25.0		92	70-125			
Carbon tetrachloride	19.8	5.0	ug/l	25.0		79	65-140			
Chlorobenzene	20.9	2.0	ug/l	25.0		84	75-120			
Chloroethane	24.1	5.0	ug/l	25.0		97	60-140			
Chloroform	21.0	2.0	ug/l	25.0		84	70-130			
Chloromethane	19.0	5.0	ug/l	25.0		76	50-140			
2-Chlorotoluene	22.4	5.0	ug/l	25.0		90	70-125			
4-Chlorotoluene	22.7	5.0	ug/l	25.0		91	75-125			
1,2-Dibromo-3-chloropropane	19.1	5.0	ug/l	25.0		76	50-135			
Dibromochloromethane	19.2	2.0	ug/l	25.0		77	70-140			
1,2-Dibromoethane (EDB)	20.9	2.0	ug/l	25.0		84	75-125			
Dibromomethane	21.5	2.0	ug/l	25.0		86	70-125			
1,2-Dichlorobenzene	22.4	2.0	ug/l	25.0		90	75-120			
1,3-Dichlorobenzene	23.4	2.0	ug/l	25.0		93	75-120			
1,4-Dichlorobenzene	22.2	2.0	ug/l	25.0		89	75-120			
Dichlorodifluoromethane	18.8	5.0	ug/l	25.0		75	35-155			
1,1-Dichloroethane	21.9	2.0	ug/l	25.0		88	70-125			
1,2-Dichloroethane	21.0	2.0	ug/l	25.0		84	60-140			
1,1-Dichloroethene	22.7	5.0	ug/l	25.0		91	70-125			
cis-1,2-Dichloroethene	23.3	2.0	ug/l	25.0		93	70-125			
trans-1,2-Dichloroethene	23.1	2.0	ug/l	25.0		92	70-125			
1,2-Dichloropropane	22.6	2.0	ug/l	25.0		91	70-125			
1,3-Dichloropropane	21.1	2.0	ug/l	25.0		84	70-120			
2,2-Dichloropropane	25.2	2.0	ug/l	25.0		101	65-140			
cis-1,3-Dichloropropene	22.9	2.0	ug/l	25.0		92	75-125			
trans-1,3-Dichloropropene	25.5	2.0	ug/l	25.0		102	70-125			
1,1-Dichloropropene	21.8	2.0	ug/l	25.0		87	75-130			
Ethylbenzene	22.7	2.0	ug/l	25.0		91	75-125			

**TestAmerica Irvine**

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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>LCS Analyzed: 11/24/2010 (10K3087-BS1)</b>										
Hexachlorobutadiene	18.9	5.0	ug/l	25.0		75	65-135			
Isopropylbenzene	21.9	2.0	ug/l	25.0		88	75-130			
p-Isopropyltoluene	23.5	2.0	ug/l	25.0		94	75-125			
Methylene chloride	20.0	5.0	ug/l	25.0		80	55-130			
Naphthalene	23.5	5.0	ug/l	25.0		94	55-135			
n-Propylbenzene	23.2	2.0	ug/l	25.0		93	75-130			
Styrene	22.8	2.0	ug/l	25.0		91	75-130			
1,1,1,2-Tetrachloroethane	19.9	5.0	ug/l	25.0		80	70-130			
1,1,2,2-Tetrachloroethane	23.8	2.0	ug/l	25.0		95	55-130			
Tetrachloroethene	21.4	2.0	ug/l	25.0		86	70-125			
Toluene	22.5	2.0	ug/l	25.0		90	70-120			
1,2,3-Trichlorobenzene	19.7	5.0	ug/l	25.0		79	65-125			
1,2,4-Trichlorobenzene	19.0	5.0	ug/l	25.0		76	70-135			
1,1,1-Trichloroethane	21.9	2.0	ug/l	25.0		88	65-135			
1,1,2-Trichloroethane	22.0	2.0	ug/l	25.0		88	70-125			
Trichloroethene	21.2	2.0	ug/l	25.0		85	70-125			
Trichlorofluoromethane	23.8	5.0	ug/l	25.0		95	65-145			
1,2,3-Trichloropropane	21.8	10	ug/l	25.0		87	60-130			
1,2,4-Trimethylbenzene	23.8	2.0	ug/l	25.0		95	75-125			
1,3,5-Trimethylbenzene	23.2	2.0	ug/l	25.0		93	75-125			
Vinyl chloride	20.7	5.0	ug/l	25.0		83	55-135			
m,p-Xylenes	47.3	2.0	ug/l	50.0		95	75-125			
o-Xylene	23.2	2.0	ug/l	25.0		93	75-125			
Surrogate: 4-Bromofluorobenzene	23.8		ug/l	25.0		95	80-120			
Surrogate: Dibromofluoromethane	25.3		ug/l	25.0		101	80-120			
Surrogate: Toluene-d8	25.6		ug/l	25.0		103	80-120			

#### Matrix Spike Analyzed: 11/24/2010 (10K3087-MS1)

Source: ITK2261-02

Benzene	22.9	2.0	ug/l	25.0	ND	92	65-125			
Bromobenzene	23.0	5.0	ug/l	25.0	ND	92	70-125			
Bromochloromethane	20.5	5.0	ug/l	25.0	ND	82	65-135			
Bromodichloromethane	20.3	2.0	ug/l	25.0	ND	81	70-135			
Bromoform	18.7	5.0	ug/l	25.0	ND	75	55-135			
Bromomethane	20.3	5.0	ug/l	25.0	ND	81	55-145			
n-Butylbenzene	24.5	5.0	ug/l	25.0	ND	98	65-135			
sec-Butylbenzene	25.1	5.0	ug/l	25.0	ND	100	70-125			

#### TestAmerica Irvine

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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>Matrix Spike Analyzed: 11/24/2010 (10K3087-MS1)</b>					<b>Source: ITK2261-02</b>					
tert-Butylbenzene	23.4	5.0	ug/l	25.0	ND	94	65-130			
Carbon tetrachloride	19.8	5.0	ug/l	25.0	ND	79	65-140			
Chlorobenzene	21.8	2.0	ug/l	25.0	ND	87	75-125			
Chloroethane	24.7	5.0	ug/l	25.0	ND	99	55-140			
Chloroform	20.3	2.0	ug/l	25.0	ND	81	65-135			
Chloromethane	20.8	5.0	ug/l	25.0	ND	83	45-145			
2-Chlorotoluene	23.2	5.0	ug/l	25.0	ND	93	65-135			
4-Chlorotoluene	23.8	5.0	ug/l	25.0	ND	95	70-135			
1,2-Dibromo-3-chloropropane	19.7	5.0	ug/l	25.0	ND	79	45-145			
Dibromochloromethane	19.6	2.0	ug/l	25.0	ND	78	65-140			
1,2-Dibromoethane (EDB)	21.3	2.0	ug/l	25.0	ND	85	70-130			
Dibromomethane	21.4	2.0	ug/l	25.0	ND	86	65-135			
1,2-Dichlorobenzene	23.2	2.0	ug/l	25.0	ND	93	75-125			
1,3-Dichlorobenzene	23.6	2.0	ug/l	25.0	ND	94	75-125			
1,4-Dichlorobenzene	22.5	2.0	ug/l	25.0	ND	90	75-125			
Dichlorodifluoromethane	21.3	5.0	ug/l	25.0	ND	85	25-155			
1,1-Dichloroethane	22.0	2.0	ug/l	25.0	ND	88	65-130			
1,2-Dichloroethane	21.1	2.0	ug/l	25.0	ND	84	60-140			
1,1-Dichloroethene	23.4	5.0	ug/l	25.0	ND	94	60-130			
cis-1,2-Dichloroethene	23.6	2.0	ug/l	25.0	ND	94	65-130			
trans-1,2-Dichloroethene	23.6	2.0	ug/l	25.0	ND	94	65-130			
1,2-Dichloropropane	23.3	2.0	ug/l	25.0	ND	93	65-130			
1,3-Dichloropropane	21.2	2.0	ug/l	25.0	ND	85	65-135			
2,2-Dichloropropane	24.8	2.0	ug/l	25.0	ND	99	60-145			
cis-1,3-Dichloropropene	22.8	2.0	ug/l	25.0	ND	91	70-130			
trans-1,3-Dichloropropene	25.5	2.0	ug/l	25.0	ND	102	65-135			
1,1-Dichloropropene	22.0	2.0	ug/l	25.0	ND	88	70-135			
Ethylbenzene	27.4	2.0	ug/l	25.0	4.10	93	65-130			
Hexachlorobutadiene	19.1	5.0	ug/l	25.0	ND	76	60-135			
Isopropylbenzene	22.7	2.0	ug/l	25.0	ND	91	70-135			
p-Isopropyltoluene	24.0	2.0	ug/l	25.0	ND	96	65-130			
Methylene chloride	19.6	5.0	ug/l	25.0	ND	78	50-135			
Naphthalene	24.8	5.0	ug/l	25.0	0.710	96	50-140			
n-Propylbenzene	23.9	2.0	ug/l	25.0	0.350	94	70-135			
Styrene	2.48	2.0	ug/l	25.0	ND	10	50-145			M2
1,1,1,2-Tetrachloroethane	20.2	5.0	ug/l	25.0	ND	81	65-140			

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 Report Number: ITK1839

Sampled: 11/16/10  
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## METHOD BLANK/QC DATA

### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>Matrix Spike Analyzed: 11/24/2010 (10K3087-MS1)</b>					<b>Source: ITK2261-02</b>					
1,1,2,2-Tetrachloroethane	23.9	2.0	ug/l	25.0	ND	96	55-135			
Tetrachloroethene	21.7	2.0	ug/l	25.0	ND	87	65-130			
Toluene	22.7	2.0	ug/l	25.0	ND	91	70-125			
1,2,3-Trichlorobenzene	19.2	5.0	ug/l	25.0	ND	77	60-135			
1,2,4-Trichlorobenzene	19.6	5.0	ug/l	25.0	ND	79	65-135			
1,1,1-Trichloroethane	21.9	2.0	ug/l	25.0	ND	88	65-140			
1,1,2-Trichloroethane	22.4	2.0	ug/l	25.0	ND	90	65-130			
Trichloroethene	21.6	2.0	ug/l	25.0	ND	86	65-125			
Trichlorofluoromethane	24.3	5.0	ug/l	25.0	ND	97	60-145			
1,2,3-Trichloropropane	21.3	10	ug/l	25.0	ND	85	55-135			
1,2,4-Trimethylbenzene	24.5	2.0	ug/l	25.0	ND	98	55-135			
1,3,5-Trimethylbenzene	23.8	2.0	ug/l	25.0	ND	95	70-130			
Vinyl chloride	21.6	5.0	ug/l	25.0	ND	86	45-140			
m,p-Xylenes	49.7	2.0	ug/l	50.0	ND	99	65-130			
o-Xylene	27.0	2.0	ug/l	25.0	4.17	92	65-125			
Surrogate: 4-Bromofluorobenzene	23.9		ug/l	25.0		96	80-120			
Surrogate: Dibromofluoromethane	24.5		ug/l	25.0		98	80-120			
Surrogate: Toluene-d8	24.9		ug/l	25.0		100	80-120			
<b>Matrix Spike Dup Analyzed: 11/24/2010 (10K3087-MSD1)</b>					<b>Source: ITK2261-02</b>					
Benzene	22.4	2.0	ug/l	25.0	ND	90	65-125	2	20	
Bromobenzene	21.7	5.0	ug/l	25.0	ND	87	70-125	6	20	
Bromochloromethane	20.5	5.0	ug/l	25.0	ND	82	65-135	0.2	25	
Bromodichloromethane	20.0	2.0	ug/l	25.0	ND	80	70-135	2	20	
Bromoform	17.8	5.0	ug/l	25.0	ND	71	55-135	5	25	
Bromomethane	20.4	5.0	ug/l	25.0	ND	82	55-145	0.6	25	
n-Butylbenzene	23.4	5.0	ug/l	25.0	ND	94	65-135	5	20	
sec-Butylbenzene	24.5	5.0	ug/l	25.0	ND	98	70-125	2	20	
tert-Butylbenzene	22.5	5.0	ug/l	25.0	ND	90	65-130	4	20	
Carbon tetrachloride	20.0	5.0	ug/l	25.0	ND	80	65-140	0.8	25	
Chlorobenzene	20.8	2.0	ug/l	25.0	ND	83	75-125	5	20	
Chloroethane	23.5	5.0	ug/l	25.0	ND	94	55-140	5	25	
Chloroform	19.9	2.0	ug/l	25.0	ND	80	65-135	2	20	
Chloromethane	20.7	5.0	ug/l	25.0	ND	83	45-145	0.6	25	
2-Chlorotoluene	22.2	5.0	ug/l	25.0	ND	89	65-135	4	20	
4-Chlorotoluene	22.7	5.0	ug/l	25.0	ND	91	70-135	5	20	

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### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>Matrix Spike Dup Analyzed: 11/24/2010 (10K3087-MSD1)</b>					<b>Source: ITK2261-02</b>					
1,2-Dibromo-3-chloropropane	19.2	5.0	ug/l	25.0	ND	77	45-145	3	30	
Dibromochloromethane	18.1	2.0	ug/l	25.0	ND	72	65-140	8	25	
1,2-Dibromoethane (EDB)	20.3	2.0	ug/l	25.0	ND	81	70-130	5	25	
Dibromomethane	20.7	2.0	ug/l	25.0	ND	83	65-135	3	25	
1,2-Dichlorobenzene	22.1	2.0	ug/l	25.0	ND	89	75-125	5	20	
1,3-Dichlorobenzene	22.9	2.0	ug/l	25.0	ND	92	75-125	3	20	
1,4-Dichlorobenzene	22.0	2.0	ug/l	25.0	ND	88	75-125	2	20	
Dichlorodifluoromethane	20.7	5.0	ug/l	25.0	ND	83	25-155	3	30	
1,1-Dichloroethane	21.4	2.0	ug/l	25.0	ND	86	65-130	3	20	
1,2-Dichloroethane	20.7	2.0	ug/l	25.0	ND	83	60-140	2	20	
1,1-Dichloroethene	22.8	5.0	ug/l	25.0	ND	91	60-130	3	20	
cis-1,2-Dichloroethene	23.0	2.0	ug/l	25.0	ND	92	65-130	2	20	
trans-1,2-Dichloroethene	23.1	2.0	ug/l	25.0	ND	92	65-130	2	20	
1,2-Dichloropropane	22.1	2.0	ug/l	25.0	ND	89	65-130	5	20	
1,3-Dichloropropane	20.5	2.0	ug/l	25.0	ND	82	65-135	3	25	
2,2-Dichloropropane	24.8	2.0	ug/l	25.0	ND	99	60-145	0.1	25	
cis-1,3-Dichloropropene	21.7	2.0	ug/l	25.0	ND	87	70-130	5	20	
trans-1,3-Dichloropropene	25.2	2.0	ug/l	25.0	ND	101	65-135	1	25	
1,1-Dichloropropene	22.1	2.0	ug/l	25.0	ND	89	70-135	0.8	20	
Ethylbenzene	26.1	2.0	ug/l	25.0	4.10	88	65-130	5	20	
Hexachlorobutadiene	18.6	5.0	ug/l	25.0	ND	75	60-135	2	20	
Isopropylbenzene	22.0	2.0	ug/l	25.0	ND	88	70-135	3	20	
p-Isopropyltoluene	23.4	2.0	ug/l	25.0	ND	94	65-130	2	20	
Methylene chloride	19.2	5.0	ug/l	25.0	ND	77	50-135	2	20	
Naphthalene	23.4	5.0	ug/l	25.0	0.710	91	50-140	6	30	
n-Propylbenzene	23.5	2.0	ug/l	25.0	0.350	93	70-135	2	20	
Styrene	0.910	2.0	ug/l	25.0	ND	4	50-145	93	30	M2
1,1,1,2-Tetrachloroethane	18.8	5.0	ug/l	25.0	ND	75	65-140	7	20	
1,1,2,2-Tetrachloroethane	22.4	2.0	ug/l	25.0	ND	90	55-135	6	30	
Tetrachloroethene	21.3	2.0	ug/l	25.0	ND	85	65-130	2	20	
Toluene	22.7	2.0	ug/l	25.0	ND	91	70-125	0.1	20	
1,2,3-Trichlorobenzene	19.2	5.0	ug/l	25.0	ND	77	60-135	0.5	20	
1,2,4-Trichlorobenzene	18.6	5.0	ug/l	25.0	ND	74	65-135	5	20	
1,1,1-Trichloroethane	20.9	2.0	ug/l	25.0	ND	84	65-140	5	20	
1,1,2-Trichloroethane	21.7	2.0	ug/l	25.0	ND	87	65-130	3	25	
Trichloroethene	20.6	2.0	ug/l	25.0	ND	82	65-125	5	20	

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### VOLATILE ORGANICS by GC/MS (EPA 5030B/8260B)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K3087 Extracted: 11/24/10</b>										
<b>Matrix Spike Dup Analyzed: 11/24/2010 (10K3087-MSD1)</b>					<b>Source: ITK2261-02</b>					
Trichlorofluoromethane	23.2	5.0	ug/l	25.0	ND	93	60-145	5	25	
1,2,3-Trichloropropane	20.6	10	ug/l	25.0	ND	83	55-135	3	30	
1,2,4-Trimethylbenzene	23.5	2.0	ug/l	25.0	ND	94	55-135	4	25	
1,3,5-Trimethylbenzene	23.0	2.0	ug/l	25.0	ND	92	70-130	3	20	
Vinyl chloride	21.5	5.0	ug/l	25.0	ND	86	45-140	0.3	30	
m,p-Xylenes	47.9	2.0	ug/l	50.0	ND	96	65-130	4	25	
o-Xylene	25.9	2.0	ug/l	25.0	4.17	87	65-125	4	20	
Surrogate: 4-Bromofluorobenzene	23.7		ug/l	25.0		95	80-120			
Surrogate: Dibromofluoromethane	23.8		ug/l	25.0		95	80-120			
Surrogate: Toluene-d8	25.0		ug/l	25.0		100	80-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Blank Analyzed: 11/23/2010 (10K2349-BLK1)</b>										
Acenaphthene	ND	10	ug/l							
Acenaphthylene	ND	10	ug/l							
Aniline	ND	10	ug/l							
Anthracene	ND	10	ug/l							
Benzidine	ND	20	ug/l							
Benzo(a)anthracene	ND	10	ug/l							
Benzo(a)pyrene	ND	10	ug/l							
Benzo(b)fluoranthene	ND	10	ug/l							
Benzo(g,h,i)perylene	ND	10	ug/l							
Benzo(k)fluoranthene	ND	10	ug/l							
Benzoic acid	ND	20	ug/l							
Benzyl alcohol	ND	20	ug/l							
4-Bromophenyl phenyl ether	ND	10	ug/l							
Butyl benzyl phthalate	ND	20	ug/l							
4-Chloro-3-methylphenol	ND	20	ug/l							
4-Chloroaniline	ND	10	ug/l							
Bis(2-chloroethoxy)methane	ND	10	ug/l							
Bis(2-chloroethyl)ether	ND	10	ug/l							
Bis(2-chloroisopropyl)ether	ND	10	ug/l							
Bis(2-ethylhexyl)phthalate	ND	50	ug/l							
2-Chloronaphthalene	ND	10	ug/l							
2-Chlorophenol	ND	10	ug/l							
4-Chlorophenyl phenyl ether	ND	10	ug/l							
Chrysene	ND	10	ug/l							
Dibenz(a,h)anthracene	ND	20	ug/l							
Dibenzofuran	ND	10	ug/l							
Di-n-butyl phthalate	ND	20	ug/l							
1,2-Dichlorobenzene	ND	10	ug/l							
1,3-Dichlorobenzene	ND	10	ug/l							
1,4-Dichlorobenzene	ND	10	ug/l							
3,3'-Dichlorobenzidine	ND	20	ug/l							
2,4-Dichlorophenol	ND	10	ug/l							
Diethyl phthalate	ND	10	ug/l							
2,4-Dimethylphenol	ND	20	ug/l							
Dimethyl phthalate	ND	10	ug/l							
4,6-Dinitro-2-methylphenol	ND	20	ug/l							

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Blank Analyzed: 11/23/2010 (10K2349-BLK1)</b>										
2,4-Dinitrophenol	ND	20	ug/l							
2,4-Dinitrotoluene	ND	10	ug/l							
2,6-Dinitrotoluene	ND	10	ug/l							
Di-n-octyl phthalate	ND	20	ug/l							
1,2-Diphenylhydrazine/Azobenzene	ND	20	ug/l							
Fluoranthene	ND	10	ug/l							
Fluorene	ND	10	ug/l							
Hexachlorobenzene	ND	10	ug/l							
Hexachlorobutadiene	ND	10	ug/l							
Hexachlorocyclopentadiene	ND	20	ug/l							
Hexachloroethane	ND	10	ug/l							
Indeno(1,2,3-cd)pyrene	ND	20	ug/l							
Isophorone	ND	10	ug/l							
2-Methylnaphthalene	ND	10	ug/l							
2-Methylphenol	ND	10	ug/l							
4-Methylphenol	ND	10	ug/l							
Naphthalene	ND	10	ug/l							
2-Nitroaniline	ND	20	ug/l							
3-Nitroaniline	ND	20	ug/l							
4-Nitroaniline	ND	20	ug/l							
Nitrobenzene	ND	20	ug/l							
2-Nitrophenol	ND	10	ug/l							
4-Nitrophenol	ND	20	ug/l							
N-Nitroso-di-n-propylamine	ND	10	ug/l							
N-Nitrosodiphenylamine	ND	10	ug/l							
Pentachlorophenol	ND	20	ug/l							
Phenanthrene	ND	10	ug/l							
Phenol	ND	10	ug/l							
Pyrene	ND	10	ug/l							
1,2,4-Trichlorobenzene	ND	10	ug/l							
2,4,5-Trichlorophenol	ND	20	ug/l							
2,4,6-Trichlorophenol	ND	20	ug/l							
Surrogate: 2,4,6-Tribromophenol	181		ug/l	200		91	40-120			
Surrogate: 2-Fluorobiphenyl	74.1		ug/l	100		74	50-120			
Surrogate: 2-Fluorophenol	146		ug/l	200		73	30-120			
Surrogate: Nitrobenzene-d5	77.0		ug/l	100		77	45-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Blank Analyzed: 11/23/2010 (10K2349-BLK1)</b>										
Surrogate: Phenol-d6	159		ug/l	200		79	35-120			
Surrogate: Terphenyl-d14	86.7		ug/l	100		87	50-125			
<b>LCS Analyzed: 11/23/2010 (10K2349-BS1)</b>										
Acenaphthene	81.7	10	ug/l	100		82	60-120			
Acenaphthylene	89.7	10	ug/l	100		90	60-120			
Aniline	80.4	10	ug/l	100		80	35-120			
Anthracene	90.8	10	ug/l	100		91	65-120			
Benzidine	122	20	ug/l	100		122	30-160			
Benzo(a)anthracene	89.4	10	ug/l	100		89	65-120			
Benzo(a)pyrene	91.3	10	ug/l	100		91	55-130			
Benzo(b)fluoranthene	89.8	10	ug/l	100		90	55-125			
Benzo(g,h,i)perylene	90.8	10	ug/l	100		91	45-135			
Benzo(k)fluoranthene	92.4	10	ug/l	100		92	50-125			
Benzoic acid	58.4	20	ug/l	100		58	25-120			
Benzyl alcohol	78.2	20	ug/l	100		78	50-120			
4-Bromophenyl phenyl ether	94.3	10	ug/l	100		94	60-120			
Butyl benzyl phthalate	81.4	20	ug/l	100		81	55-130			
4-Chloro-3-methylphenol	98.5	20	ug/l	100		98	60-120			
4-Chloroaniline	82.7	10	ug/l	100		83	55-120			
Bis(2-chloroethoxy)methane	83.9	10	ug/l	100		84	55-120			
Bis(2-chloroethyl)ether	77.1	10	ug/l	100		77	50-120			
Bis(2-chloroisopropyl)ether	64.8	10	ug/l	100		65	45-120			
Bis(2-ethylhexyl)phthalate	97.9	50	ug/l	100		98	65-130			
2-Chloronaphthalene	79.6	10	ug/l	100		80	60-120			
2-Chlorophenol	70.3	10	ug/l	100		70	45-120			
4-Chlorophenyl phenyl ether	102	10	ug/l	100		102	65-120			
Chrysene	87.7	10	ug/l	100		88	65-120			
Dibenz(a,h)anthracene	89.5	20	ug/l	100		90	50-135			
Dibenzofuran	88.7	10	ug/l	100		89	65-120			
Di-n-butyl phthalate	96.5	20	ug/l	100		96	60-125			
1,2-Dichlorobenzene	63.5	10	ug/l	100		64	40-120			
1,3-Dichlorobenzene	56.9	10	ug/l	100		57	35-120			
1,4-Dichlorobenzene	59.7	10	ug/l	100		60	35-120			
3,3'-Dichlorobenzidine	72.0	20	ug/l	100		72	45-135			
2,4-Dichlorophenol	79.3	10	ug/l	100		79	55-120			

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### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>LCS Analyzed: 11/23/2010 (10K2349-BS1)</b>										
Diethyl phthalate	103	10	ug/l	100		103	55-120			
2,4-Dimethylphenol	67.2	20	ug/l	100		67	40-120			
Dimethyl phthalate	96.5	10	ug/l	100		96	30-120			
4,6-Dinitro-2-methylphenol	94.0	20	ug/l	100		94	45-120			
2,4-Dinitrophenol	86.1	20	ug/l	100		86	40-120			
2,4-Dinitrotoluene	105	10	ug/l	100		105	65-120			
2,6-Dinitrotoluene	96.7	10	ug/l	100		97	65-120			
Di-n-octyl phthalate	92.3	20	ug/l	100		92	65-135			
1,2-Diphenylhydrazine/Azobenzene	96.0	20	ug/l	100		96	60-120			
Fluoranthene	88.5	10	ug/l	100		88	60-120			
Fluorene	98.0	10	ug/l	100		98	65-120			
Hexachlorobenzene	92.1	10	ug/l	100		92	60-120			
Hexachlorobutadiene	68.1	10	ug/l	100		68	40-120			
Hexachlorocyclopentadiene	47.6	20	ug/l	100		48	25-120			
Hexachloroethane	56.8	10	ug/l	100		57	35-120			
Indeno(1,2,3-cd)pyrene	88.9	20	ug/l	100		89	45-135			
Isophorone	92.1	10	ug/l	100		92	50-120			
2-Methylnaphthalene	85.1	10	ug/l	100		85	55-120			
2-Methylphenol	77.7	10	ug/l	100		78	50-120			
4-Methylphenol	83.8	10	ug/l	100		84	50-120			
Naphthalene	76.4	10	ug/l	100		76	55-120			
2-Nitroaniline	91.3	20	ug/l	100		91	65-120			
3-Nitroaniline	93.1	20	ug/l	100		93	60-120			
4-Nitroaniline	99.4	20	ug/l	100		99	55-125			
Nitrobenzene	82.1	20	ug/l	100		82	55-120			
2-Nitrophenol	75.4	10	ug/l	100		75	50-120			
4-Nitrophenol	115	20	ug/l	100		115	45-120			
N-Nitroso-di-n-propylamine	93.4	10	ug/l	100		93	45-120			
N-Nitrosodiphenylamine	94.5	10	ug/l	100		95	60-120			
Pentachlorophenol	97.0	20	ug/l	100		97	24-121			
Phenanthrene	90.0	10	ug/l	100		90	65-120			
Phenol	69.6	10	ug/l	100		70	40-120			
Pyrene	93.8	10	ug/l	100		94	55-125			
1,2,4-Trichlorobenzene	68.3	10	ug/l	100		68	45-120			
2,4,5-Trichlorophenol	83.2	20	ug/l	100		83	55-120			
2,4,6-Trichlorophenol	80.8	20	ug/l	100		81	55-120			

**TestAmerica Irvine**

Lena Davidkova  
 Project Manager



Siemens Water Technologies  
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 Attention: Todd Guimond

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Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>LCS Analyzed: 11/23/2010 (10K2349-BS1)</b>										
Surrogate: 2,4,6-Tribromophenol	178		ug/l	200		89	40-120			
Surrogate: 2-Fluorobiphenyl	79.0		ug/l	100		79	50-120			
Surrogate: 2-Fluorophenol	141		ug/l	200		71	30-120			
Surrogate: Nitrobenzene-d5	78.8		ug/l	100		79	45-120			
Surrogate: Phenol-d6	136		ug/l	200		68	35-120			
Surrogate: Terphenyl-d14	90.9		ug/l	100		91	50-125			
<b>Matrix Spike Analyzed: 11/23/2010 (10K2349-MS1)</b>					<b>Source: ITK1710-04</b>					
Acenaphthene	55.6	9.5	ug/l	95.2	ND	58	60-120			M2
Acenaphthylene	47.2	9.5	ug/l	95.2	ND	50	60-120			M2
Aniline	ND	9.5	ug/l	95.2	ND		35-120			M2
Anthracene	67.8	9.5	ug/l	95.2	ND	71	65-120			
Benzidine	ND	19	ug/l	95.2	ND		30-160			M2
Benzo(a)anthracene	80.1	9.5	ug/l	95.2	ND	84	65-120			
Benzo(a)pyrene	71.8	9.5	ug/l	95.2	ND	75	55-130			
Benzo(b)fluoranthene	90.1	9.5	ug/l	95.2	ND	95	55-125			
Benzo(g,h,i)perylene	90.4	9.5	ug/l	95.2	ND	95	45-135			
Benzo(k)fluoranthene	90.9	9.5	ug/l	95.2	ND	95	55-125			
Benzoic acid	63.5	19	ug/l	95.2	ND	67	25-125			
Benzyl alcohol	80.5	19	ug/l	95.2	ND	85	40-120			
4-Bromophenyl phenyl ether	94.6	9.5	ug/l	95.2	ND	99	60-120			
Butyl benzyl phthalate	77.8	19	ug/l	95.2	ND	82	55-130			
4-Chloro-3-methylphenol	37.9	19	ug/l	95.2	ND	40	60-120			M2
4-Chloroaniline	ND	9.5	ug/l	95.2	ND		55-120			M2
Bis(2-chloroethoxy)methane	80.7	9.5	ug/l	95.2	ND	85	50-120			
Bis(2-chloroethyl)ether	72.4	9.5	ug/l	95.2	ND	76	50-120			
Bis(2-chloroisopropyl)ether	75.8	9.5	ug/l	95.2	ND	80	45-120			
Bis(2-ethylhexyl)phthalate	94.2	48	ug/l	95.2	ND	99	65-130			
2-Chloronaphthalene	72.6	9.5	ug/l	95.2	ND	76	60-120			
2-Chlorophenol	50.2	9.5	ug/l	95.2	ND	53	45-120			
4-Chlorophenyl phenyl ether	93.7	9.5	ug/l	95.2	ND	98	65-120			
Chrysene	83.5	9.5	ug/l	95.2	ND	88	65-120			
Dibenz(a,h)anthracene	85.8	19	ug/l	95.2	ND	90	45-135			
Dibenzofuran	81.4	9.5	ug/l	95.2	ND	86	65-120			
Di-n-butyl phthalate	94.6	19	ug/l	95.2	ND	99	60-125			
1,2-Dichlorobenzene	109	9.5	ug/l	95.2	ND	114	40-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Matrix Spike Analyzed: 11/23/2010 (10K2349-MS1)</b>					<b>Source: ITK1710-04</b>					
1,3-Dichlorobenzene	65.3	9.5	ug/l	95.2	ND	69	35-120			
1,4-Dichlorobenzene	62.8	9.5	ug/l	95.2	ND	66	35-120			
3,3'-Dichlorobenzidine	ND	19	ug/l	95.2	ND		45-135			M2
2,4-Dichlorophenol	60.7	9.5	ug/l	95.2	ND	64	55-120			
Diethyl phthalate	94.4	9.5	ug/l	95.2	ND	99	55-120			
2,4-Dimethylphenol	ND	19	ug/l	95.2	ND		40-120			M2
Dimethyl phthalate	89.1	9.5	ug/l	95.2	ND	94	30-120			
4,6-Dinitro-2-methylphenol	104	19	ug/l	95.2	ND	109	45-120			
2,4-Dinitrophenol	75.9	19	ug/l	95.2	ND	80	40-120			
2,4-Dinitrotoluene	95.0	9.5	ug/l	95.2	ND	100	65-120			
2,6-Dinitrotoluene	89.5	9.5	ug/l	95.2	ND	94	65-120			
Di-n-octyl phthalate	90.0	19	ug/l	95.2	ND	95	65-135			
1,2-Diphenylhydrazine/Azobenzene	80.2	19	ug/l	95.2	ND	84	60-120			
Fluoranthene	82.9	9.5	ug/l	95.2	ND	87	60-120			
Fluorene	87.8	9.5	ug/l	95.2	ND	92	65-120			
Hexachlorobenzene	93.8	9.5	ug/l	95.2	ND	99	60-120			
Hexachlorobutadiene	71.4	9.5	ug/l	95.2	ND	75	40-120			
Hexachlorocyclopentadiene	57.0	19	ug/l	95.2	ND	60	25-120			
Hexachloroethane	70.1	9.5	ug/l	95.2	ND	74	35-120			
Indeno(1,2,3-cd)pyrene	86.6	19	ug/l	95.2	ND	91	40-135			
Isophorone	101	9.5	ug/l	95.2	ND	106	50-120			
2-Methylnaphthalene	54.5	9.5	ug/l	95.2	ND	57	55-120			
2-Methylphenol	13.4	9.5	ug/l	95.2	ND	14	50-120			M2
4-Methylphenol	13.1	9.5	ug/l	95.2	ND	14	50-120			M2
Naphthalene	61.0	9.5	ug/l	95.2	ND	64	55-120			
2-Nitroaniline	27.7	19	ug/l	95.2	ND	29	65-120			M2
3-Nitroaniline	4.74	19	ug/l	95.2	ND	5	60-120			M2
4-Nitroaniline	9.35	19	ug/l	95.2	ND	10	55-125			M2
Nitrobenzene	195	19	ug/l	95.2	ND	205	55-120			M1
2-Nitrophenol	88.2	9.5	ug/l	95.2	ND	93	50-120			
4-Nitrophenol	128	19	ug/l	95.2	ND	134	45-120			M1
N-Nitroso-di-n-propylamine	106	9.5	ug/l	95.2	ND	111	45-120			
N-Nitrosodiphenylamine	32.3	9.5	ug/l	95.2	ND	34	60-120			M2
Pentachlorophenol	93.4	19	ug/l	95.2	ND	98	24-121			
Phenanthrene	79.3	9.5	ug/l	95.2	ND	83	65-120			
Phenol	10.6	9.5	ug/l	95.2	ND	11	40-120			M2

TestAmerica Irvine

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Matrix Spike Analyzed: 11/23/2010 (10K2349-MS1)</b>					<b>Source: ITK1710-04</b>					
Pyrene	66.8	9.5	ug/l	95.2	ND	70	55-125			
1,2,4-Trichlorobenzene	70.6	9.5	ug/l	95.2	ND	74	45-120			
2,4,5-Trichlorophenol	73.0	19	ug/l	95.2	ND	77	55-120			
2,4,6-Trichlorophenol	78.1	19	ug/l	95.2	ND	82	55-120			
Surrogate: 2,4,6-Tribromophenol	102		ug/l	190		54	40-120			
Surrogate: 2-Fluorobiphenyl	75.5		ug/l	95.2		79	50-120			
Surrogate: 2-Fluorophenol	59.9		ug/l	190		31	30-120			
Surrogate: Nitrobenzene-d5	87.3		ug/l	95.2		92	45-120			
Surrogate: Phenol-d6	14.3		ug/l	190		8	35-120			Z
Surrogate: Terphenyl-d14	46.6		ug/l	95.2		49	50-125			Z
<b>Matrix Spike Dup Analyzed: 11/23/2010 (10K2349-MSD1)</b>					<b>Source: ITK1710-04</b>					
Acenaphthene	55.9	9.5	ug/l	95.2	ND	59	60-120	0.4	25	M2
Acenaphthylene	54.9	9.5	ug/l	95.2	ND	58	60-120	15	25	M2
Aniline	ND	9.5	ug/l	95.2	ND		35-120		30	M2
Anthracene	63.4	9.5	ug/l	95.2	ND	67	65-120	7	25	
Benzidine	ND	19	ug/l	95.2	ND		30-160		35	M2
Benzo(a)anthracene	78.2	9.5	ug/l	95.2	ND	82	65-120	2	20	
Benzo(a)pyrene	66.0	9.5	ug/l	95.2	ND	69	55-130	8	25	
Benzo(b)fluoranthene	84.0	9.5	ug/l	95.2	ND	88	55-125	7	25	
Benzo(g,h,i)perylene	87.0	9.5	ug/l	95.2	ND	91	45-135	4	30	
Benzo(k)fluoranthene	90.2	9.5	ug/l	95.2	ND	95	55-125	0.7	30	
Benzoic acid	59.2	19	ug/l	95.2	ND	62	25-125	7	30	
Benzyl alcohol	76.2	19	ug/l	95.2	ND	80	40-120	5	30	
4-Bromophenyl phenyl ether	91.0	9.5	ug/l	95.2	ND	96	60-120	4	25	
Butyl benzyl phthalate	75.1	19	ug/l	95.2	ND	79	55-130	4	25	
4-Chloro-3-methylphenol	33.9	19	ug/l	95.2	ND	36	60-120	11	25	M2
4-Chloroaniline	ND	9.5	ug/l	95.2	ND		55-120		25	M2
Bis(2-chloroethoxy)methane	77.1	9.5	ug/l	95.2	ND	81	50-120	5	25	
Bis(2-chloroethyl)ether	72.0	9.5	ug/l	95.2	ND	76	50-120	0.6	25	
Bis(2-chloroisopropyl)ether	59.4	9.5	ug/l	95.2	ND	62	45-120	24	25	
Bis(2-ethylhexyl)phthalate	89.5	48	ug/l	95.2	ND	94	65-130	5	25	
2-Chloronaphthalene	80.8	9.5	ug/l	95.2	ND	85	60-120	11	20	
2-Chlorophenol	46.7	9.5	ug/l	95.2	ND	49	45-120	7	25	
4-Chlorophenyl phenyl ether	90.3	9.5	ug/l	95.2	ND	95	65-120	4	25	
Chrysene	79.5	9.5	ug/l	95.2	ND	83	65-120	5	25	

**TestAmerica Irvine**

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Matrix Spike Dup Analyzed: 11/23/2010 (10K2349-MSD1)</b>					<b>Source: ITK1710-04</b>					
Dibenz(a,h)anthracene	83.0	19	ug/l	95.2	ND	87	45-135	3	30	
Dibenzofuran	78.6	9.5	ug/l	95.2	ND	83	65-120	4	25	
Di-n-butyl phthalate	95.2	19	ug/l	95.2	ND	100	60-125	0.6	25	
1,2-Dichlorobenzene	99.4	9.5	ug/l	95.2	ND	104	40-120	9	25	
1,3-Dichlorobenzene	65.4	9.5	ug/l	95.2	ND	69	35-120	0.1	25	
1,4-Dichlorobenzene	62.9	9.5	ug/l	95.2	ND	66	35-120	0.2	25	
3,3'-Dichlorobenzidine	ND	19	ug/l	95.2	ND		45-135		25	M2
2,4-Dichlorophenol	54.9	9.5	ug/l	95.2	ND	58	55-120	10	25	
Diethyl phthalate	93.8	9.5	ug/l	95.2	ND	98	55-120	0.6	30	
2,4-Dimethylphenol	ND	19	ug/l	95.2	ND		40-120		25	M2
Dimethyl phthalate	99.8	9.5	ug/l	95.2	ND	105	30-120	11	30	
4,6-Dinitro-2-methylphenol	96.6	19	ug/l	95.2	ND	101	45-120	7	25	
2,4-Dinitrophenol	65.1	19	ug/l	95.2	ND	68	40-120	15	25	
2,4-Dinitrotoluene	94.3	9.5	ug/l	95.2	ND	99	65-120	0.7	25	
2,6-Dinitrotoluene	101	9.5	ug/l	95.2	ND	106	65-120	12	20	
Di-n-octyl phthalate	86.6	19	ug/l	95.2	ND	91	65-135	4	20	
1,2-Diphenylhydrazine/Azobenzene	77.8	19	ug/l	95.2	ND	82	60-120	3	25	
Fluoranthene	84.6	9.5	ug/l	95.2	ND	89	60-120	2	25	
Fluorene	85.0	9.5	ug/l	95.2	ND	89	65-120	3	25	
Hexachlorobenzene	89.7	9.5	ug/l	95.2	ND	94	60-120	5	25	
Hexachlorobutadiene	70.7	9.5	ug/l	95.2	ND	74	40-120	1	25	
Hexachlorocyclopentadiene	61.8	19	ug/l	95.2	ND	65	25-120	8	30	
Hexachloroethane	61.7	9.5	ug/l	95.2	ND	65	35-120	13	25	
Indeno(1,2,3-cd)pyrene	81.4	19	ug/l	95.2	ND	85	40-135	6	30	
Isophorone	82.6	9.5	ug/l	95.2	ND	87	50-120	20	25	
2-Methylnaphthalene	63.7	9.5	ug/l	95.2	ND	67	55-120	16	20	
2-Methylphenol	9.77	9.5	ug/l	95.2	ND	10	50-120	32	25	M2, R-3
4-Methylphenol	9.43	9.5	ug/l	95.2	ND	10	50-120	32	25	M2, R-3
Naphthalene	57.5	9.5	ug/l	95.2	ND	60	55-120	6	25	
2-Nitroaniline	29.5	19	ug/l	95.2	ND	31	65-120	6	25	M2
3-Nitroaniline	ND	19	ug/l	95.2	ND		60-120		25	M2
4-Nitroaniline	5.28	19	ug/l	95.2	ND	6	55-125	56	25	M2, R-3
Nitrobenzene	169	19	ug/l	95.2	ND	177	55-120	15	25	M1
2-Nitrophenol	72.2	9.5	ug/l	95.2	ND	76	50-120	20	25	
4-Nitrophenol	111	19	ug/l	95.2	ND	117	45-120	14	30	
N-Nitroso-di-n-propylamine	86.7	9.5	ug/l	95.2	ND	91	45-120	20	25	

**TestAmerica Irvine**

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2349 Extracted: 11/18/10</b>										
<b>Matrix Spike Dup Analyzed: 11/23/2010 (10K2349-MSD1)</b>					<b>Source: ITK1710-04</b>					
N-Nitrosodiphenylamine	11.2	9.5	ug/l	95.2	ND	12	60-120	97	25	M2, R-3
Pentachlorophenol	88.5	19	ug/l	95.2	ND	93	24-121	5	25	
Phenanthrene	77.8	9.5	ug/l	95.2	ND	82	65-120	2	25	
Phenol	7.14	9.5	ug/l	95.2	ND	8	40-120	39	25	M2, R-3
Pyrene	64.7	9.5	ug/l	95.2	ND	68	55-125	3	25	
1,2,4-Trichlorobenzene	68.6	9.5	ug/l	95.2	ND	72	45-120	3	20	
2,4,5-Trichlorophenol	77.3	19	ug/l	95.2	ND	81	55-120	6	30	
2,4,6-Trichlorophenol	83.9	19	ug/l	95.2	ND	88	55-120	7	30	
Surrogate: 2,4,6-Tribromophenol	91.2		ug/l	190		48	40-120			
Surrogate: 2-Fluorobiphenyl	84.0		ug/l	95.2		88	50-120			
Surrogate: 2-Fluorophenol	52.2		ug/l	190		27	30-120			Z
Surrogate: Nitrobenzene-d5	74.9		ug/l	95.2		79	45-120			
Surrogate: Phenol-d6	9.16		ug/l	190		5	35-120			Z
Surrogate: Terphenyl-d14	47.2		ug/l	95.2		50	50-125			

**Batch: 10K2798 Extracted: 11/22/10**

**Blank Analyzed: 11/24/2010 (10K2798-BLK1)**

Acenaphthene	ND	10	ug/l
Acenaphthylene	ND	10	ug/l
Aniline	ND	10	ug/l
Anthracene	ND	10	ug/l
Benzidine	ND	20	ug/l
Benzo(a)anthracene	ND	10	ug/l
Benzo(a)pyrene	ND	10	ug/l
Benzo(b)fluoranthene	ND	10	ug/l
Benzo(g,h,i)perylene	ND	10	ug/l
Benzo(k)fluoranthene	ND	10	ug/l
Benzoic acid	ND	20	ug/l
Benzyl alcohol	ND	20	ug/l
4-Bromophenyl phenyl ether	ND	10	ug/l
Butyl benzyl phthalate	ND	20	ug/l
4-Chloro-3-methylphenol	ND	20	ug/l
4-Chloroaniline	ND	10	ug/l
Bis(2-chloroethoxy)methane	ND	10	ug/l
Bis(2-chloroethyl)ether	ND	10	ug/l

**TestAmerica Irvine**

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 Received: 11/17/10

## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2798 Extracted: 11/22/10</b>										
<b>Blank Analyzed: 11/24/2010 (10K2798-BLK1)</b>										
Bis(2-chloroisopropyl)ether	ND	10	ug/l							
Bis(2-ethylhexyl)phthalate	ND	50	ug/l							
2-Chloronaphthalene	ND	10	ug/l							
2-Chlorophenol	ND	10	ug/l							
4-Chlorophenyl phenyl ether	ND	10	ug/l							
Chrysene	ND	10	ug/l							
Dibenz(a,h)anthracene	ND	20	ug/l							
Dibenzofuran	ND	10	ug/l							
Di-n-butyl phthalate	ND	20	ug/l							
1,2-Dichlorobenzene	ND	10	ug/l							
1,3-Dichlorobenzene	ND	10	ug/l							
1,4-Dichlorobenzene	ND	10	ug/l							
3,3'-Dichlorobenzidine	ND	20	ug/l							
2,4-Dichlorophenol	ND	10	ug/l							
Diethyl phthalate	ND	10	ug/l							
2,4-Dimethylphenol	ND	20	ug/l							
Dimethyl phthalate	ND	10	ug/l							
4,6-Dinitro-2-methylphenol	ND	20	ug/l							
2,4-Dinitrophenol	ND	20	ug/l							
2,4-Dinitrotoluene	ND	10	ug/l							
2,6-Dinitrotoluene	ND	10	ug/l							
Di-n-octyl phthalate	ND	20	ug/l							
1,2-Diphenylhydrazine/Azobenzene	ND	20	ug/l							
Fluoranthene	ND	10	ug/l							
Fluorene	ND	10	ug/l							
Hexachlorobenzene	ND	10	ug/l							
Hexachlorobutadiene	ND	10	ug/l							
Hexachlorocyclopentadiene	ND	20	ug/l							
Hexachloroethane	ND	10	ug/l							
Indeno(1,2,3-cd)pyrene	ND	20	ug/l							
Isophorone	ND	10	ug/l							
2-Methylnaphthalene	ND	10	ug/l							
2-Methylphenol	ND	10	ug/l							
4-Methylphenol	ND	10	ug/l							
Naphthalene	ND	10	ug/l							
2-Nitroaniline	ND	20	ug/l							

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 Parker, AZ 85344  
 Attention: Todd Guimond

Project ID: Subpart CC  
 Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2798 Extracted: 11/22/10</b>										
<b>Blank Analyzed: 11/24/2010 (10K2798-BLK1)</b>										
3-Nitroaniline	ND	20	ug/l							
4-Nitroaniline	ND	20	ug/l							
Nitrobenzene	ND	20	ug/l							
2-Nitrophenol	ND	10	ug/l							
4-Nitrophenol	ND	20	ug/l							
N-Nitroso-di-n-propylamine	ND	10	ug/l							
N-Nitrosodiphenylamine	ND	10	ug/l							
Pentachlorophenol	ND	20	ug/l							
Phenanthrene	ND	10	ug/l							
Phenol	ND	10	ug/l							
Pyrene	ND	10	ug/l							
1,2,4-Trichlorobenzene	ND	10	ug/l							
2,4,5-Trichlorophenol	ND	20	ug/l							
2,4,6-Trichlorophenol	ND	20	ug/l							
Surrogate: 2,4,6-Tribromophenol	162		ug/l	200		81	40-120			
Surrogate: 2-Fluorobiphenyl	72.7		ug/l	100		73	50-120			
Surrogate: 2-Fluorophenol	117		ug/l	200		59	30-120			
Surrogate: Nitrobenzene-d5	67.7		ug/l	100		68	45-120			
Surrogate: Phenol-d6	121		ug/l	200		61	35-120			
Surrogate: Terphenyl-d14	85.7		ug/l	100		86	50-125			
<b>LCS Analyzed: 11/24/2010 (10K2798-BS1)</b>										
Acenaphthene	78.1	10	ug/l	100		78	60-120			MNR1
Acenaphthylene	78.6	10	ug/l	100		79	60-120			
Aniline	64.6	10	ug/l	100		65	35-120			
Anthracene	81.4	10	ug/l	100		81	65-120			
Benzidine	114	20	ug/l	100		114	30-160			
Benzo(a)anthracene	81.6	10	ug/l	100		82	65-120			
Benzo(a)pyrene	86.3	10	ug/l	100		86	55-130			
Benzo(b)fluoranthene	82.0	10	ug/l	100		82	55-125			
Benzo(g,h,i)perylene	80.1	10	ug/l	100		80	45-135			
Benzo(k)fluoranthene	90.7	10	ug/l	100		91	50-125			
Benzoic acid	63.1	20	ug/l	100		63	25-120			
Benzyl alcohol	68.4	20	ug/l	100		68	50-120			
4-Bromophenyl phenyl ether	79.1	10	ug/l	100		79	60-120			
Butyl benzyl phthalate	88.6	20	ug/l	100		89	55-130			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2798 Extracted: 11/22/10</b>										
<b>LCS Analyzed: 11/24/2010 (10K2798-BS1)</b>										
										<b>MNR1</b>
4-Chloro-3-methylphenol	81.5	20	ug/l	100		82	60-120			
4-Chloroaniline	74.2	10	ug/l	100		74	55-120			
Bis(2-chloroethoxy)methane	71.4	10	ug/l	100		71	55-120			
Bis(2-chloroethyl)ether	64.5	10	ug/l	100		64	50-120			
Bis(2-chloroisopropyl)ether	59.0	10	ug/l	100		59	45-120			
Bis(2-ethylhexyl)phthalate	89.3	50	ug/l	100		89	65-130			
2-Chloronaphthalene	69.4	10	ug/l	100		69	60-120			
2-Chlorophenol	62.9	10	ug/l	100		63	45-120			
4-Chlorophenyl phenyl ether	80.9	10	ug/l	100		81	65-120			
Chrysene	82.1	10	ug/l	100		82	65-120			
Dibenz(a,h)anthracene	86.0	20	ug/l	100		86	50-135			
Dibenzofuran	74.9	10	ug/l	100		75	65-120			
Di-n-butyl phthalate	89.0	20	ug/l	100		89	60-125			
1,2-Dichlorobenzene	58.9	10	ug/l	100		59	40-120			
1,3-Dichlorobenzene	55.9	10	ug/l	100		56	35-120			
1,4-Dichlorobenzene	56.6	10	ug/l	100		57	35-120			
3,3'-Dichlorobenzidine	70.5	20	ug/l	100		70	45-135			
2,4-Dichlorophenol	74.1	10	ug/l	100		74	55-120			
Diethyl phthalate	86.0	10	ug/l	100		86	55-120			
2,4-Dimethylphenol	67.6	20	ug/l	100		68	40-120			
Dimethyl phthalate	80.7	10	ug/l	100		81	30-120			
4,6-Dinitro-2-methylphenol	87.2	20	ug/l	100		87	45-120			
2,4-Dinitrophenol	92.4	20	ug/l	100		92	40-120			
2,4-Dinitrotoluene	95.3	10	ug/l	100		95	65-120			
2,6-Dinitrotoluene	85.5	10	ug/l	100		85	65-120			
Di-n-octyl phthalate	98.6	20	ug/l	100		99	65-135			
1,2-Diphenylhydrazine/Azobenzene	76.8	20	ug/l	100		77	60-120			
Fluoranthene	89.2	10	ug/l	100		89	60-120			
Fluorene	80.4	10	ug/l	100		80	65-120			
Hexachlorobenzene	82.2	10	ug/l	100		82	60-120			
Hexachlorobutadiene	60.7	10	ug/l	100		61	40-120			
Hexachlorocyclopentadiene	61.2	20	ug/l	100		61	25-120			
Hexachloroethane	52.2	10	ug/l	100		52	35-120			
Indeno(1,2,3-cd)pyrene	85.6	20	ug/l	100		86	45-135			
Isophorone	73.4	10	ug/l	100		73	50-120			
2-Methylnaphthalene	73.2	10	ug/l	100		73	55-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2798 Extracted: 11/22/10</b>										
<b>LCS Analyzed: 11/24/2010 (10K2798-BS1)</b>										
<b>MNR1</b>										
2-Methylphenol	66.5	10	ug/l	100		66	50-120			
4-Methylphenol	71.1	10	ug/l	100		71	50-120			
Naphthalene	69.0	10	ug/l	100		69	55-120			
2-Nitroaniline	74.8	20	ug/l	100		75	65-120			
3-Nitroaniline	83.9	20	ug/l	100		84	60-120			
4-Nitroaniline	89.5	20	ug/l	100		90	55-125			
Nitrobenzene	67.9	20	ug/l	100		68	55-120			
2-Nitrophenol	71.6	10	ug/l	100		72	50-120			
4-Nitrophenol	83.3	20	ug/l	100		83	45-120			
N-Nitroso-di-n-propylamine	71.4	10	ug/l	100		71	45-120			
N-Nitrosodiphenylamine	79.3	10	ug/l	100		79	60-120			
Pentachlorophenol	88.6	20	ug/l	100		89	24-121			
Phenanthrene	80.3	10	ug/l	100		80	65-120			
Phenol	58.7	10	ug/l	100		59	40-120			
Pyrene	84.8	10	ug/l	100		85	55-125			
1,2,4-Trichlorobenzene	63.4	10	ug/l	100		63	45-120			
2,4,5-Trichlorophenol	76.5	20	ug/l	100		77	55-120			
2,4,6-Trichlorophenol	77.9	20	ug/l	100		78	55-120			
Surrogate: 2,4,6-Tribromophenol	167		ug/l	200		84	40-120			
Surrogate: 2-Fluorobiphenyl	69.7		ug/l	100		70	50-120			
Surrogate: 2-Fluorophenol	104		ug/l	200		52	30-120			
Surrogate: Nitrobenzene-d5	66.4		ug/l	100		66	45-120			
Surrogate: Phenol-d6	114		ug/l	200		57	35-120			
Surrogate: Terphenyl-d14	85.5		ug/l	100		85	50-125			
<b>LCS Dup Analyzed: 11/24/2010 (10K2798-BSD1)</b>										
Acenaphthene	81.9	10	ug/l	100		82	60-120	5	20	
Acenaphthylene	82.4	10	ug/l	100		82	60-120	5	20	
Aniline	68.2	10	ug/l	100		68	35-120	5	30	
Anthracene	84.8	10	ug/l	100		85	65-120	4	20	
Benzidine	114	20	ug/l	100		114	30-160	0.04	35	
Benzo(a)anthracene	82.9	10	ug/l	100		83	65-120	2	20	
Benzo(a)pyrene	88.6	10	ug/l	100		89	55-130	3	25	
Benzo(b)fluoranthene	84.9	10	ug/l	100		85	55-125	3	25	
Benzo(g,h,i)perylene	89.0	10	ug/l	100		89	45-135	10	25	
Benzo(k)fluoranthene	94.0	10	ug/l	100		94	50-125	4	20	

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2798 Extracted: 11/22/10</b>										
<b>LCS Dup Analyzed: 11/24/2010 (10K2798-BSD1)</b>										
Benzoic acid	71.7	20	ug/l	100		72	25-120	13	30	
Benzyl alcohol	73.2	20	ug/l	100		73	50-120	7	20	
4-Bromophenyl phenyl ether	84.8	10	ug/l	100		85	60-120	7	25	
Butyl benzyl phthalate	90.2	20	ug/l	100		90	55-130	2	20	
4-Chloro-3-methylphenol	87.5	20	ug/l	100		88	60-120	7	25	
4-Chloroaniline	79.8	10	ug/l	100		80	55-120	7	25	
Bis(2-chloroethoxy)methane	74.7	10	ug/l	100		75	55-120	4	20	
Bis(2-chloroethyl)ether	68.2	10	ug/l	100		68	50-120	6	20	
Bis(2-chloroisopropyl)ether	63.4	10	ug/l	100		63	45-120	7	20	
Bis(2-ethylhexyl)phthalate	90.2	50	ug/l	100		90	65-130	1	20	
2-Chloronaphthalene	71.5	10	ug/l	100		71	60-120	3	20	
2-Chlorophenol	65.7	10	ug/l	100		66	45-120	4	25	
4-Chlorophenyl phenyl ether	84.8	10	ug/l	100		85	65-120	5	20	
Chrysene	83.8	10	ug/l	100		84	65-120	2	20	
Dibenz(a,h)anthracene	91.3	20	ug/l	100		91	50-135	6	25	
Dibenzofuran	77.9	10	ug/l	100		78	65-120	4	20	
Di-n-butyl phthalate	92.3	20	ug/l	100		92	60-125	4	20	
1,2-Dichlorobenzene	62.3	10	ug/l	100		62	40-120	6	25	
1,3-Dichlorobenzene	57.9	10	ug/l	100		58	35-120	4	25	
1,4-Dichlorobenzene	59.9	10	ug/l	100		60	35-120	6	25	
3,3'-Dichlorobenzidine	72.1	20	ug/l	100		72	45-135	2	25	
2,4-Dichlorophenol	78.3	10	ug/l	100		78	55-120	5	20	
Diethyl phthalate	89.1	10	ug/l	100		89	55-120	4	30	
2,4-Dimethylphenol	67.2	20	ug/l	100		67	40-120	0.6	25	
Dimethyl phthalate	83.9	10	ug/l	100		84	30-120	4	30	
4,6-Dinitro-2-methylphenol	89.8	20	ug/l	100		90	45-120	3	25	
2,4-Dinitrophenol	93.2	20	ug/l	100		93	40-120	0.8	25	
2,4-Dinitrotoluene	96.6	10	ug/l	100		97	65-120	1	20	
2,6-Dinitrotoluene	88.3	10	ug/l	100		88	65-120	3	20	
Di-n-octyl phthalate	99.4	20	ug/l	100		99	65-135	0.8	20	
1,2-Diphenylhydrazine/Azobenzene	79.5	20	ug/l	100		80	60-120	4	25	
Fluoranthene	90.7	10	ug/l	100		91	60-120	2	20	
Fluorene	84.4	10	ug/l	100		84	65-120	5	20	
Hexachlorobenzene	85.7	10	ug/l	100		86	60-120	4	20	
Hexachlorobutadiene	61.9	10	ug/l	100		62	40-120	2	25	
Hexachlorocyclopentadiene	61.0	20	ug/l	100		61	25-120	0.4	30	

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2798 Extracted: 11/22/10</b>										
<b>LCS Dup Analyzed: 11/24/2010 (10K2798-BSD1)</b>										
Hexachloroethane	55.3	10	ug/l	100		55	35-120	6	25	
Indeno(1,2,3-cd)pyrene	88.2	20	ug/l	100		88	45-135	3	25	
Isophorone	78.5	10	ug/l	100		79	50-120	7	20	
2-Methylnaphthalene	78.7	10	ug/l	100		79	55-120	7	20	
2-Methylphenol	70.8	10	ug/l	100		71	50-120	6	20	
4-Methylphenol	76.8	10	ug/l	100		77	50-120	8	20	
Naphthalene	72.4	10	ug/l	100		72	55-120	5	20	
2-Nitroaniline	77.1	20	ug/l	100		77	65-120	3	20	
3-Nitroaniline	85.2	20	ug/l	100		85	60-120	2	25	
4-Nitroaniline	86.0	20	ug/l	100		86	55-125	4	20	
Nitrobenzene	68.8	20	ug/l	100		69	55-120	1	25	
2-Nitrophenol	74.0	10	ug/l	100		74	50-120	3	25	
4-Nitrophenol	83.3	20	ug/l	100		83	45-120	0.02	30	
N-Nitroso-di-n-propylamine	80.3	10	ug/l	100		80	45-120	12	20	
N-Nitrosodiphenylamine	85.5	10	ug/l	100		86	60-120	8	20	
Pentachlorophenol	92.1	20	ug/l	100		92	24-121	4	25	
Phenanthrene	83.8	10	ug/l	100		84	65-120	4	20	
Phenol	61.9	10	ug/l	100		62	40-120	5	25	
Pyrene	90.5	10	ug/l	100		91	55-125	7	25	
1,2,4-Trichlorobenzene	65.6	10	ug/l	100		66	45-120	3	20	
2,4,5-Trichlorophenol	79.2	20	ug/l	100		79	55-120	3	30	
2,4,6-Trichlorophenol	81.5	20	ug/l	100		82	55-120	5	30	
Surrogate: 2,4,6-Tribromophenol	178		ug/l	200		89	40-120			
Surrogate: 2-Fluorobiphenyl	72.0		ug/l	100		72	50-120			
Surrogate: 2-Fluorophenol	103		ug/l	200		52	30-120			
Surrogate: Nitrobenzene-d5	67.9		ug/l	100		68	45-120			
Surrogate: Phenol-d6	118		ug/l	200		59	35-120			
Surrogate: Terphenyl-d14	88.5		ug/l	100		89	50-125			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2443 Extracted: 11/19/10</b>										
<b>Blank Analyzed: 11/19/2010 (10K2443-BLK1)</b>										
4,4'-DDD	ND	0.10	ug/l							
4,4'-DDE	ND	0.10	ug/l							
4,4'-DDT	ND	0.10	ug/l							
Aldrin	ND	0.10	ug/l							
alpha-BHC	ND	0.10	ug/l							
beta-BHC	ND	0.10	ug/l							
delta-BHC	ND	0.20	ug/l							
Dieldrin	ND	0.10	ug/l							
Endosulfan I	ND	0.10	ug/l							
Endosulfan II	ND	0.10	ug/l							
Endosulfan sulfate	ND	0.20	ug/l							
Endrin	ND	0.10	ug/l							
Endrin aldehyde	ND	0.10	ug/l							
Endrin ketone	ND	0.10	ug/l							
gamma-BHC (Lindane)	ND	0.10	ug/l							
Heptachlor	ND	0.10	ug/l							
Heptachlor epoxide	ND	0.10	ug/l							
Methoxychlor	ND	0.10	ug/l							
Chlordane	ND	1.0	ug/l							
Toxaphene	ND	5.0	ug/l							
Surrogate: Decachlorobiphenyl	0.471		ug/l	0.500		94	45-120			
Surrogate: Tetrachloro-m-xylene	0.408		ug/l	0.500		82	35-115			

### LCS Analyzed: 11/22/2010 (10K2443-BS1)

4,4'-DDD	0.520	0.10	ug/l	0.500		104	55-120			
4,4'-DDE	0.515	0.10	ug/l	0.500		103	50-120			
4,4'-DDT	0.614	0.10	ug/l	0.500		123	55-120			L
Aldrin	0.488	0.10	ug/l	0.500		98	40-115			
alpha-BHC	0.473	0.10	ug/l	0.500		95	45-115			
beta-BHC	0.503	0.10	ug/l	0.500		101	55-115			
delta-BHC	0.535	0.20	ug/l	0.500		107	55-115			
Dieldrin	0.519	0.10	ug/l	0.500		104	55-115			
Endosulfan I	0.480	0.10	ug/l	0.500		96	55-115			
Endosulfan II	0.497	0.10	ug/l	0.500		99	55-120			
Endosulfan sulfate	0.556	0.20	ug/l	0.500		111	60-120			
Endrin	0.507	0.10	ug/l	0.500		101	55-115			

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 Attention: Todd Guimond

Project ID: Subpart CC  
 Report Number: ITK1839

Sampled: 11/16/10  
 Received: 11/17/10

## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2443 Extracted: 11/19/10</b>										
<b>LCS Analyzed: 11/22/2010 (10K2443-BS1)</b>										
Endrin aldehyde	0.509	0.10	ug/l	0.500		102	50-120			
Endrin ketone	0.542	0.10	ug/l	0.500		108	55-120			
gamma-BHC (Lindane)	0.482	0.10	ug/l	0.500		96	45-115			
Heptachlor	0.525	0.10	ug/l	0.500		105	45-115			
Heptachlor epoxide	0.483	0.10	ug/l	0.500		97	55-115			
Methoxychlor	0.495	0.10	ug/l	0.500		99	60-120			
Surrogate: Decachlorobiphenyl	0.454		ug/l	0.500		91	45-120			
Surrogate: Tetrachloro-m-xylene	0.405		ug/l	0.500		81	35-115			
<b>Matrix Spike Analyzed: 11/19/2010 (10K2443-MS1)</b>										
<b>Source: ITK1710-04</b>										
4,4'-DDD	0.490	0.094	ug/l	0.472	ND	104	50-125			
4,4'-DDE	0.481	0.094	ug/l	0.472	ND	102	45-125			
4,4'-DDT	0.590	0.094	ug/l	0.472	ND	125	50-125			
Aldrin	0.521	0.094	ug/l	0.472	ND	110	35-120			
alpha-BHC	0.441	0.094	ug/l	0.472	ND	93	40-120			
beta-BHC	0.464	0.094	ug/l	0.472	ND	98	50-120			
delta-BHC	0.490	0.19	ug/l	0.472	ND	104	50-120			
Dieldrin	0.484	0.094	ug/l	0.472	ND	103	50-120			
Endosulfan I	0.443	0.094	ug/l	0.472	ND	94	50-120			
Endosulfan II	0.469	0.094	ug/l	0.472	ND	99	50-125			
Endosulfan sulfate	0.527	0.19	ug/l	0.472	ND	112	55-125			
Endrin	0.476	0.094	ug/l	0.472	ND	101	50-120			
Endrin aldehyde	0.479	0.094	ug/l	0.472	ND	102	45-125			
Endrin ketone	0.495	0.094	ug/l	0.472	ND	105	50-125			
gamma-BHC (Lindane)	0.456	0.094	ug/l	0.472	ND	97	40-120			
Heptachlor	0.531	0.094	ug/l	0.472	ND	113	40-120			
Heptachlor epoxide	0.446	0.094	ug/l	0.472	ND	95	50-120			
Methoxychlor	0.493	0.094	ug/l	0.472	ND	105	55-125			
Surrogate: Decachlorobiphenyl	0.432		ug/l	0.472		92	45-120			
Surrogate: Tetrachloro-m-xylene	0.360		ug/l	0.472		76	35-115			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2443 Extracted: 11/19/10</b>										
<b>Matrix Spike Dup Analyzed: 11/19/2010 (10K2443-MSD1)</b>					<b>Source: ITK1710-04</b>					
4,4'-DDD	0.492	0.094	ug/l	0.472	ND	104	50-125	0.4	30	
4,4'-DDE	0.485	0.094	ug/l	0.472	ND	103	45-125	1	30	
4,4'-DDT	0.591	0.094	ug/l	0.472	ND	125	50-125	0.1	30	
Aldrin	0.529	0.094	ug/l	0.472	ND	112	35-120	2	30	
alpha-BHC	0.443	0.094	ug/l	0.472	ND	94	40-120	0.5	30	
beta-BHC	0.468	0.094	ug/l	0.472	ND	99	50-120	0.8	30	
delta-BHC	0.492	0.19	ug/l	0.472	ND	104	50-120	0.4	30	
Dieldrin	0.488	0.094	ug/l	0.472	ND	103	50-120	0.8	30	
Endosulfan I	0.446	0.094	ug/l	0.472	ND	95	50-120	0.7	30	
Endosulfan II	0.470	0.094	ug/l	0.472	ND	100	50-125	0.3	30	
Endosulfan sulfate	0.521	0.19	ug/l	0.472	ND	111	55-125	1	30	
Endrin	0.478	0.094	ug/l	0.472	ND	101	50-120	0.4	30	
Endrin aldehyde	0.488	0.094	ug/l	0.472	ND	103	45-125	2	30	
Endrin ketone	0.500	0.094	ug/l	0.472	ND	106	50-125	0.9	30	
gamma-BHC (Lindane)	0.459	0.094	ug/l	0.472	ND	97	40-120	0.6	30	
Heptachlor	0.537	0.094	ug/l	0.472	ND	114	40-120	1	30	
Heptachlor epoxide	0.449	0.094	ug/l	0.472	ND	95	50-120	0.7	30	
Methoxychlor	0.494	0.094	ug/l	0.472	ND	105	55-125	0.2	30	
Surrogate: Decachlorobiphenyl	0.432		ug/l	0.472		92	45-120			
Surrogate: Tetrachloro-m-xylene	0.365		ug/l	0.472		77	35-115			

**Batch: 10K2972 Extracted: 11/23/10**

**Blank Analyzed: 11/23/2010 (10K2972-BLK1)**

4,4'-DDD	ND	0.10	ug/l							
4,4'-DDE	ND	0.10	ug/l							
4,4'-DDT	ND	0.10	ug/l							
Aldrin	ND	0.10	ug/l							
alpha-BHC	ND	0.10	ug/l							
beta-BHC	ND	0.10	ug/l							
delta-BHC	ND	0.20	ug/l							
Dieldrin	ND	0.10	ug/l							
Endosulfan I	ND	0.10	ug/l							
Endosulfan II	ND	0.10	ug/l							
Endosulfan sulfate	ND	0.20	ug/l							
Endrin	ND	0.10	ug/l							

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2972 Extracted: 11/23/10</b>										
<b>Blank Analyzed: 11/23/2010 (10K2972-BLK1)</b>										
Endrin aldehyde	ND	0.10	ug/l							
Endrin ketone	ND	0.10	ug/l							
gamma-BHC (Lindane)	ND	0.10	ug/l							
Heptachlor	ND	0.10	ug/l							
Heptachlor epoxide	ND	0.10	ug/l							
Methoxychlor	ND	0.10	ug/l							
Chlordane	ND	1.0	ug/l							
Toxaphene	ND	5.0	ug/l							
Surrogate: Decachlorobiphenyl	0.309		ug/l	0.500		62	45-120			
Surrogate: Tetrachloro-m-xylene	0.390		ug/l	0.500		78	35-115			
<b>LCS Analyzed: 11/23/2010 (10K2972-BS1)</b>										
4,4'-DDD	0.487	0.10	ug/l	0.500		97	55-120			MNR1
4,4'-DDE	0.469	0.10	ug/l	0.500		94	50-120			
4,4'-DDT	0.492	0.10	ug/l	0.500		98	55-120			
Aldrin	0.433	0.10	ug/l	0.500		87	40-115			
alpha-BHC	0.462	0.10	ug/l	0.500		92	45-115			
beta-BHC	0.478	0.10	ug/l	0.500		96	55-115			
delta-BHC	0.500	0.20	ug/l	0.500		100	55-115			
Dieldrin	0.473	0.10	ug/l	0.500		95	55-115			
Endosulfan I	0.475	0.10	ug/l	0.500		95	55-115			
Endosulfan II	0.467	0.10	ug/l	0.500		93	55-120			
Endosulfan sulfate	0.490	0.20	ug/l	0.500		98	60-120			
Endrin	0.507	0.10	ug/l	0.500		101	55-115			
Endrin aldehyde	0.442	0.10	ug/l	0.500		88	50-120			
Endrin ketone	0.444	0.10	ug/l	0.500		89	55-120			
gamma-BHC (Lindane)	0.475	0.10	ug/l	0.500		95	45-115			
Heptachlor	0.480	0.10	ug/l	0.500		96	45-115			
Heptachlor epoxide	0.481	0.10	ug/l	0.500		96	55-115			
Methoxychlor	0.485	0.10	ug/l	0.500		97	60-120			
Surrogate: Decachlorobiphenyl	0.264		ug/l	0.500		53	45-120			
Surrogate: Tetrachloro-m-xylene	0.404		ug/l	0.500		81	35-115			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2972 Extracted: 11/23/10</b>										
<b>LCS Dup Analyzed: 11/23/2010 (10K2972-BSD1)</b>										
4,4'-DDD	0.479	0.10	ug/l	0.500		96	55-120	2	30	
4,4'-DDE	0.460	0.10	ug/l	0.500		92	50-120	2	30	
4,4'-DDT	0.507	0.10	ug/l	0.500		101	55-120	3	30	
Aldrin	0.424	0.10	ug/l	0.500		85	40-115	2	30	
alpha-BHC	0.456	0.10	ug/l	0.500		91	45-115	1	30	
beta-BHC	0.460	0.10	ug/l	0.500		92	55-115	4	30	
delta-BHC	0.492	0.20	ug/l	0.500		98	55-115	2	30	
Dieldrin	0.466	0.10	ug/l	0.500		93	55-115	2	30	
Endosulfan I	0.466	0.10	ug/l	0.500		93	55-115	2	30	
Endosulfan II	0.459	0.10	ug/l	0.500		92	55-120	2	30	
Endosulfan sulfate	0.490	0.20	ug/l	0.500		98	60-120	0.1	30	
Endrin	0.514	0.10	ug/l	0.500		103	55-115	1	30	
Endrin aldehyde	0.437	0.10	ug/l	0.500		87	50-120	1	30	
Endrin ketone	0.448	0.10	ug/l	0.500		90	55-120	0.9	30	
gamma-BHC (Lindane)	0.470	0.10	ug/l	0.500		94	45-115	1	30	
Heptachlor	0.474	0.10	ug/l	0.500		95	45-115	1	30	
Heptachlor epoxide	0.472	0.10	ug/l	0.500		94	55-115	2	30	
Methoxychlor	0.503	0.10	ug/l	0.500		101	60-120	4	30	
Surrogate: Decachlorobiphenyl	0.273		ug/l	0.500		55	45-120			
Surrogate: Tetrachloro-m-xylene	0.398		ug/l	0.500		80	35-115			

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## METHOD BLANK/QC DATA

### POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K2443 Extracted: 11/19/10</b>										
<b>Blank Analyzed: 11/19/2010 (10K2443-BLK1)</b>										
Aroclor 1016	ND	1.0	ug/l							
Aroclor 1221	ND	1.0	ug/l							
Aroclor 1232	ND	1.0	ug/l							
Aroclor 1242	ND	1.0	ug/l							
Aroclor 1248	ND	1.0	ug/l							
Aroclor 1254	ND	1.0	ug/l							
Aroclor 1260	ND	1.0	ug/l							
Surrogate: Decachlorobiphenyl	0.418		ug/l	0.500		84	45-120			
<b>LCS Analyzed: 11/19/2010 (10K2443-BS2)</b>										
Aroclor 1016	3.64	1.0	ug/l	4.00		91	50-115			
Aroclor 1260	3.70	1.0	ug/l	4.00		93	60-120			
Surrogate: Decachlorobiphenyl	0.451		ug/l	0.500		90	45-120			
<b>Matrix Spike Analyzed: 11/19/2010 (10K2443-MS2)</b>					<b>Source: ITK1710-04</b>					
Aroclor 1016	3.36	0.94	ug/l	3.77	ND	89	45-120			
Aroclor 1260	3.39	0.94	ug/l	3.77	ND	90	55-125			
Surrogate: Decachlorobiphenyl	0.385		ug/l	0.472		82	45-120			
<b>Matrix Spike Dup Analyzed: 11/19/2010 (10K2443-MSD2)</b>					<b>Source: ITK1710-04</b>					
Aroclor 1016	3.44	0.94	ug/l	3.77	ND	91	45-120	2	30	
Aroclor 1260	3.44	0.94	ug/l	3.77	ND	91	55-125	2	25	
Surrogate: Decachlorobiphenyl	0.385		ug/l	0.472		82	45-120			
<b>Batch: 10K2972 Extracted: 11/23/10</b>										
<b>Blank Analyzed: 11/23/2010 (10K2972-BLK1)</b>										
Aroclor 1016	ND	1.0	ug/l							
Aroclor 1221	ND	1.0	ug/l							
Aroclor 1232	ND	1.0	ug/l							
Aroclor 1242	ND	1.0	ug/l							
Aroclor 1248	ND	1.0	ug/l							
Aroclor 1254	ND	1.0	ug/l							
Aroclor 1260	ND	1.0	ug/l							
Surrogate: Decachlorobiphenyl	0.351		ug/l	0.500		70	45-120			

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## METHOD BLANK/QC DATA

### POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b><u>Batch: 10K2972 Extracted: 11/23/10</u></b>										
<b>LCS Analyzed: 11/23/2010 (10K2972-BS2)</b>										
Aroclor 1016	3.44	1.0	ug/l	4.00		86	50-115			MNR1
Aroclor 1260	3.76	1.0	ug/l	4.00		94	60-120			
Surrogate: Decachlorobiphenyl	0.358		ug/l	0.500		72	45-120			
<b>LCS Dup Analyzed: 11/23/2010 (10K2972-BSD2)</b>										
Aroclor 1016	3.45	1.0	ug/l	4.00		86	50-115	0.1	30	
Aroclor 1260	3.72	1.0	ug/l	4.00		93	60-120	0.9	25	
Surrogate: Decachlorobiphenyl	0.374		ug/l	0.500		75	45-120			

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## METHOD BLANK/QC DATA

### Alcohols/Gycols by GC

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K1202 Extracted: 12/01/10</b>										
<b>Blank Analyzed: 12/01/2010 (10K1202-BLK1)</b>										
Ethanol	ND	10.0	mg/L							
Isopropyl alcohol	ND	10.0	mg/L							
Methanol	ND	10.0	mg/L							
1-Propanol	ND	10.0	mg/L							
<b>LCS Analyzed: 12/01/2010 (10K1202-BS1)</b>										
Ethanol	274	10.0	mg/L	269		102	80-120			
Isopropyl alcohol	272	10.0	mg/L	267		102	80-120			
Methanol	291	10.0	mg/L	288		101	80-120			
1-Propanol	288	10.0	mg/L	284		101	80-120			
<b>Calibration Check Analyzed: 12/01/2010 (10K1202-CCV1)</b>										
Ethanol	140	NA	mg/L	133		105	80-120			
Isopropyl alcohol	140	NA	mg/L	132		106	80-120			
Methanol	148	NA	mg/L	140		106	80-120			
1-Propanol	146	NA	mg/L	139		105	80-120			
<b>Calibration Check Analyzed: 12/01/2010 (10K1202-CCV2)</b>										
Ethanol	120	NA	mg/L	133		90	80-120			
Isopropyl alcohol	121	NA	mg/L	132		91	80-120			
Methanol	128	NA	mg/L	140		91	80-120			
1-Propanol	124	NA	mg/L	139		89	80-120			
<b>Matrix Spike Analyzed: 12/01/2010 (10K1202-MS1) Source: CTK1181-01</b>										
Ethanol	275	10.0	mg/L	269	ND	102	80-120			
Isopropyl alcohol	272	10.0	mg/L	267	ND	102	80-120			
Methanol	293	10.0	mg/L	288	ND	102	80-120			
1-Propanol	291	10.0	mg/L	284	ND	102	80-120			

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## METHOD BLANK/QC DATA

### Alcohols/Gycols by GC

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 10K1202 Extracted: 12/01/10</b>										
<b>Matrix Spike Dup Analyzed: 12/01/2010 (10K1202-MSD1)</b>					<b>Source: CTK1181-01</b>					
Ethanol	283	10.0	mg/L	269	ND	105	80-120	3	20	
Isopropyl alcohol	278	10.0	mg/L	267	ND	104	80-120	2	20	
Methanol	301	10.0	mg/L	288	ND	104	80-120	3	20	
1-Propanol	299	10.0	mg/L	284	ND	105	80-120	3	20	

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## GC CALIBRATION CHECK CRITERIA

Per Method 8000B of SW-846, the percent recovery of the calibration checks for GC analyses must be within  $\pm 15\%$  from the true value for each individual compound or the average % recovery of all compounds in the calibration check solution must be within  $\pm 15\%$  recovery. Per Method 8000B, the end user is to be notified if the latter situation occurs.

The % recovery for the following individual compounds fell outside the  $\pm 15\%$  criteria, however the average % recovery of all compounds in the calibration check solution was within  $\pm 15\%$ , thus meeting the overall calibration check criteria.

<u>Compound</u>	<u>Footnote</u>	<u>Calibration Check</u> <u>% Recovery</u>	<u>Lab Number</u>	<u>Batch</u>
4,4'-DDT	1	122	ITK1839-01	10K2443
Endrin ketone	2	84	ITK1839-02	10K2972
Endrin ketone	2	84	ITK1839-03	10K2972
Endrin ketone	2	84	ITK1839-04	10K2972

Footnotes:

- 1 The calibration demonstrated a high bias for this compound. Samples were flagged to indicate a possible high bias in the result for this compound.
- 2 The calibration demonstrated a low bias for this compound. Samples were flagged to indicate a possible low bias in the result for this compound.

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## DATA QUALIFIERS AND DEFINITIONS

- C** Calibration Verification recovery was above the method control limit for this analyte. Analyte not detected, data not impacted.
- C-1** Calibration Verification recovery was above the method control limit for this analyte, however the average % difference for all analytes met method criteria. See Calibration Summary form.
- C-2** Calibration Verification recovery was below the method control limit for this analyte, however the average % difference for all analytes met method criteria. See Calibration Summary form.
- L** Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was above the acceptance limits. Analyte not detected, data not impacted.
- M1** The MS and/or MSD were above the acceptance limits due to sample matrix interference. See Blank Spike (LCS).
- M2** The MS and/or MSD were below the acceptance limits due to sample matrix interference. See Blank Spike (LCS).
- MNR1** There was no MS/MSD analyzed with this batch due to insufficient sample volume. See Blank Spike/Blank Spike Duplicate.
- P-HS** Sample container contained headspace.
- R-3** The RPD exceeded the acceptance limit due to sample matrix effects.
- Z** Due to sample matrix effects, the surrogate recovery was below the acceptance limits.
- ND** Analyte NOT DETECTED at or above the reporting limit or MDL, if MDL is specified.
- RPD** Relative Percent Difference

## ADDITIONAL COMMENTS

### For 1,2-Diphenylhydrazine:

The result for 1,2-Diphenylhydrazine is based upon the reading of its breakdown product, Azobenzene.

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Parker, AZ 85344  
Attention: Todd Guimond

Project ID: Subpart CC

Report Number: ITK1839

Sampled: 11/16/10  
Received: 11/17/10

## Certification Summary

### TestAmerica Irvine

Method	Matrix	Nelac	California
EPA 3510C/8081A	Water	X	X
EPA 8082	Water	X	X
EPA 8260B	Water	X	X
EPA 8270C	Water	X	X

*Nevada and NELAP provide analyte specific accreditations. Analyte specific information for TestAmerica may be obtained by contacting the laboratory or visiting our website at [www.testamericainc.com](http://www.testamericainc.com)*

### Subcontracted Laboratories

#### TestAmerica - Cedar Falls, IA

704 Enterprise Drive - Cedar Falls, IA 50613

Method Performed: SW 8015

Samples: ITK1839-01, ITK1839-02, ITK1839-03, ITK1839-04

### TestAmerica Irvine

Lena Davidkova  
Project Manager



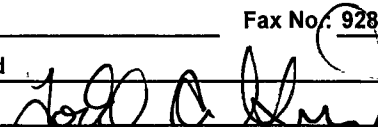




**CHAIN OF CUSTODY RECORD**

Nashville, TN    Dayton, OH    Indianapolis, IN  
 Orlando, FL    Watertown, WI    Irvine, CA  
 Cedar Falls, IA    Pontiac, MI

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

**Client Name/Account #:** SIEMENS WATER TECHNOLOGIES  
**Address:** POB 3308 / 2523 MUTAHAR ST  
**City/State/Zip:** PARKER, AZ 85344  
**Project Manager:** Lena Davidkova  
**Telephone Number:** 928-669-5758   **Fax No.:** 928-669-5775  
**Sampler Name: (Print)** Todd Guimond  
**Sampler Signature:** 

Compliance Monitoring?   Yes   No  
Enforcement Action?   Yes   No

**Report To:** Todd.Guimond@siemens.com

**Invoice To:** \_\_\_\_\_

**TA Quote #:** \_\_\_\_\_

**Project ID:** Subpart CC

**Project #:** ZTK1839

Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Preservative									Matrix				Analyze For:				RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report													
							Ice	HNO <sub>3</sub> (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H <sub>2</sub> SO <sub>4</sub> Plastic (Yellow Label)	H <sub>2</sub> SO <sub>4</sub> Glass (Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Water	Drinking Water	Sludge	Soil	Other (specify):	8260	8015 Alcohol Scan	8270																	
Subpart CC VOA	16-Nov	1030	1																																					
Subpart CC VOA	16-Nov	1030	1																																					
Subpart CC VOA	16-Nov	1030	1																																					
Subpart CC VOA	16-Nov	1030	1																																					
Subpart CC VOA	16-Nov	1030	1																																					
Subpart CC VOA	16-Nov	1030	1																																					
Subpart CC ( A ) 1L AMB	16-Nov	1030	1																																					
Subpart CC ( B ) 1L AMB	16-Nov	1030	1																																					

**Special Instructions:** \* 2 coolers   **Method of Shipment:** UPS

**Laboratory Comments:**  
Temperature Upon Receipt: 4.2 °C  
VOCs Free of Headspace? Y N

Relinquished by:	Date	Time	Received by:	Date	Time
todd guimond	11/16/2010	1400			
Relinquished by:	Date	Time	Received by TestAmerica:	Date	Time
			<i>W Bank</i>	11/17/10	12:00



**CHAIN OF CUSTODY RECORD**

Nashville, TN  
 Orlando, FL  
 Cedar Falls, IA

Dayton, OH  
 Watertown, WI  
 Pontiac, MI

Indianapolis, IN  
 Irvine, CA

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Compliance Monitoring?  Yes  No  
 Enforcement Action?  Yes  No

Client Name/Account #: **SIEMENS WATER TECHNOLOGIES - PARKER / PO# 318570**  
 Address: **POB 3308 / 2523 MUTAHAR ST**

City/State/Zip: **PARKER, AZ 85344**

Project Manager: **Lena Davidkova**

Telephone Number: **928-669-5758**

Fax No.: **928-669-5775**

Sampler Name: (Print) **Todd Guimond**

Sampler Signature:

Report To: **Todd.Guimond@siemens.com**

Invoice To:

TA Quote #:

Project ID: **Subpart CC**

Project #:

**TK1839**

Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Preservative											Matrix				Analyze For:				RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report												
							Ice	HNO <sub>3</sub> (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H <sub>2</sub> SO <sub>4</sub> Plastic (Yellow Label)	H <sub>2</sub> SO <sub>4</sub> Glass (Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Water	Drinking Water	Sludge	Soil	Other (specify):																					
Subpart CC ( C ) 1L AMB	16-Nov	1030	1																																						
Subpart CC ( D ) 1L AMB	16-Nov	1030	1																																						
Subpart CC ( A ) 1L AMB	16-Nov	1030	1																																						
Subpart CC ( B ) 1L AMB	16-Nov	1030	1																																						
Subpart CC ( C ) 1L AMB	16-Nov	1030	1																																						
Subpart CC ( D ) 1L AMB	16-Nov	1030	1																																						
Subpart CC EXTRA	16-Nov	1030	1																																						
Subpart CC EXTRA	16-Nov	1030	1																																						
Subpart CC EXTRA	16-Nov	1030	1																																						
<b>Special Instructions:</b>																																									
Relinquished by:	Date	Time	Received by:															Date	Time	<b>Laboratory Comments:</b> Temperature Upon Receipt: Sample Containers Intact? <input type="checkbox"/> Y <input type="checkbox"/> N																					
todd guimond	11/16/2010	1400																																							
Relinquished by:	Date	Time	Received by TestAmerica:															Date	Time																						

## LABORATORY REPORT

Prepared For: Siemens Industry, Inc.  
P.O. Box 3308 (2523 Mutahar St.) IMA065  
Parker, AZ 85344  
Attention: Monte McCue

Project: Subpart CC  
Subpart CC

Sampled: 11/15/11  
Received: 11/16/11  
Issued: 12/01/11 21:39

NELAP #01108CA California ELAP#2706 CSDLAC #10256 AZ #AZ0671 NV #CA01531

*The results listed within this Laboratory Report pertain only to the samples tested in the laboratory. The analyses contained in this report were performed in accordance with the applicable certifications as noted. All soil samples are reported on a wet weight basis unless otherwise noted in the report. This Laboratory Report is confidential and is intended for the sole use of TestAmerica and its client. This report shall not be reproduced, except in full, without written permission from TestAmerica. The Chain(s) of Custody, 3 pages, are included and are an integral part of this report.*

*This entire report was reviewed and approved for release.*

## SAMPLE CROSS REFERENCE

SUBCONTRACTED: Refer to the last page for specific subcontract laboratory information included in this report.

LABORATORY ID	CLIENT ID	MATRIX
IUK2238-01	Subpart CC #A	Water
IUK2238-02	Subpart CC #B	Water
IUK2238-03	Subpart CC #C	Water
IUK2238-04	Subpart CC #D	Water
IUK2238-05	Subpart CC #E	Water
IUK2238-06	Trip Blank	Water

All Testing Non-Detect for 2011 Samples Taken for Scrubber Water Entering T-11.

Conclusion: T-11 not subject to CC.

Monte McCue 12/2/11

Reviewed By:



TestAmerica Irvine

Sushmitha Reddy  
Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Acenaphthylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Aniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(b)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(g,h,i)perylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(k)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzoic acid	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzyl alcohol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Bromophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Butyl benzyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloro-3-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloroaniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethoxy)methane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroisopropyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-ethylhexyl)phthalate	EPA 8270C	11K2924	48	ND	0.952	11/20/2011	11/27/2011	
2-Chloronaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Chlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Chlorophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Chrysene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Dibenz(a,h)anthracene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dibenzofuran	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-butyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,3-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,4-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
3,3'-Dichlorobenzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dichlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Diethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4-Dimethylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dimethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4,6-Dinitro-2-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,6-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-octyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Fluorene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobutadiene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorocyclopentadiene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Hexachloroethane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Indeno(1,2,3-cd)pyrene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Isophorone	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylnaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Naphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
3-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Nitrobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2-Nitrophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Nitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
N-Nitroso-di-n-propylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
N-Nitrosodiphenylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pentachlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Phenanthrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Phenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,2,4-Trichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4,5-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4,6-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Surrogate: 2,4,6-Tribromophenol (40-120%)								71 %
Surrogate: 2-Fluorobiphenyl (50-120%)								62 %
Surrogate: 2-Fluorophenol (30-120%)								75 %
Surrogate: Nitrobenzene-d5 (45-120%)								73 %
Surrogate: Phenol-d6 (35-120%)								84 %
Surrogate: Terphenyl-d14 (50-125%)								48 %

Z

TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Acenaphthylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Aniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(b)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(g,h,i)perylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(k)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzoic acid	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzyl alcohol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Bromophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Butyl benzyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloro-3-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloroaniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethoxy)methane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroisopropyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-ethylhexyl)phthalate	EPA 8270C	11K2924	48	ND	0.952	11/20/2011	11/27/2011	
2-Chloronaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Chlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Chlorophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Chrysene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Dibenz(a,h)anthracene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dibenzofuran	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-butyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,3-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,4-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
3,3'-Dichlorobenzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dichlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Diethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4-Dimethylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dimethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4,6-Dinitro-2-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,6-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-octyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	

### TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Fluorene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobutadiene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorocyclopentadiene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Hexachloroethane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Indeno(1,2,3-cd)pyrene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Isophorone	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylnaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Naphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
3-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Nitrobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2-Nitrophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Nitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
N-Nitroso-di-n-propylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
N-Nitrosodiphenylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pentachlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Phenanthrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Phenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,2,4-Trichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4,5-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4,6-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Surrogate: 2,4,6-Tribromophenol (40-120%)				89 %				
Surrogate: 2-Fluorobiphenyl (50-120%)				79 %				
Surrogate: 2-Fluorophenol (30-120%)				71 %				
Surrogate: Nitrobenzene-d5 (45-120%)				80 %				
Surrogate: Phenol-d6 (35-120%)				79 %				
Surrogate: Terphenyl-d14 (50-125%)				86 %				

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

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Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Acenaphthylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Aniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(b)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(g,h,i)perylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(k)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzoic acid	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzyl alcohol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Bromophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Butyl benzyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloro-3-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloroaniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethoxy)methane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroisopropyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-ethylhexyl)phthalate	EPA 8270C	11K2924	48	ND	0.952	11/20/2011	11/27/2011	
2-Chloronaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Chlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Chlorophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Chrysene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Dibenz(a,h)anthracene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dibenzofuran	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-butyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,3-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,4-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
3,3'-Dichlorobenzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dichlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Diethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4-Dimethylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dimethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4,6-Dinitro-2-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,6-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-octyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	

### TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

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 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Fluorene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobutadiene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorocyclopentadiene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Hexachloroethane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Indeno(1,2,3-cd)pyrene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Isophorone	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylnaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Naphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
3-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Nitrobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2-Nitrophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Nitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
N-Nitroso-di-n-propylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
N-Nitrosodiphenylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pentachlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Phenanthrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Phenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,2,4-Trichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4,5-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4,6-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Surrogate: 2,4,6-Tribromophenol (40-120%)				81 %				
Surrogate: 2-Fluorobiphenyl (50-120%)				70 %				
Surrogate: 2-Fluorophenol (30-120%)				73 %				
Surrogate: Nitrobenzene-d5 (45-120%)				72 %				
Surrogate: Phenol-d6 (35-120%)				79 %				
Surrogate: Terphenyl-d14 (50-125%)				71 %				

TestAmerica Irvine

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 Project Manager

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 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Acenaphthylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Aniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(b)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(g,h,i)perylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(k)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzoic acid	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzyl alcohol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Bromophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Butyl benzyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloro-3-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloroaniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethoxy)methane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroisopropyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-ethylhexyl)phthalate	EPA 8270C	11K2924	48	ND	0.952	11/20/2011	11/27/2011	
2-Chloronaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Chlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Chlorophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Chrysene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Dibenz(a,h)anthracene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dibenzofuran	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-butyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,3-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,4-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
3,3'-Dichlorobenzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dichlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Diethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4-Dimethylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dimethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4,6-Dinitro-2-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,6-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-octyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	

### TestAmerica Irvine

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 Project Manager

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Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Fluorene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobutadiene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorocyclopentadiene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Hexachloroethane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Indeno(1,2,3-cd)pyrene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Isophorone	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylnaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Naphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
3-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Nitrobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2-Nitrophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Nitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
N-Nitroso-di-n-propylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
N-Nitrosodiphenylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pentachlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Phenanthrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Phenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,2,4-Trichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4,5-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4,6-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Surrogate: 2,4,6-Tribromophenol (40-120%)								82 %
Surrogate: 2-Fluorobiphenyl (50-120%)								74 %
Surrogate: 2-Fluorophenol (30-120%)								75 %
Surrogate: Nitrobenzene-d5 (45-120%)								78 %
Surrogate: Phenol-d6 (35-120%)								87 %
Surrogate: Terphenyl-d14 (50-125%)								63 %

TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water)</b>								
<b>Reporting Units: ug/l</b>								
Acenaphthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Acenaphthylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Aniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)anthracene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(a)pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(b)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(g,h,i)perylene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzo(k)fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Benzoic acid	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Benzyl alcohol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Bromophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Butyl benzyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloro-3-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Chloroaniline	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethoxy)methane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroethyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-chloroisopropyl)ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Bis(2-ethylhexyl)phthalate	EPA 8270C	11K2924	48	ND	0.952	11/20/2011	11/27/2011	
2-Chloronaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Chlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Chlorophenyl phenyl ether	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Chrysene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Dibenz(a,h)anthracene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dibenzofuran	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-butyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,3-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,4-Dichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
3,3'-Dichlorobenzidine	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dichlorophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Diethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4-Dimethylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Dimethyl phthalate	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4,6-Dinitro-2-methylphenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,6-Dinitrotoluene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Di-n-octyl phthalate	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
1,2-Diphenylhydrazine/Azobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water) - cont.</b>								
<b>Reporting Units: ug/l</b>								
Fluoranthene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Fluorene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorobutadiene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Hexachlorocyclopentadiene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Hexachloroethane	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Indeno(1,2,3-cd)pyrene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Isophorone	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylnaphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Methylphenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Naphthalene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
3-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
4-Nitroaniline	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Nitrobenzene	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2-Nitrophenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
4-Nitrophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
N-Nitroso-di-n-propylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
N-Nitrosodiphenylamine	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pentachlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Phenanthrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Phenol	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
Pyrene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
1,2,4-Trichlorobenzene	EPA 8270C	11K2924	9.5	ND	0.952	11/20/2011	11/27/2011	
2,4,5-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
2,4,6-Trichlorophenol	EPA 8270C	11K2924	19	ND	0.952	11/20/2011	11/27/2011	
Surrogate: 2,4,6-Tribromophenol (40-120%)								82 %
Surrogate: 2-Fluorobiphenyl (50-120%)								72 %
Surrogate: 2-Fluorophenol (30-120%)								76 %
Surrogate: Nitrobenzene-d5 (45-120%)								75 %
Surrogate: Phenol-d6 (35-120%)								83 %
Surrogate: Terphenyl-d14 (50-125%)								68 %

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

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Siemens Industry, Inc.

P.O. Box 3308 (2523 Mutahar St.) IMA065

Parker, AZ 85344

Attention: Monte McCue

Project ID: Subpart CC

Subpart CC

Report Number: IUK2238

Sampled: 11/15/11

Received: 11/16/11

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDE	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDT	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Aldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
alpha-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
beta-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
delta-BHC	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Dieldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan I	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan II	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan sulfate	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Endrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin aldehyde	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin ketone	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
gamma-BHC (Lindane)	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor epoxide	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Methoxychlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Chlordane	EPA 8081A	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Toxaphene	EPA 8081A	11K3154	4.8	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)								83 %
Surrogate: Tetrachloro-m-xylene (35-115%)								70 %

TestAmerica Irvine

Sushmitha Reddy  
Project Manager

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IUK2238 <Page 12 of 49>

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDE	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDT	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Aldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
alpha-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
beta-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
delta-BHC	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Dieldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan I	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan II	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan sulfate	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Endrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin aldehyde	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin ketone	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
gamma-BHC (Lindane)	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor epoxide	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Methoxychlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Chlordane	EPA 8081A	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Toxaphene	EPA 8081A	11K3154	4.8	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)								76 %
Surrogate: Tetrachloro-m-xylene (35-115%)								66 %

TestAmerica Irvine

Sushmitha Reddy  
 Project Manager



Siemens Industry, Inc.

P.O. Box 3308 (2523 Mutahar St.) IMA065

Parker, AZ 85344

Attention: Monte McCue

Project ID: Subpart CC

Subpart CC

Report Number: IUK2238

Sampled: 11/15/11

Received: 11/16/11

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDE	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDT	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Aldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
alpha-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
beta-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
delta-BHC	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Dieldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan I	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan II	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan sulfate	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Endrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin aldehyde	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin ketone	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
gamma-BHC (Lindane)	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor epoxide	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Methoxychlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Chlordane	EPA 8081A	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Toxaphene	EPA 8081A	11K3154	4.8	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)								85 %
Surrogate: Tetrachloro-m-xylene (35-115%)								71 %

TestAmerica Irvine

Sushmitha Reddy  
Project Manager

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IUK2238 <Page 14 of 49>

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDE	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDT	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Aldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
alpha-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
beta-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
delta-BHC	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Dieldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan I	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan II	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan sulfate	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Endrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin aldehyde	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin ketone	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
gamma-BHC (Lindane)	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor epoxide	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Methoxychlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Chlordane	EPA 8081A	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Toxaphene	EPA 8081A	11K3154	4.8	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)								83 %
Surrogate: Tetrachloro-m-xylene (35-115%)								73 %

TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water)</b>								
<b>Reporting Units: ug/l</b>								
4,4'-DDD	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDE	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
4,4'-DDT	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Aldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
alpha-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
beta-BHC	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
delta-BHC	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Dieldrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan I	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan II	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endosulfan sulfate	EPA 8081A	11K3154	0.19	ND	0.952	11/22/2011	11/22/2011	
Endrin	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin aldehyde	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Endrin ketone	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
gamma-BHC (Lindane)	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Heptachlor epoxide	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Methoxychlor	EPA 8081A	11K3154	0.095	ND	0.952	11/22/2011	11/22/2011	
Chlordane	EPA 8081A	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Toxaphene	EPA 8081A	11K3154	4.8	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)								79 %
Surrogate: Tetrachloro-m-xylene (35-115%)								62 %

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Siemens Industry, Inc.  
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 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1221	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1232	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1242	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1248	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1254	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1260	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)				75 %				
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1221	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1232	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1242	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1248	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1254	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1260	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)				70 %				
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1221	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1232	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1242	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1248	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1254	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1260	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)				80 %				

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 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1221	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1232	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1242	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1248	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1254	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1260	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)				78 %				
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water)</b>								
Reporting Units: ug/l								
Aroclor 1016	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1221	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1232	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1242	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1248	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1254	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Aroclor 1260	EPA 8082	11K3154	0.95	ND	0.952	11/22/2011	11/22/2011	
Surrogate: Decachlorobiphenyl (45-120%)				73 %				

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 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## Alcohols/Gycols by GC

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Isopropyl alcohol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Methanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
1-Propanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Isopropyl alcohol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Methanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
1-Propanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Isopropyl alcohol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Methanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
1-Propanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Isopropyl alcohol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Methanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
1-Propanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water)</b>								
Reporting Units: mg/L								
Ethanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Isopropyl alcohol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
Methanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	
1-Propanol	SW 8015	11K1333	10.0	ND	1	11/28/2011	11/28/2011	

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Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water)</b>								
<b>Reporting Units: ug/L</b>								
1,1,1,2-Tetrachloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1,1-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2,2-Tetrachloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichloropropane	SW846 8260B	1333134	10	ND	1	11/28/2011	11/28/2011	
1,2,4-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,4-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dibromo-3-chloropropane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2-Dibromoethane (EDB)	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3,5-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,4-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
4-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Benzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromochloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromodichloromethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromoform	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromomethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Carbon tetrachloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chloroform	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
cis-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
cis-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dibromochloromethane	SW846 8260B	1333134	1	ND	1	11/28/2011	11/28/2011	
Dibromomethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dichlorodifluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Ethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Hexachlorobutadiene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	

### TestAmerica Irvine

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Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-01 (Subpart CC #A - Water) - cont.</b>								
<b>Reporting Units: ug/L</b>								
Isopropylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Methylene chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
m-Xylene & p-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Naphthalene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Propylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
o-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
p-Isopropyltoluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
sec-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Styrene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
tert-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Tetrachloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Toluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichlorofluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Vinyl chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Surrogate: 1,2-Dichloroethane-d4 (72-123%)				92 %				
Surrogate: 4-Bromofluorobenzene (74-120%)				83 %				
Surrogate: Dibromofluoromethane (80-123%)				94 %				
Surrogate: Toluene-d8 (78-120%)				104 %				

**TestAmerica Irvine**

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Project ID: Subpart CC  
 Subpart CC  
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Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water)</b>								
<b>Reporting Units: ug/L</b>								
1,1,1,2-Tetrachloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1,1-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2,2-Tetrachloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichloropropane	SW846 8260B	1333134	10	ND	1	11/28/2011	11/28/2011	
1,2,4-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,4-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dibromo-3-chloropropane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2-Dibromoethane (EDB)	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3,5-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,4-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
4-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Benzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromochloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromodichloromethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromoform	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromomethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Carbon tetrachloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chloroform	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
cis-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
cis-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dibromochloromethane	SW846 8260B	1333134	1	ND	1	11/28/2011	11/28/2011	
Dibromomethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dichlorodifluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Ethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Hexachlorobutadiene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	

### TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-02 (Subpart CC #B - Water) - cont.</b>								
<b>Reporting Units: ug/L</b>								
Isopropylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Methylene chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
m-Xylene & p-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Naphthalene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Propylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
o-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
p-Isopropyltoluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
sec-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Styrene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
tert-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Tetrachloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Toluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichlorofluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Vinyl chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Surrogate: 1,2-Dichloroethane-d4 (72-123%)				94 %				
Surrogate: 4-Bromofluorobenzene (74-120%)				83 %				
Surrogate: Dibromofluoromethane (80-123%)				97 %				
Surrogate: Toluene-d8 (78-120%)				105 %				

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water)</b>								
<b>Reporting Units: ug/L</b>								
1,1,1,2-Tetrachloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1,1-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2,2-Tetrachloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichloropropane	SW846 8260B	1333134	10	ND	1	11/28/2011	11/28/2011	
1,2,4-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,4-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dibromo-3-chloropropane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2-Dibromoethane (EDB)	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3,5-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,4-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
4-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Benzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromochloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromodichloromethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromoform	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromomethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Carbon tetrachloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chloroform	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
cis-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
cis-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dibromochloromethane	SW846 8260B	1333134	1	ND	1	11/28/2011	11/28/2011	
Dibromomethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dichlorodifluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Ethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Hexachlorobutadiene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	

### TestAmerica Irvine

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 Project Manager

Siemens Industry, Inc.  
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 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-03 (Subpart CC #C - Water) - cont.</b>								
<b>Reporting Units: ug/L</b>								
Isopropylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Methylene chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
m-Xylene & p-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Naphthalene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Propylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
o-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
p-Isopropyltoluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
sec-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Styrene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
tert-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Tetrachloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Toluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichlorofluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Vinyl chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Surrogate: 1,2-Dichloroethane-d4 (72-123%)				93 %				
Surrogate: 4-Bromofluorobenzene (74-120%)				84 %				
Surrogate: Dibromofluoromethane (80-123%)				98 %				
Surrogate: Toluene-d8 (78-120%)				107 %				

TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

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 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water)</b>								
<b>Reporting Units: ug/L</b>								
1,1,1,2-Tetrachloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1,1-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2,2-Tetrachloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichloropropane	SW846 8260B	1333134	10	ND	1	11/28/2011	11/28/2011	
1,2,4-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,4-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dibromo-3-chloropropane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2-Dibromoethane (EDB)	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3,5-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,4-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
4-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Benzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromochloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromodichloromethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromoform	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromomethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Carbon tetrachloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chloroform	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
cis-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
cis-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dibromochloromethane	SW846 8260B	1333134	1	ND	1	11/28/2011	11/28/2011	
Dibromomethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dichlorodifluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Ethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Hexachlorobutadiene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	

### TestAmerica Irvine

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 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-04 (Subpart CC #D - Water) - cont.</b>								
<b>Reporting Units: ug/L</b>								
Isopropylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Methylene chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
m-Xylene & p-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Naphthalene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Propylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
o-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
p-Isopropyltoluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
sec-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Styrene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
tert-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Tetrachloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Toluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichlorofluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Vinyl chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Surrogate: 1,2-Dichloroethane-d4 (72-123%)				95 %				
Surrogate: 4-Bromofluorobenzene (74-120%)				83 %				
Surrogate: Dibromofluoromethane (80-123%)				99 %				
Surrogate: Toluene-d8 (78-120%)				106 %				

TestAmerica Irvine

Sushmitha Reddy  
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 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water)</b>								
<b>Reporting Units: ug/L</b>								
1,1,1,2-Tetrachloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1,1-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2,2-Tetrachloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1,2-Trichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,1-Dichloroethene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,1-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,3-Trichloropropane	SW846 8260B	1333134	10	ND	1	11/28/2011	11/28/2011	
1,2,4-Trichlorobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2,4-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dibromo-3-chloropropane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
1,2-Dibromoethane (EDB)	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloroethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3,5-Trimethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,3-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
1,4-Dichlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2,2-Dichloropropane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
2-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
4-Chlorotoluene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Benzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromobenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromochloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromodichloromethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Bromoform	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Bromomethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Carbon tetrachloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chlorobenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloroethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Chloroform	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Chloromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
cis-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
cis-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dibromochloromethane	SW846 8260B	1333134	1	ND	1	11/28/2011	11/28/2011	
Dibromomethane	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Dichlorodifluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Ethylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Hexachlorobutadiene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	

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 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## SW846 8260Bx

Analyte	Method	Batch	Reporting Limit	Sample Result	Dilution Factor	Date Extracted	Date Analyzed	Data Qualifiers
<b>Sample ID: IUK2238-05 (Subpart CC #E - Water) - cont.</b>								
<b>Reporting Units: ug/L</b>								
Isopropylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Methylene chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
m-Xylene & p-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Naphthalene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
n-Propylbenzene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
o-Xylene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
p-Isopropyltoluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
sec-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Styrene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
tert-Butylbenzene	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Tetrachloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Toluene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,2-Dichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
trans-1,3-Dichloropropene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichloroethene	SW846 8260B	1333134	2	ND	1	11/28/2011	11/28/2011	
Trichlorofluoromethane	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Vinyl chloride	SW846 8260B	1333134	5	ND	1	11/28/2011	11/28/2011	
Surrogate: 1,2-Dichloroethane-d4 (72-123%)				95 %				
Surrogate: 4-Bromofluorobenzene (74-120%)				83 %				
Surrogate: Dibromofluoromethane (80-123%)				100 %				
Surrogate: Toluene-d8 (78-120%)				106 %				

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>Blank Analyzed: 11/27/2011 (11K2924-BLK1)</b>										
Acenaphthene	ND	10	ug/l							
Acenaphthylene	ND	10	ug/l							
Aniline	ND	10	ug/l							
Anthracene	ND	10	ug/l							
Benzidine	ND	20	ug/l							
Benzo(a)anthracene	ND	10	ug/l							
Benzo(a)pyrene	ND	10	ug/l							
Benzo(b)fluoranthene	ND	10	ug/l							
Benzo(g,h,i)perylene	ND	10	ug/l							
Benzo(k)fluoranthene	ND	10	ug/l							
Benzoic acid	ND	20	ug/l							
Benzyl alcohol	ND	20	ug/l							
4-Bromophenyl phenyl ether	ND	10	ug/l							
Butyl benzyl phthalate	ND	20	ug/l							
4-Chloro-3-methylphenol	ND	20	ug/l							
4-Chloroaniline	ND	10	ug/l							
Bis(2-chloroethoxy)methane	ND	10	ug/l							
Bis(2-chloroethyl)ether	ND	10	ug/l							
Bis(2-chloroisopropyl)ether	ND	10	ug/l							
Bis(2-ethylhexyl)phthalate	ND	50	ug/l							
2-Chloronaphthalene	ND	10	ug/l							
2-Chlorophenol	ND	10	ug/l							
4-Chlorophenyl phenyl ether	ND	10	ug/l							
Chrysene	ND	10	ug/l							
Dibenz(a,h)anthracene	ND	20	ug/l							
Dibenzofuran	ND	10	ug/l							
Di-n-butyl phthalate	ND	20	ug/l							
1,2-Dichlorobenzene	ND	10	ug/l							
1,3-Dichlorobenzene	ND	10	ug/l							
1,4-Dichlorobenzene	ND	10	ug/l							
3,3'-Dichlorobenzidine	ND	20	ug/l							
2,4-Dichlorophenol	ND	10	ug/l							
Diethyl phthalate	ND	10	ug/l							
2,4-Dimethylphenol	ND	20	ug/l							
Dimethyl phthalate	ND	10	ug/l							
4,6-Dinitro-2-methylphenol	ND	20	ug/l							

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>Blank Analyzed: 11/27/2011 (11K2924-BLK1)</b>										
2,4-Dinitrophenol	ND	20	ug/l							
2,4-Dinitrotoluene	ND	10	ug/l							
2,6-Dinitrotoluene	ND	10	ug/l							
Di-n-octyl phthalate	ND	20	ug/l							
1,2-Diphenylhydrazine/Azobenzene	ND	20	ug/l							
Fluoranthene	ND	10	ug/l							
Fluorene	ND	10	ug/l							
Hexachlorobenzene	ND	10	ug/l							
Hexachlorobutadiene	ND	10	ug/l							
Hexachlorocyclopentadiene	ND	20	ug/l							
Hexachloroethane	ND	10	ug/l							
Indeno(1,2,3-cd)pyrene	ND	20	ug/l							
Isophorone	ND	10	ug/l							
2-Methylnaphthalene	ND	10	ug/l							
2-Methylphenol	ND	10	ug/l							
4-Methylphenol	ND	10	ug/l							
Naphthalene	ND	10	ug/l							
2-Nitroaniline	ND	20	ug/l							
3-Nitroaniline	ND	20	ug/l							
4-Nitroaniline	ND	20	ug/l							
Nitrobenzene	ND	20	ug/l							
2-Nitrophenol	ND	10	ug/l							
4-Nitrophenol	ND	20	ug/l							
N-Nitroso-di-n-propylamine	ND	10	ug/l							
N-Nitrosodiphenylamine	ND	10	ug/l							
Pentachlorophenol	ND	20	ug/l							
Phenanthrene	ND	10	ug/l							
Phenol	ND	10	ug/l							
Pyrene	ND	10	ug/l							
1,2,4-Trichlorobenzene	ND	10	ug/l							
2,4,5-Trichlorophenol	ND	20	ug/l							
2,4,6-Trichlorophenol	ND	20	ug/l							
Surrogate: 2,4,6-Tribromophenol	184		ug/l	200		92	40-120			
Surrogate: 2-Fluorobiphenyl	85.7		ug/l	100		86	50-120			
Surrogate: 2-Fluorophenol	164		ug/l	200		82	30-120			
Surrogate: Nitrobenzene-d5	88.7		ug/l	100		89	45-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>Blank Analyzed: 11/27/2011 (11K2924-BLK1)</b>										
Surrogate: Phenol-d6	161		ug/l	200		81	35-120			
Surrogate: Terphenyl-d14	102		ug/l	100		102	50-125			
<b>LCS Analyzed: 11/27/2011 (11K2924-BS1)</b>										
Acenaphthene	79.4	10	ug/l	100		79	60-120			MNR1
Acenaphthylene	89.7	10	ug/l	100		90	60-120			
Aniline	75.4	10	ug/l	100		75	35-120			
Anthracene	83.1	10	ug/l	100		83	65-120			
Benzidine	105	20	ug/l	100		105	30-160			
Benzo(a)anthracene	80.2	10	ug/l	100		80	65-120			
Benzo(a)pyrene	83.0	10	ug/l	100		83	55-130			
Benzo(b)fluoranthene	76.3	10	ug/l	100		76	55-125			
Benzo(g,h,i)perylene	83.6	10	ug/l	100		84	45-135			
Benzo(k)fluoranthene	82.8	10	ug/l	100		83	50-125			
Benzoic acid	57.2	20	ug/l	100		57	25-120			
Benzyl alcohol	79.6	20	ug/l	100		80	50-120			
4-Bromophenyl phenyl ether	78.6	10	ug/l	100		79	60-120			
Butyl benzyl phthalate	79.9	20	ug/l	100		80	55-130			
4-Chloro-3-methylphenol	87.3	20	ug/l	100		87	60-120			
4-Chloroaniline	83.1	10	ug/l	100		83	55-120			
Bis(2-chloroethoxy)methane	78.2	10	ug/l	100		78	55-120			
Bis(2-chloroethyl)ether	76.1	10	ug/l	100		76	50-120			
Bis(2-chloroisopropyl)ether	71.7	10	ug/l	100		72	45-120			
Bis(2-ethylhexyl)phthalate	85.7	50	ug/l	100		86	65-130			
2-Chloronaphthalene	74.3	10	ug/l	100		74	60-120			
2-Chlorophenol	73.0	10	ug/l	100		73	45-120			
4-Chlorophenyl phenyl ether	80.3	10	ug/l	100		80	65-120			
Chrysene	79.7	10	ug/l	100		80	65-120			
Dibenz(a,h)anthracene	87.0	20	ug/l	100		87	50-135			
Dibenzofuran	81.8	10	ug/l	100		82	65-120			
Di-n-butyl phthalate	84.1	20	ug/l	100		84	60-125			
1,2-Dichlorobenzene	60.4	10	ug/l	100		60	40-120			
1,3-Dichlorobenzene	55.1	10	ug/l	100		55	35-120			
1,4-Dichlorobenzene	57.1	10	ug/l	100		57	35-120			
3,3'-Dichlorobenzidine	68.6	20	ug/l	100		69	45-135			
2,4-Dichlorophenol	80.8	10	ug/l	100		81	55-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>LCS Analyzed: 11/27/2011 (11K2924-BS1)</b>										
										<b>MNR1</b>
Diethyl phthalate	82.1	10	ug/l	100		82	55-120			
2,4-Dimethylphenol	77.3	20	ug/l	100		77	40-120			
Dimethyl phthalate	83.8	10	ug/l	100		84	30-120			
4,6-Dinitro-2-methylphenol	79.0	20	ug/l	100		79	45-120			
2,4-Dinitrophenol	76.3	20	ug/l	100		76	40-120			
2,4-Dinitrotoluene	87.0	10	ug/l	100		87	65-120			
2,6-Dinitrotoluene	84.4	10	ug/l	100		84	65-120			
Di-n-octyl phthalate	81.8	20	ug/l	100		82	65-135			
1,2-Diphenylhydrazine/Azobenzene	78.4	20	ug/l	100		78	60-120			
Fluoranthene	83.3	10	ug/l	100		83	60-120			
Fluorene	81.8	10	ug/l	100		82	65-120			
Hexachlorobenzene	79.9	10	ug/l	100		80	60-120			
Hexachlorobutadiene	55.0	10	ug/l	100		55	40-120			
Hexachlorocyclopentadiene	50.9	20	ug/l	100		51	25-120			
Hexachloroethane	50.0	10	ug/l	100		50	35-120			
Indeno(1,2,3-cd)pyrene	93.1	20	ug/l	100		93	45-135			
Isophorone	83.1	10	ug/l	100		83	50-120			
2-Methylnaphthalene	79.6	10	ug/l	100		80	55-120			
2-Methylphenol	77.8	10	ug/l	100		78	50-120			
4-Methylphenol	74.3	10	ug/l	100		74	50-120			
Naphthalene	71.6	10	ug/l	100		72	55-120			
2-Nitroaniline	82.3	20	ug/l	100		82	65-120			
3-Nitroaniline	86.5	20	ug/l	100		86	60-120			
4-Nitroaniline	82.3	20	ug/l	100		82	55-125			
Nitrobenzene	74.8	20	ug/l	100		75	55-120			
2-Nitrophenol	82.3	10	ug/l	100		82	50-120			
4-Nitrophenol	69.6	20	ug/l	100		70	45-120			
N-Nitroso-di-n-propylamine	78.6	10	ug/l	100		79	45-120			
N-Nitrosodiphenylamine	75.9	10	ug/l	100		76	60-120			
Pentachlorophenol	70.0	20	ug/l	100		70	24-121			
Phenanthrene	80.9	10	ug/l	100		81	65-120			
Phenol	70.6	10	ug/l	100		71	40-120			
Pyrene	86.8	10	ug/l	100		87	55-125			
1,2,4-Trichlorobenzene	61.8	10	ug/l	100		62	45-120			
2,4,5-Trichlorophenol	79.0	20	ug/l	100		79	55-120			
2,4,6-Trichlorophenol	80.6	20	ug/l	100		81	55-120			

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>LCS Analyzed: 11/27/2011 (11K2924-BS1)</b>										
Surrogate: 2,4,6-Tribromophenol	178		ug/l	200		89	40-120			MNR1
Surrogate: 2-Fluorobiphenyl	77.8		ug/l	100		78	50-120			
Surrogate: 2-Fluorophenol	125		ug/l	200		62	30-120			
Surrogate: Nitrobenzene-d5	79.3		ug/l	100		79	45-120			
Surrogate: Phenol-d6	135		ug/l	200		67	35-120			
Surrogate: Terphenyl-d14	89.2		ug/l	100		89	50-125			
<b>LCS Dup Analyzed: 11/27/2011 (11K2924-BSD1)</b>										
Acenaphthene	74.8	10	ug/l	100		75	60-120	6	20	
Acenaphthylene	85.4	10	ug/l	100		85	60-120	5	20	
Aniline	79.1	10	ug/l	100		79	35-120	5	30	
Anthracene	75.6	10	ug/l	100		76	65-120	9	20	
Benzidine	117	20	ug/l	100		117	30-160	10	35	
Benzo(a)anthracene	74.4	10	ug/l	100		74	65-120	8	20	
Benzo(a)pyrene	78.1	10	ug/l	100		78	55-130	6	25	
Benzo(b)fluoranthene	72.6	10	ug/l	100		73	55-125	5	25	
Benzo(g,h,i)perylene	80.2	10	ug/l	100		80	45-135	4	25	
Benzo(k)fluoranthene	77.2	10	ug/l	100		77	50-125	7	20	
Benzoic acid	55.9	20	ug/l	100		56	25-120	2	30	
Benzyl alcohol	78.3	20	ug/l	100		78	50-120	2	20	
4-Bromophenyl phenyl ether	74.6	10	ug/l	100		75	60-120	5	25	
Butyl benzyl phthalate	75.2	20	ug/l	100		75	55-130	6	20	
4-Chloro-3-methylphenol	82.6	20	ug/l	100		83	60-120	6	25	
4-Chloroaniline	79.5	10	ug/l	100		80	55-120	4	25	
Bis(2-chloroethoxy)methane	72.7	10	ug/l	100		73	55-120	7	20	
Bis(2-chloroethyl)ether	74.4	10	ug/l	100		74	50-120	2	20	
Bis(2-chloroisopropyl)ether	68.3	10	ug/l	100		68	45-120	5	20	
Bis(2-ethylhexyl)phthalate	82.3	50	ug/l	100		82	65-130	4	20	
2-Chloronaphthalene	70.7	10	ug/l	100		71	60-120	5	20	
2-Chlorophenol	71.7	10	ug/l	100		72	45-120	2	25	
4-Chlorophenyl phenyl ether	74.6	10	ug/l	100		75	65-120	7	20	
Chrysene	75.9	10	ug/l	100		76	65-120	5	20	
Dibenz(a,h)anthracene	82.0	20	ug/l	100		82	50-135	6	25	
Dibenzofuran	76.9	10	ug/l	100		77	65-120	6	20	
Di-n-butyl phthalate	78.1	20	ug/l	100		78	60-125	7	20	
1,2-Dichlorobenzene	57.5	10	ug/l	100		58	40-120	5	25	

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## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>LCS Dup Analyzed: 11/27/2011 (11K2924-BSD1)</b>										
1,3-Dichlorobenzene	53.3	10	ug/l	100		53	35-120	3	25	
1,4-Dichlorobenzene	54.1	10	ug/l	100		54	35-120	5	25	
3,3'-Dichlorobenzidine	62.8	20	ug/l	100		63	45-135	9	25	
2,4-Dichlorophenol	76.7	10	ug/l	100		77	55-120	5	20	
Diethyl phthalate	74.5	10	ug/l	100		74	55-120	10	30	
2,4-Dimethylphenol	73.6	20	ug/l	100		74	40-120	5	25	
Dimethyl phthalate	76.6	10	ug/l	100		77	30-120	9	30	
4,6-Dinitro-2-methylphenol	72.8	20	ug/l	100		73	45-120	8	25	
2,4-Dinitrophenol	67.1	20	ug/l	100		67	40-120	13	25	
2,4-Dinitrotoluene	76.3	10	ug/l	100		76	65-120	13	20	
2,6-Dinitrotoluene	78.2	10	ug/l	100		78	65-120	8	20	
Di-n-octyl phthalate	78.7	20	ug/l	100		79	65-135	4	20	
1,2-Diphenylhydrazine/Azobenzene	71.4	20	ug/l	100		71	60-120	9	25	
Fluoranthene	73.5	10	ug/l	100		74	60-120	12	20	
Fluorene	75.0	10	ug/l	100		75	65-120	9	20	
Hexachlorobenzene	76.1	10	ug/l	100		76	60-120	5	20	
Hexachlorobutadiene	53.4	10	ug/l	100		53	40-120	3	25	
Hexachlorocyclopentadiene	48.5	20	ug/l	100		49	25-120	5	30	
Hexachloroethane	47.6	10	ug/l	100		48	35-120	5	25	
Indeno(1,2,3-cd)pyrene	83.3	20	ug/l	100		83	45-135	11	25	
Isophorone	77.8	10	ug/l	100		78	50-120	7	20	
2-Methylnaphthalene	75.1	10	ug/l	100		75	55-120	6	20	
2-Methylphenol	76.0	10	ug/l	100		76	50-120	2	20	
4-Methylphenol	72.3	10	ug/l	100		72	50-120	3	20	
Naphthalene	67.4	10	ug/l	100		67	55-120	6	20	
2-Nitroaniline	74.2	20	ug/l	100		74	65-120	10	20	
3-Nitroaniline	77.0	20	ug/l	100		77	60-120	12	25	
4-Nitroaniline	71.8	20	ug/l	100		72	55-125	14	20	
Nitrobenzene	71.0	20	ug/l	100		71	55-120	5	25	
2-Nitrophenol	79.4	10	ug/l	100		79	50-120	4	25	
4-Nitrophenol	67.3	20	ug/l	100		67	45-120	3	30	
N-Nitroso-di-n-propylamine	73.8	10	ug/l	100		74	45-120	6	20	
N-Nitrosodiphenylamine	71.0	10	ug/l	100		71	60-120	7	20	
Pentachlorophenol	63.9	20	ug/l	100		64	24-121	9	25	
Phenanthrene	74.7	10	ug/l	100		75	65-120	8	20	
Phenol	71.2	10	ug/l	100		71	40-120	0.8	25	

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Project ID: Subpart CC

Subpart CC

Report Number: IUK2238

Sampled: 11/15/11

Received: 11/16/11

## METHOD BLANK/QC DATA

### SEMI-VOLATILE ORGANICS BY GC/MS (EPA 3520C/8270C)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K2924 Extracted: 11/20/11</b>										
<b>LCS Dup Analyzed: 11/27/2011 (11K2924-BSD1)</b>										
Pyrene	81.5	10	ug/l	100		81	55-125	6	25	
1,2,4-Trichlorobenzene	61.0	10	ug/l	100		61	45-120	1	20	
2,4,5-Trichlorophenol	76.9	20	ug/l	100		77	55-120	3	30	
2,4,6-Trichlorophenol	78.1	20	ug/l	100		78	55-120	3	30	
Surrogate: 2,4,6-Tribromophenol	158		ug/l	200		79	40-120			
Surrogate: 2-Fluorobiphenyl	73.1		ug/l	100		73	50-120			
Surrogate: 2-Fluorophenol	121		ug/l	200		61	30-120			
Surrogate: Nitrobenzene-d5	73.7		ug/l	100		74	45-120			
Surrogate: Phenol-d6	134		ug/l	200		67	35-120			
Surrogate: Terphenyl-d14	82.6		ug/l	100		83	50-125			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K3154 Extracted: 11/22/11</b>										
<b>Blank Analyzed: 11/22/2011 (11K3154-BLK1)</b>										
4,4'-DDD	ND	0.10	ug/l							
4,4'-DDE	ND	0.10	ug/l							
4,4'-DDT	ND	0.10	ug/l							
Aldrin	ND	0.10	ug/l							
alpha-BHC	ND	0.10	ug/l							
beta-BHC	ND	0.10	ug/l							
delta-BHC	ND	0.20	ug/l							
Dieldrin	ND	0.10	ug/l							
Endosulfan I	ND	0.10	ug/l							
Endosulfan II	ND	0.10	ug/l							
Endosulfan sulfate	ND	0.20	ug/l							
Endrin	ND	0.10	ug/l							
Endrin aldehyde	ND	0.10	ug/l							
Endrin ketone	ND	0.10	ug/l							
gamma-BHC (Lindane)	ND	0.10	ug/l							
Heptachlor	ND	0.10	ug/l							
Heptachlor epoxide	ND	0.10	ug/l							
Methoxychlor	ND	0.10	ug/l							
Chlordane	ND	1.0	ug/l							
Toxaphene	ND	5.0	ug/l							
Surrogate: Decachlorobiphenyl	0.425		ug/l	0.500		85	45-120			
Surrogate: Tetrachloro-m-xylene	0.358		ug/l	0.500		72	35-115			

### LCS Analyzed: 11/22/2011 (11K3154-BS1)

MNR1

4,4'-DDD	0.435	0.10	ug/l	0.500		87	55-120			
4,4'-DDE	0.428	0.10	ug/l	0.500		86	50-120			
4,4'-DDT	0.447	0.10	ug/l	0.500		89	55-120			
Aldrin	0.416	0.10	ug/l	0.500		83	40-115			
alpha-BHC	0.441	0.10	ug/l	0.500		88	45-115			
beta-BHC	0.409	0.10	ug/l	0.500		82	55-115			
delta-BHC	0.448	0.20	ug/l	0.500		90	55-115			
Dieldrin	0.430	0.10	ug/l	0.500		86	55-115			
Endosulfan I	0.414	0.10	ug/l	0.500		83	55-115			
Endosulfan II	0.426	0.10	ug/l	0.500		85	55-120			
Endosulfan sulfate	0.453	0.20	ug/l	0.500		91	60-120			
Endrin	0.467	0.10	ug/l	0.500		93	55-115			

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## METHOD BLANK/QC DATA

### ORGANOCHLORINE PESTICIDES (EPA 3510C/8081A)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC %REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K3154 Extracted: 11/22/11</b>										
<b>LCS Analyzed: 11/22/2011 (11K3154-BS1)</b>										
Endrin aldehyde	0.399	0.10	ug/l	0.500		80	50-120			MNR1
Endrin ketone	0.447	0.10	ug/l	0.500		89	55-120			
gamma-BHC (Lindane)	0.436	0.10	ug/l	0.500		87	45-115			
Heptachlor	0.424	0.10	ug/l	0.500		85	45-115			
Heptachlor epoxide	0.416	0.10	ug/l	0.500		83	55-115			
Methoxychlor	0.448	0.10	ug/l	0.500		90	60-120			
Surrogate: Decachlorobiphenyl	0.432		ug/l	0.500		86	45-120			
Surrogate: Tetrachloro-m-xylene	0.409		ug/l	0.500		82	35-115			
<b>LCS Dup Analyzed: 11/22/2011 (11K3154-BSD1)</b>										
4,4'-DDD	0.438	0.10	ug/l	0.500		88	55-120	0.8	30	
4,4'-DDE	0.430	0.10	ug/l	0.500		86	50-120	0.5	30	
4,4'-DDT	0.450	0.10	ug/l	0.500		90	55-120	0.7	30	
Aldrin	0.411	0.10	ug/l	0.500		82	40-115	1	30	
alpha-BHC	0.443	0.10	ug/l	0.500		89	45-115	0.5	30	
beta-BHC	0.428	0.10	ug/l	0.500		86	55-115	5	30	
delta-BHC	0.462	0.20	ug/l	0.500		92	55-115	3	30	
Dieldrin	0.431	0.10	ug/l	0.500		86	55-115	0.4	30	
Endosulfan I	0.416	0.10	ug/l	0.500		83	55-115	0.5	30	
Endosulfan II	0.426	0.10	ug/l	0.500		85	55-120	0.09	30	
Endosulfan sulfate	0.454	0.20	ug/l	0.500		91	60-120	0.2	30	
Endrin	0.468	0.10	ug/l	0.500		94	55-115	0.3	30	
Endrin aldehyde	0.409	0.10	ug/l	0.500		82	50-120	2	30	
Endrin ketone	0.453	0.10	ug/l	0.500		91	55-120	1	30	
gamma-BHC (Lindane)	0.445	0.10	ug/l	0.500		89	45-115	2	30	
Heptachlor	0.446	0.10	ug/l	0.500		89	45-115	5	30	
Heptachlor epoxide	0.417	0.10	ug/l	0.500		83	55-115	0.2	30	
Methoxychlor	0.446	0.10	ug/l	0.500		89	60-120	0.4	30	
Surrogate: Decachlorobiphenyl	0.431		ug/l	0.500		86	45-120			
Surrogate: Tetrachloro-m-xylene	0.412		ug/l	0.500		82	35-115			

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## METHOD BLANK/QC DATA

### POLYCHLORINATED BIPHENYLS (EPA 3510C/8082)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K3154 Extracted: 11/22/11</b>										
<b>Blank Analyzed: 11/22/2011 (11K3154-BLK1)</b>										
Aroclor 1016	ND	1.0	ug/l							
Aroclor 1221	ND	1.0	ug/l							
Aroclor 1232	ND	1.0	ug/l							
Aroclor 1242	ND	1.0	ug/l							
Aroclor 1248	ND	1.0	ug/l							
Aroclor 1254	ND	1.0	ug/l							
Aroclor 1260	ND	1.0	ug/l							
Surrogate: Decachlorobiphenyl	0.391		ug/l	0.500		78	45-120			
<b>LCS Analyzed: 11/22/2011 (11K3154-BS2)</b>										
Aroclor 1016	3.27	1.0	ug/l	4.00		82	50-115			MNR1
Aroclor 1260	3.10	1.0	ug/l	4.00		77	60-120			
Surrogate: Decachlorobiphenyl	0.381		ug/l	0.500		76	45-120			
<b>LCS Dup Analyzed: 11/22/2011 (11K3154-BSD2)</b>										
Aroclor 1016	3.35	1.0	ug/l	4.00		84	50-115	2	30	
Aroclor 1260	3.20	1.0	ug/l	4.00		80	60-120	3	25	
Surrogate: Decachlorobiphenyl	0.404		ug/l	0.500		81	45-120			

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Sampled: 11/15/11  
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## METHOD BLANK/QC DATA

### Alcohols/Gycols by GC

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K1333 Extracted: 11/28/11</b>										
<b>Blank Analyzed: 11/28/2011 (11K1333-BLK1)</b>										
Ethanol	ND	10.0	mg/L							
Isopropyl alcohol	ND	10.0	mg/L							
Methanol	ND	10.0	mg/L							
1-Propanol	ND	10.0	mg/L							
<b>LCS Analyzed: 11/28/2011 (11K1333-BS1)</b>										
Ethanol	83.9	10.0	mg/L	93.8		89	80-120			
Isopropyl alcohol	75.3	10.0	mg/L	87.2		86	80-120			
Methanol	88.1	10.0	mg/L	98.8		89	80-120			
1-Propanol	87.9	10.0	mg/L	98.8		89	80-120			
<b>Calibration Check Analyzed: 11/28/2011 (11K1333-CCV1)</b>										
Ethanol	81.8	NA	mg/L	93.8		87	80-120			
Isopropyl alcohol	74.7	NA	mg/L	87.2		86	80-120			
Methanol	84.6	NA	mg/L	98.8		86	80-120			
1-Propanol	86.0	NA	mg/L	98.8		87	80-120			
<b>Calibration Check Analyzed: 11/28/2011 (11K1333-CCV2)</b>										
Ethanol	88.1	NA	mg/L	93.8		94	80-120			
Isopropyl alcohol	80.3	NA	mg/L	87.2		92	80-120			
Methanol	90.5	NA	mg/L	98.8		92	80-120			
1-Propanol	92.8	NA	mg/L	98.8		94	80-120			
<b>Calibration Check Analyzed: 11/28/2011 (11K1333-CCV3)</b>										
Ethanol	88.6	NA	mg/L	93.8		94	80-120			
Isopropyl alcohol	81.0	NA	mg/L	87.2		93	80-120			
Methanol	91.8	NA	mg/L	98.8		93	80-120			
1-Propanol	93.1	NA	mg/L	98.8		94	80-120			

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## METHOD BLANK/QC DATA

### Alcohols/Gycols by GC

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 11K1333 Extracted: 11/28/11</b>										
<b>Matrix Spike Analyzed: 11/28/2011 (11K1333-MS1)</b>					<b>Source: CUK1207-06</b>					
Ethanol	85.1	10.0	mg/L	93.8	ND	91	80-120			
Isopropyl alcohol	78.1	10.0	mg/L	87.2	ND	90	80-120			
Methanol	87.6	10.0	mg/L	98.8	1.05	88	80-120			
1-Propanol	89.8	10.0	mg/L	98.8	ND	91	80-120			
<b>Matrix Spike Dup Analyzed: 11/28/2011 (11K1333-MSD1)</b>					<b>Source: CUK1207-06</b>					
Ethanol	87.5	10.0	mg/L	93.8	ND	93	80-120	3	20	
Isopropyl alcohol	79.0	10.0	mg/L	87.2	ND	91	80-120	1	20	
Methanol	92.4	10.0	mg/L	98.8	1.05	92	80-120	5	20	
1-Propanol	91.7	10.0	mg/L	98.8	ND	93	80-120	2	20	

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## METHOD BLANK/QC DATA

### SW846 8260Bx

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 1333134 Extracted: 11/28/11</b>										
<b>Blank Analyzed: 11/28/2011 (G1K290000134B)</b>					<b>Source:</b>					
1,1,1,2-Tetrachloroethane	ND	5	ug/L				-			
1,1,1-Trichloroethane	ND	2	ug/L				-			
1,1,2,2-Tetrachloroethane	ND	2	ug/L				-			
1,1,2-Trichloroethane	ND	2	ug/L				-			
1,1-Dichloroethane	ND	2	ug/L				-			
1,1-Dichloroethene	ND	5	ug/L				-			
1,1-Dichloropropene	ND	2	ug/L				-			
1,2,3-Trichlorobenzene	ND	5	ug/L				-			
1,2,3-Trichloropropane	ND	10	ug/L				-			
1,2,4-Trichlorobenzene	ND	5	ug/L				-			
1,2,4-Trimethylbenzene	ND	2	ug/L				-			
1,2-Dibromo-3-chloropropane	ND	5	ug/L				-			
1,2-Dibromoethane (EDB)	ND	2	ug/L				-			
1,2-Dichlorobenzene	ND	2	ug/L				-			
1,2-Dichloroethane	ND	2	ug/L				-			
1,2-Dichloropropane	ND	2	ug/L				-			
1,3,5-Trimethylbenzene	ND	2	ug/L				-			
1,3-Dichlorobenzene	ND	2	ug/L				-			
1,3-Dichloropropane	ND	2	ug/L				-			
1,4-Dichlorobenzene	ND	2	ug/L				-			
2,2-Dichloropropane	ND	2	ug/L				-			
2-Chlorotoluene	ND	5	ug/L				-			
4-Chlorotoluene	ND	5	ug/L				-			
Benzene	ND	2	ug/L				-			
Bromobenzene	ND	5	ug/L				-			
Bromochloromethane	ND	5	ug/L				-			
Bromodichloromethane	ND	2	ug/L				-			
Bromoform	ND	5	ug/L				-			
Bromomethane	ND	5	ug/L				-			
Carbon tetrachloride	ND	5	ug/L				-			
Chlorobenzene	ND	2	ug/L				-			
Chloroethane	ND	5	ug/L				-			
Chloroform	ND	2	ug/L				-			
Chloromethane	ND	5	ug/L				-			
cis-1,2-Dichloroethene	ND	2	ug/L				-			
cis-1,3-Dichloropropene	ND	2	ug/L				-			

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## METHOD BLANK/QC DATA

### SW846 8260Bx

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 1333134 Extracted: 11/28/11</b>										
<b>Blank Analyzed: 11/28/2011 (G1K290000134B)</b>					<b>Source:</b>					
Dibromochloromethane	ND	1	ug/L				-			
Dibromomethane	ND	2	ug/L				-			
Dichlorodifluoromethane	ND	5	ug/L				-			
Ethylbenzene	ND	2	ug/L				-			
Hexachlorobutadiene	ND	5	ug/L				-			
Isopropylbenzene	ND	2	ug/L				-			
Methylene chloride	ND	5	ug/L				-			
m-Xylene & p-Xylene	ND	2	ug/L				-			
Naphthalene	ND	5	ug/L				-			
n-Butylbenzene	ND	5	ug/L				-			
n-Propylbenzene	ND	2	ug/L				-			
o-Xylene	ND	2	ug/L				-			
p-Isopropyltoluene	ND	2	ug/L				-			
sec-Butylbenzene	ND	5	ug/L				-			
Styrene	ND	2	ug/L				-			
tert-Butylbenzene	ND	5	ug/L				-			
Tetrachloroethene	ND	2	ug/L				-			
Toluene	ND	2	ug/L				-			
trans-1,2-Dichloroethene	ND	2	ug/L				-			
trans-1,3-Dichloropropene	ND	2	ug/L				-			
Trichloroethene	ND	2	ug/L				-			
Trichlorofluoromethane	ND	5	ug/L				-			
Vinyl chloride	ND	5	ug/L				-			
Surrogate: 1,2-Dichloroethane-d4	19		ug/L	20		94	72-123			
Surrogate: 4-Bromofluorobenzene	17		ug/L	20		85	74-120			
Surrogate: Dibromofluoromethane	19		ug/L	20		97	80-123			
Surrogate: Toluene-d8	21		ug/L	20		105	78-120			

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## METHOD BLANK/QC DATA

### SW846 8260Bx

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 1333134 Extracted: 11/28/11</b>										
<b>LCS Analyzed: 11/28/2011 (G1K290000134C)</b>					<b>Source:</b>					
1,1,1,2-Tetrachloroethane	18.7	1	ug/L	20		93	79-120			
1,1,1-Trichloroethane	18.8	1	ug/L	20		94	79-121			
1,1,2,2-Tetrachloroethane	18.2	1	ug/L	20		91	74-137			
1,1,2-Trichloroethane	21.3	1	ug/L	20		106	79-127			
1,1-Dichloroethane	17.3	1	ug/L	20		86	79-120			
1,1-Dichloroethene	19.1	1	ug/L	20		96	74-120			
1,1-Dichloropropene	18.7	1	ug/L	20		94	77-120			
1,2,3-Trichlorobenzene	19.4	1	ug/L	20		97	47-162			
1,2,3-Trichloropropane	21.6	1	ug/L	20		108	73-120			
1,2,4-Trichlorobenzene	19	1	ug/L	20		95	61-130			
1,2,4-Trimethylbenzene	18.7	1	ug/L	20		93	76-120			
1,2-Dibromo-3-chloropropane	18.2	2	ug/L	20		91	66-121			
1,2-Dibromoethane (EDB)	21.5	2	ug/L	20		108	78-120			
1,2-Dichlorobenzene	19.5	1	ug/L	20		97	77-120			
1,2-Dichloroethane	17.6	1	ug/L	20		88	77-128			
1,2-Dichloropropane	17.9	1	ug/L	20		90	75-125			
1,3,5-Trimethylbenzene	18.8	1	ug/L	20		94	79-120			
1,3-Dichlorobenzene	19.3	1	ug/L	20		96	78-120			
1,3-Dichloropropane	19.2	1	ug/L	20		96	79-120			
1,4-Dichlorobenzene	18.7	1	ug/L	20		94	74-120			
2,2-Dichloropropane	15.4	1	ug/L	20		77	75-127			
2-Chlorotoluene	18.3	1	ug/L	20		92	79-120			
4-Chlorotoluene	18.1	1	ug/L	20		91	80-121			
Benzene	19.1	1	ug/L	20		95	79-120			
Bromobenzene	20.4	1	ug/L	20		102	80-120			
Bromochloromethane	20.6	1	ug/L	20		103	80-120			
Bromodichloromethane	18	1	ug/L	20		90	80-124			
Bromoform	19.1	1	ug/L	20		95	80-120			
Bromomethane	15.2	1	ug/L	20		76	65-132			
Carbon tetrachloride	18.7	1	ug/L	20		93	78-124			
Chlorobenzene	19.9	1	ug/L	20		100	78-120			
Chloroethane	15.7	1	ug/L	20		79	65-123			
Chloroform	18.9	1	ug/L	20		94	80-120			
Chloromethane	15.4	1	ug/L	20		77	62-129			
cis-1,2-Dichloroethene	18.8	1	ug/L	20		94	78-120			
cis-1,3-Dichloropropene	18.9	1	ug/L	20		95	80-131			

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
P.O. Box 3308 (2523 Mutahar St.) IMA065  
Parker, AZ 85344  
Attention: Monte McCue

Project ID: Subpart CC  
Subpart CC  
Report Number: IUK2238

Sampled: 11/15/11  
Received: 11/16/11

## METHOD BLANK/QC DATA

### SW846 8260Bx

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 1333134 Extracted: 11/28/11</b>										
<b>LCS Analyzed: 11/28/2011 (G1K290000134C)</b>					<b>Source:</b>					
Dibromochloromethane	20.2	1	ug/L	20		101	80-122			
Dibromomethane	19.7	1	ug/L	20		98	80-121			
Dichlorodifluoromethane	20.1	1	ug/L	20		101	39-161			
Ethylbenzene	19.8	1	ug/L	20		99	80-120			
Hexachlorobutadiene	19.4	1	ug/L	20		97	69-120			
Isopropylbenzene	18.6	1	ug/L	20		93	80-121			
Methylene chloride	19.1	1	ug/L	20		96	77-120			
m-Xylene & p-Xylene	39.1	1	ug/L	40		98	80-121			
Naphthalene	19.6	1	ug/L	20		98	56-143			
n-Butylbenzene	17.7	1	ug/L	20		89	72-120			
n-Propylbenzene	18.8	1	ug/L	20		94	76-120			
o-Xylene	19.3	1	ug/L	20		96	80-124			
p-Isopropyltoluene	18.8	1	ug/L	20		94	76-120			
sec-Butylbenzene	18.6	1	ug/L	20		93	77-120			
Styrene	19.4	1	ug/L	20		97	80-120			
tert-Butylbenzene	18.8	1	ug/L	20		94	78-120			
Tetrachloroethene	21.2	1	ug/L	20		106	74-120			
Toluene	20.4	1	ug/L	20		102	79-126			
trans-1,2-Dichloroethene	20.1	1	ug/L	20		100	76-120			
trans-1,3-Dichloropropene	18.9	1	ug/L	20		94	75-133			
Trichloroethene	19.7	1	ug/L	20		99	74-120			
Trichlorofluoromethane	20.2	1	ug/L	20		101	60-135			
Vinyl chloride	16.2	1	ug/L	20		81	68-121			
Surrogate: 1,2-Dichloroethane-d4	17.6		ug/L	20		88	72-123			
Surrogate: 4-Bromofluorobenzene	18		ug/L	20		90	74-120			
Surrogate: Dibromofluoromethane	19.1		ug/L	20		96	80-123			
Surrogate: Toluene-d8	20.6		ug/L	20		103	78-120			

TestAmerica Irvine

Sushmitha Reddy  
Project Manager



Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## METHOD BLANK/QC DATA

### SW846 8260Bx

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 1333134 Extracted: 11/28/11</b>										
<b>LCS Dup Analyzed: 11/28/2011 (G1K290000134L)</b>					<b>Source:</b>					
1,1,1,2-Tetrachloroethane	18.3	1	ug/L	20		92	79-120	1.8	23	
1,1,1-Trichloroethane	19.3	1	ug/L	20		96	79-121	2.5	25	
1,1,2,2-Tetrachloroethane	17.9	1	ug/L	20		89	74-137	1.7	27	
1,1,2-Trichloroethane	21.9	1	ug/L	20		109	79-127	2.8	30	
1,1-Dichloroethane	17.9	1	ug/L	20		89	79-120	3.4	21	
1,1-Dichloroethene	19.2	1	ug/L	20		96	74-120	0.53	22	
1,1-Dichloropropene	19.3	1	ug/L	20		96	77-120	2.8	20	
1,2,3-Trichlorobenzene	20.2	1	ug/L	20		101	47-162	3.7	45	
1,2,3-Trichloropropane	20.8	1	ug/L	20		104	73-120	3.9	22	
1,2,4-Trichlorobenzene	18.8	1	ug/L	20		94	61-130	1	40	
1,2,4-Trimethylbenzene	18.4	1	ug/L	20		92	76-120	1.6	17	
1,2-Dibromo-3-chloropropane	18.4	2	ug/L	20		92	66-121	0.78	33	
1,2-Dibromoethane (EDB)	21.1	2	ug/L	20		105	78-120	2.2	15	
1,2-Dichlorobenzene	19.3	1	ug/L	20		96	77-120	0.98	19	
1,2-Dichloroethane	17.7	1	ug/L	20		88	77-128	0.48	25	
1,2-Dichloropropane	18.6	1	ug/L	20		93	75-125	3.7	27	
1,3,5-Trimethylbenzene	18.6	1	ug/L	20		93	79-120	1.1	20	
1,3-Dichlorobenzene	19.2	1	ug/L	20		96	78-120	0.27	17	
1,3-Dichloropropane	19	1	ug/L	20		95	79-120	1.2	15	
1,4-Dichlorobenzene	18.6	1	ug/L	20		93	74-120	0.63	15	
2,2-Dichloropropane	16	1	ug/L	20		80	75-127	3.6	25	
2-Chlorotoluene	18.1	1	ug/L	20		90	79-120	1.4	19	
4-Chlorotoluene	17.9	1	ug/L	20		89	80-121	1.4	19	
Benzene	19.5	1	ug/L	20		97	79-120	2.2	21	
Bromobenzene	20.6	1	ug/L	20		103	80-120	1.1	17	
Bromochloromethane	21.3	1	ug/L	20		106	80-120	3.2	19	
Bromodichloromethane	18.7	1	ug/L	20		94	80-124	3.8	20	
Bromoform	18.5	1	ug/L	20		92	80-120	3.4	16	
Bromomethane	15.4	1	ug/L	20		77	65-132	1.5	40	
Carbon tetrachloride	18.9	1	ug/L	20		95	78-124	1.2	25	
Chlorobenzene	19.7	1	ug/L	20		98	78-120	1.2	15	
Chloroethane	15.3	1	ug/L	20		76	65-123	2.8	40	
Chloroform	19.3	1	ug/L	20		97	80-120	2.1	22	
Chloromethane	15.8	1	ug/L	20		79	62-129	2.9	25	
cis-1,2-Dichloroethene	19.3	1	ug/L	20		97	78-120	2.6	18	
cis-1,3-Dichloropropene	19.8	1	ug/L	20		99	80-131	4.5	24	

**TestAmerica Irvine**

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
 P.O. Box 3308 (2523 Mutahar St.) IMA065  
 Parker, AZ 85344  
 Attention: Monte McCue

Project ID: Subpart CC  
 Subpart CC  
 Report Number: IUK2238

Sampled: 11/15/11  
 Received: 11/16/11

## METHOD BLANK/QC DATA

### SW846 8260Bx

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Data Qualifiers
<b>Batch: 1333134 Extracted: 11/28/11</b>										
<b>LCS Dup Analyzed: 11/28/2011 (G1K290000134L)</b>					<b>Source:</b>					
Dibromochloromethane	20	1	ug/L	20		100	80-122	1.3	17	
Dibromomethane	19.9	1	ug/L	20		99	80-121	0.84	17	
Dichlorodifluoromethane	20.3	1	ug/L	20		101	39-161	0.77	51	
Ethylbenzene	19.7	1	ug/L	20		98	80-120	0.68	15	
Hexachlorobutadiene	19.5	1	ug/L	20		97	69-120	0.66	30	
Isopropylbenzene	18.5	1	ug/L	20		93	80-121	0.46	17	
Methylene chloride	19.2	1	ug/L	20		96	77-120	0.41	20	
m-Xylene & p-Xylene	39.2	1	ug/L	40		98	80-121	0.43	15	
Naphthalene	19.8	1	ug/L	20		99	56-143	1.3	48	
n-Butylbenzene	17.7	1	ug/L	20		89	72-120	0.18	25	
n-Propylbenzene	18.7	1	ug/L	20		93	76-120	0.38	26	
o-Xylene	19.3	1	ug/L	20		96	80-124	0.01	18	
p-Isopropyltoluene	18.7	1	ug/L	20		93	76-120	0.34	18	
sec-Butylbenzene	18.6	1	ug/L	20		93	77-120	0.04	19	
Styrene	19.2	1	ug/L	20		96	80-120	0.83	15	
tert-Butylbenzene	18.8	1	ug/L	20		94	78-120	0.26	19	
Tetrachloroethene	21	1	ug/L	20		105	74-120	0.88	18	
Toluene	20.9	1	ug/L	20		104	79-126	2.5	20	
trans-1,2-Dichloroethene	19.9	1	ug/L	20		100	76-120	0.65	20	
trans-1,3-Dichloropropene	19.5	1	ug/L	20		97	75-133	3.1	29	
Trichloroethene	20.1	1	ug/L	20		100	74-120	1.9	20	
Trichlorofluoromethane	20.5	1	ug/L	20		102	60-135	1.6	41	
Vinyl chloride	16.5	1	ug/L	20		83	68-121	2.1	33	
Surrogate: 1,2-Dichloroethane-d4	17.6		ug/L	20		88	72-123			
Surrogate: 4-Bromofluorobenzene	17.7		ug/L	20		88	74-120			
Surrogate: Dibromofluoromethane	19.5		ug/L	20		97	80-123			
Surrogate: Toluene-d8	20.9		ug/L	20		105	78-120			

TestAmerica Irvine

Sushmitha Reddy  
 Project Manager

Siemens Industry, Inc.  
P.O. Box 3308 (2523 Mutahar St.) IMA065  
Parker, AZ 85344  
Attention: Monte McCue

Project ID: Subpart CC  
Subpart CC  
Report Number: IUK2238

Sampled: 11/15/11  
Received: 11/16/11

## DATA QUALIFIERS AND DEFINITIONS

- MNR1** There was no MS/MSD analyzed with this batch due to insufficient sample volume. See Blank Spike/Blank Spike Duplicate.
- Z** Due to sample matrix effects, the surrogate recovery was below the acceptance limits.
- ND** Analyte NOT DETECTED at or above the reporting limit or MDL, if MDL is specified.
- RPD** Relative Percent Difference

## ADDITIONAL COMMENTS

**For 1,2-Diphenylhydrazine:**

The result for 1,2-Diphenylhydrazine is based upon the reading of its breakdown product, Azobenzene.

**TestAmerica Irvine**

Sushmitha Reddy  
Project Manager

Siemens Industry, Inc.  
P.O. Box 3308 (2523 Mutahar St.) IMA065  
Parker, AZ 85344  
Attention: Monte McCue

Project ID: Subpart CC  
Subpart CC  
Report Number: IUK2238

Sampled: 11/15/11  
Received: 11/16/11

## Certification Summary

### TestAmerica Irvine

Method	Matrix	Nelac	California
EPA 8081A	Water	X	X
EPA 8082	Water	X	X
EPA 8270C	Water	X	N/A
None	Water		

*Nevada and NELAP provide analyte specific accreditations. Analyte specific information for TestAmerica may be obtained by contacting the laboratory or visiting our website at [www.testamericainc.com](http://www.testamericainc.com)*

### Subcontracted Laboratories

#### TestAmerica - Cedar Falls, IA

704 Enterprise Drive - Cedar Falls, IA 50613

Method Performed: SW 8015

Samples: IUK2238-01, IUK2238-02, IUK2238-03, IUK2238-04, IUK2238-05

#### TestAmerica West Sacramento *NELAC Cert #1119CA, Nevada Cert #CA44*

880 Riverside Parkway - West Sacramento, CA 95605

Method Performed: SW846 8260B

Samples: IUK2238-01, IUK2238-02, IUK2238-03, IUK2238-04, IUK2238-05

### TestAmerica Irvine

Sushmitha Reddy  
Project Manager

Nashville, TN  
 Orlando, FL  
 Cedar Falls, IA

 Dayton, OH  
 Watertown, WI  
 Pontiac, MI

 Indianapolis, IN  
 Irvine, CA

## CHAIN OF CUSTODY RECORD

**IUK2238**

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Compliance Monitoring?  Yes  No  
 Enforcement Action?  Yes  No

Client Name/Account #: **SIEMENS INDUSTRY INC. - PARKER**


Address: **POB 3308 / 2523 MUTA HAR ST**

City/State/Zip: **PARKER, AZ 85344**

Project Manager: **Sushmitha Reddy**

Telephone Number: **928-669-5758**

Sampler Name: (Print) **Monte McCue**

Sampler Signature: 

Report To: **monte.mccue@siemens.com**

Invoice To:

Fax No.: **928-669-5775**

TA Quote #:

Project ID: **Subpart CC**

Project #:

Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Ice	Preservative								Matrix								Analyze For:	RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report		
								HNO <sub>3</sub> (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H <sub>2</sub> SO <sub>4</sub> Plastic (Yellow Label)	H <sub>2</sub> SO <sub>4</sub> Glass (Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Water	Drinking Water	Sludge	Soil	Other (Specify)	8260	8015 Alcohol Scan	8270							
Subpart CC VOA # A	15-Nov	1000	3																											
Subpart CC VOA # B	15-Nov	1000	3																											
Subpart CC VOA # C	15-Nov	1000	3																											
Subpart CC VOA # D	15-Nov	1000	3																											
Subpart CC VOA # E	15-Nov	1000	3																											
Subpart CC VOA # A	15-Nov	1000	3																											
Subpart CC VOA # B	15-Nov	1000	3																											
Subpart CC VOA # C	15-Nov	1000	3																											
Subpart CC VOA # D	15-Nov	1000	3																											
Subpart CC VOA # E	15-Nov	1000	3																											

### Special Instructions:

Laboratory Comments:  
 Temperature Upon Receipt: **2.4°C**  
 VOCs Free of Headspace? **Y N**

Relinquished by:	Date	Time	Method of Shipment:		Received by:	Date	Time
			UPS	Time			
Monte McCue	11/15/2011	1400					
Relinquished by:	Date	Time			<i>Vubana</i>	11/16/11	12:20

Nashville, TN  
Orlando, FL  
Cedar Falls, IA

Indianapolis, IN  
Irvine, CA

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Client Name/Account #: **SIEMENS INDUSTRY INC. - PARKER**

Address: **POB 3308 / 2523 MUTAHAR ST**

City/State/Zip: **PARKER, AZ 85344**

Project Manager: **Sushmitha Reddy**

Telephone Number: **928-669-5758**

Sampler Name: (Print) **Monte McCue**

Sampler Signature:

Fax No.: **928-669-5775**

TA Quote #:

Project ID: **Subpart CC**

Project #:

Compliance Monitoring?  Yes  No  
Enforcement Action?  Yes  No

Report To: **monte.mccue@siemens.com**

Invoice To:

Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Preservative										Matrix			Analyze For:	RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report														
							Ice	HNO <sub>3</sub> (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H <sub>2</sub> SO <sub>4</sub> Plastic (Yellow Label)	H <sub>2</sub> SO <sub>4</sub> Glass (Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Water	Drinking Water	Sludge	Soil						Other (specify):	8270	8081 / 8082											
Subpart CC (A) 1L AMB	15-Nov	1000	2																																			
Subpart CC (B) 1L AMB	15-Nov	1000	2																																			
Subpart CC (C) 1L AMB	15-Nov	1000	2																																			
Subpart CC (D) 1L AMB	15-Nov	1000	2																																			
Subpart CC (E) 1L AMB	15-Nov	1000	2																																			
Subpart CC (A) 1L AMB	15-Nov	1000	2																																			
Subpart CC (B) 1L AMB	15-Nov	1000	2																																			
Subpart CC (C) 1L AMB	15-Nov	1000	2																																			
Subpart CC (D) 1L AMB	15-Nov	1000	2																																			
Subpart CC (E) 1L AMB	15-Nov	1000	2																																			

**Special Instructions:**

Relinquished by: **Monte McCue** Date: 11/15/2011 Time: 1400

Received by: **VuBandy** Date: 11/16/11 Time: 12:20

Relinquished by: \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

Received by: \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

**Laboratory Comments:** 2.4<sup>o</sup>  
Temperature Upon Receipt:  Y  N  
Sample Containers Intact?  Y  N

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Client Name/Account #: **SIEMENS INDUSTRY INC. - PARKER**


Address: **PO BOX 3308 / 2523 MUTAHAR ST**

City/State/Zip: **PARKER, AZ 85344**

Project Manager: **Sushmitha Reddy**

Telephone Number: **928-669-5758**

Sampler Name: (Print) **Monte McCue**

Sampler Signature: 

Compliance Monitoring? Yes  No

Enforcement Action? Yes  No

Report To: **monte.mccue@siemens.com**

Invoice To:

TA Quote #:

Project ID: **Subpart CC**

Project #:

Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Ice	Preservative								Matrix								Analyze For:	RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report								
								HNO <sub>3</sub> (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H <sub>2</sub> SO <sub>4</sub> Plastic (Yellow Label)	H <sub>2</sub> SO <sub>4</sub> Glass (Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Water	Drinking Water	Sludge	Soil	Other (specify):	8270	8081 / 8082	EXTRA													
Trip Blank	15-Nov	1000	3																																	
Trip Blank	15-Nov	1000	3																																	

Special Instructions:

Laboratory Comments: *Diff*  
 Temperature Upon Receipt: Y N  
 Sample Containers Intact? Y N

Relinquished by:	Date	Time	Received by:	Date	Time
Monte McCue	11/15/2011	1400	<i>Nw Bandy</i>	11/16/11	12:20
Relinquished by:	Date	Time	Received by TestAmerica:	Date	Time

# **APPENDIX C**

## Facility Sampling Plan



## CHAPTER FOUR ORGANIC ANALYTES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in this chapter is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

### 4.1 SAMPLING CONSIDERATIONS

#### 4.1.1 Introduction

Following the initial and critical step of designing a sampling plan (Chapter Nine) is the implementation of that plan such that a representative sample of the solid waste is collected. Once the sample has been collected it must be stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. The sample type, type of containers and their preparation, possible forms of contamination, and preservation methods are all items which must be thoroughly examined in order to maintain the integrity of the samples. This section highlights considerations which must be addressed in order to maintain a sample's integrity and representativeness. This section is, however, applicable only to trace analyses.

Quality Control (QC) requirements need not be met for all compounds presented in the Table of Analytes for the method in use, rather, they must be met for all compounds reported. A report of non-detect is considered a quantitative report, and must meet all applicable QC requirements for that compound and the method used.

#### 4.1.2 Sample Handling and Preservation

This section deals separately with volatile and semivolatile organics. Refer to Chapter Two and Table 4-1 of this section for sample containers, sample preservation, and sample holding time information.

##### Volatile Organics

Standard 40 mL glass screw-cap VOA vials with Teflon lined silicone septa may be used for liquid matrices. Special 40 mL VOA vials for purge-and-trap of solid samples are described in Method 5035. VOA vials for headspace analysis of solid samples are described in Method 5021. Standard 125 mL widemouth glass containers may be used for Methods 5031 and 5032. However, the sampling procedures described in Method 5035 may minimize sample preparation analyte loss better than the procedures described in Methods 5031 and 5032. The vials and septa should be washed with soap and water and rinsed with distilled deionized water. After thoroughly cleaning the vials and septa, they should be placed in an oven and dried at 100°C for approximately one hour.

**NOTE:** Do not heat the septa for extended periods of time (i.e., more than one hour, because the silicone begins to slowly degrade at 105°C).

When collecting the samples, liquids and solids should be introduced into the vials gently to reduce agitation which might drive off volatile compounds.

In general, liquid samples should be poured into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. The vials should be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The sample should be hermetically sealed in the vial at the time of sampling, and must not be opened prior to analysis to preserve their integrity.

- Due to differing solubility and diffusion properties of gases in LIQUID matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of micro bubbles, and should not invalidate a sample for volatiles analysis.
- The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. The latter case is usually accompanied by a buildup of pressure within the vial, (e.g. carbonate-containing samples preserved with acid). Studies conducted by the USEPA (EMSL-Ci, unpublished data) indicate that "pea-sized" bubbles (i.e., bubbles not exceeding 1/4 inch or 6 mm in diameter) did not adversely affect volatiles data. These bubbles were generally encountered in wastewater samples, which are more susceptible to variations in gas solubility than are groundwater samples.

Immediately prior to analysis of liquid samples, the aliquot to be analyzed should be taken from the vial using the instructions from the appropriate sample introduction technique:

- For smaller analysis volumes, a gas-tight syringe may be inserted directly through the septum of the vial to withdraw the sample.
- For larger analysis volumes, (e.g. purge-and-trap analyses) the sample may be carefully poured into the syringe barrel. Opening a volatile sample to pour a sample into a syringe destroys the validity of the sample for future analysis. Therefore, if there is only one VOA vial, it is strongly recommended that the analyst fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly.

If these guidelines are not followed, the validity of the data generated from the samples may be suspect.

VOA vials for samples with solid or semi-solid matrices (e.g., sludges) should be filled according to the guidance given in the appropriate 5000 series sample introduction method (see Table 4-1) to be used. When 125-mL widemouth glass containers are used, the containers should be filled as completely as possible. The 125-mL vials should be tapped slightly as they are filled to try and eliminate as much free air space as possible. A minimum of two vials should also be filled per sample location.

At least two VOA vials should be filled and labeled immediately at the point at which the sample is collected. They should NOT be filled near a running motor or any type of exhaust system

because discharged fumes and vapors may contaminate the samples. The two vials from each sampling location should then be sealed in separate plastic bags to prevent cross-contamination between samples, particularly if the sampled waste is suspected of containing high levels of volatile organics. (Activated carbon may also be included in the bags to prevent cross-contamination from highly contaminated samples). VOA samples may also be contaminated by diffusion of volatile organics through the septum during shipment and storage. To monitor possible contamination, a trip blank prepared from organic-free reagent water (as defined in Chapter One) should be carried throughout the sampling, storage, and shipping process.

#### Semivolatile Organics (including Pesticides, PCBs and Herbicides.)

Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing (see Sec. 4.1.4 for specific instructions on glassware cleaning). The sample containers should be of glass or Teflon, and have screw-caps with Teflon lined septa. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may **NOT** be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank.

#### 4.1.3 Safety

Safety should always be the primary consideration in the collection of samples. A thorough understanding of the waste production process, as well as all of the potential hazards making up the waste, should be investigated whenever possible. The site should be visually evaluated just prior to sampling to determine additional safety measures. Minimum protection of gloves and safety glasses should be worn to prevent sample contact with the skin and eyes. A respirator should be worn even when working outdoors if organic vapors are present. More hazardous sampling missions may require the use of supplied air and special clothing.

#### 4.1.4 Cleaning of Glassware

In the analysis of samples containing components in the parts per billion range, the preparation of scrupulously clean glassware is necessary. Failure to do so can lead to a myriad of problems in the interpretation of the final chromatograms due to the presence of extraneous peaks resulting from contamination. Particular care must be taken with glassware such as Soxhlet extractors, Kuderna-Danish evaporative concentrators, sampling-train components, or any other glassware coming in contact with an extract that will be evaporated to a smaller volume. The process of concentrating the compounds of interest in this operation may similarly concentrate the contaminating substance(s), which may seriously distort the results.

The basic cleaning steps are:

1. Removal of surface residuals immediately after use;
2. Hot soak to loosen and float most particulate material;

3. Hot water rinse to flush away floated particulates;
4. Soak with an oxidizing agent to destroy traces of organic compounds;
5. Hot water rinse to flush away materials loosened by the deep penetrant soak;
6. Distilled water rinse to remove metallic deposits from the tap water;
7. Alcohol, e.g., isopropanol or methanol, rinse to flush off any final traces of organic materials and remove the water; and
8. Flushing the item immediately before use with some of the same solvent that will be used in the analysis.

Each of these eight fundamental steps are discussed here in the order in which they appeared on the preceding page.

1. As soon possible after glassware (i.e., beakers, pipets, flasks, or bottles) has come in contact with sample or standards, the glassware should be flushed with alcohol before it is placed in the hot detergent soak. If this is not done, the soak bath may serve to contaminate all other glassware placed therein.
2. The hot soak consists of a bath of a suitable detergent in water of 50°C or higher. The detergent, powder or liquid, should be entirely synthetic and not a fatty acid base. There are very few areas of the country where the water hardness is sufficiently low to avoid the formation of some hard-water scum resulting from the reaction between calcium and magnesium salts with a fatty acid soap. This hard-water scum or curd would have an affinity particularly for many chlorinated compounds and, being almost wholly water-insoluble, would deposit on all glassware in the bath in a thin film.

There are many suitable detergents on the wholesale and retail market. Most of the common liquid dishwashing detergents sold at retail are satisfactory but are more expensive than other comparable products sold industrially. Alconox, in powder or tablet form, is manufactured by Alconox, Inc., New York, and is marketed by a number of laboratory supply firms. Sparkleen, another powdered product, is distributed by Fisher Scientific Company.

3. No comments required.
4. The most common and highly effective oxidizing agent for removal of traces of organic compounds is the traditional chromic acid solution made up of concentrated sulfuric acid and potassium or sodium dichromate. For maximum efficiency, the soak solution should be hot (40-50°C). Safety precautions must be rigidly observed in the handling of this solution. Prescribed safety gear should include safety goggles, rubber gloves, and apron. The bench area where this operation is conducted should be covered with fluorocarbon sheeting because spattering will disintegrate any unprotected surfaces.

The potential hazards of using chromic-sulfuric acid mixture are great and have been well publicized. There are now commercially available substitutes that possess the advantage of safety in handling. These are biodegradable concentrates with a

claimed cleaning strength equal to the chromic acid solution. They are alkaline, equivalent to ca. 0.1 N NaOH upon dilution, and are claimed to remove dried blood, silicone greases, distillation residues, insoluble organic residues, etc. They are further claimed to remove radioactive traces and will not attack glass or exert a corrosive effect on skin or clothing. One such product is "Chem Solv 2157," manufactured by Mallinckrodt and available through laboratory supply firms. Another comparable product is "Detex," a product of Borer-Chemie, Solothurn, Switzerland.

5, 6, and 7. No comments required.

8. There is always a possibility that between the time of washing and the next use, the glassware could pick up some contamination from either the air or direct contact. To ensure against this, it is good practice to flush the item immediately before use with some of the same solvent that will be used in the analysis.

The drying and storage of the cleaned glassware is of critical importance to prevent the beneficial effects of the scrupulous cleaning from being nullified. Pegboard drying is not recommended. It is recommended that laboratory glassware and equipment be dried at 100°C. Under no circumstances should such small items be left in the open without protective covering. The dust cloud raised by the daily sweeping of the laboratory floor can most effectively recontaminate the clean glassware.

As an alternate to solvent rinsing, the glassware can be heated to a minimum of 300°C to vaporize any organics. Do not use this high temperature treatment on volumetric glassware, glassware with ground glass joints, or sintered glassware.

#### 4.1.5 High Concentration Samples

Cross contamination of trace concentration samples may occur when prepared in the same laboratory with high concentration samples. Ideally, if both type samples are being handled, a laboratory and glassware dedicated solely to the preparation of high concentration samples would be available for this purpose. If this is not feasible, as a minimum when preparing high concentration samples, disposable glassware should be used or, at least, glassware dedicated entirely to the high concentration samples. Avoid cleaning glassware used for both trace and high concentration samples in the same area.

TABLE 4-1.  
 SAMPLE CONTAINERS, PRESERVATION, TECHNIQUES, AND HOLDING TIMES

VOLATILE ORGANICS			
Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	Method 5035: 40-mL vials with stirring bar. Method 5021: See method. Methods 5031 & 5032: 125-mL widemouth glass container. Use Teflon-lined lids for all procedures.	Cool to 4°C.	14 days
Aqueous Samples With No Residual Chlorine Present	Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps	Cool to 4°C and adjust pH to less than 2 with H <sub>2</sub> SO <sub>4</sub> , HCl, or solid NaHSO <sub>4</sub> .	14 days
Aqueous Samples WITH Residual Chlorine Present	Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps	Collect sample in a 125-mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial. Cool to 4°C and adjust pH to less than 2 with H <sub>2</sub> SO <sub>4</sub> , HCl, or solid NaHSO <sub>4</sub> .	14 days
Acrolein and Acrylonitrile in Aqueous Sample	Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps	Adjust to pH 4-5. Cool to 4°C.	14 days
Solid Samples (e.g. soils, sediments, sludges, ash)	Method 5035: 40-mL vials with septum and stirring bar. Method 5021: See method. Methods 5031 & 5032: 125-mL widemouth glass container with Teflon-lined lids.	See the individual methods.	14 days

TABLE 4-1 (Continued)

SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES/PCBs AND HERBICIDES			
Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	125-mL widemouth glass with Teflon-lined lid	None	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.
Aqueous Samples With No Residual Chlorine Present	1-gal., 2 x 0.5-gal., or 4 x 1-L amber glass container with Teflon-lined lid	Cool to 4°C	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.
Aqueous Samples WITH Residual Chlorine Present	1-gal., 2 x 0.5-gal., or 4 x 1-L, amber glass container with Teflon-lined lid.	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4°C.	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.
Solid Samples (e.g. soils, sediments, sludges, ash)	250-mL widemouth glass container with Teflon-lined lid	Cool to 4°C	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.

## 4.2 SAMPLE PREPARATION METHODS

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

### 4.2.1 EXTRACTIONS AND PREPARATIONS

The following methods are included in this section:

<b>Method 3500B:</b>	Organic Extraction and Sample Preparation
<b>Method 3510C:</b>	Separatory Funnel Liquid-Liquid Extraction
<b>Method 3520C:</b>	Continuous Liquid-Liquid Extraction
<b>Method 3535A:</b>	Solid-Phase Extraction (SPE)
<b>Method 3540C:</b>	Soxhlet Extraction
<b>Method 3541:</b>	Automated Soxhlet Extraction
<b>Method 3542:</b>	Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)
<b>Method 3545A:</b>	Pressurized Fluid Extraction (PFE)
<b>Method 3550B:</b>	Ultrasonic Extraction
<b>Method 3560:</b>	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
<b>Method 3561:</b>	Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons
<b>Method 3562:</b>	Supercritical Fluid Extraction of Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides
<b>Method 3580A:</b>	Waste Dilution
<b>Method 3585:</b>	Waste Dilution for Volatile Organics
<b>Method 5000:</b>	Sample Preparation for Volatile Organic Compounds
<b>Method 5021:</b>	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis
<b>Method 5030B:</b>	Purge-and-Trap for Aqueous Samples
<b>Method 5031:</b>	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
<b>Method 5032:</b>	Volatile Organic Compounds by Vacuum Distillation
<b>Method 5035:</b>	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
<b>Method 5041A:</b>	Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST)



## 4.2 SAMPLE PREPARATION METHODS

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### 4.2.2 CLEANUP

The following methods are included in this section:

<b>Method 3600C:</b>	Cleanup
<b>Method 3610B:</b>	Alumina Cleanup
<b>Method 3611B:</b>	Alumina Column Cleanup and Separation of Petroleum Wastes
<b>Method 3620B:</b>	Florisil Cleanup
<b>Method 3630C:</b>	Silica Gel Cleanup
<b>Method 3640A:</b>	Gel-Permeation Cleanup
<b>Method 3650B:</b>	Acid-Base Partition Cleanup
<b>Method 3660B:</b>	Sulfur Cleanup
<b>Method 3665A:</b>	Sulfuric Acid/Permanganate Cleanup

### 4.3 DETERMINATION OF ORGANIC ANALYTES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

#### 4.3.1 GAS CHROMATOGRAPHIC METHODS

The following methods are included in this section:

<b>Method 8000B:</b>	Determinative Chromatographic Separations
<b>Method 8011:</b>	1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatography
<b>Method 8015B:</b>	Nonhalogenated Organics Using GC/FID
<b>Method 8021B:</b>	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors
<b>Method 8031:</b>	Acrylonitrile by Gas Chromatography
<b>Method 8032A:</b>	Acrylamide by Gas Chromatography
<b>Method 8033:</b>	Acetonitrile by Gas Chromatography with Nitrogen-Phosphorus Detection
<b>Method 8041:</b>	Phenols by Gas Chromatography
<b>Method 8061A:</b>	Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC/ECD)
<b>Method 8070A:</b>	Nitrosamines by Gas Chromatography
<b>Method 8081B:</b>	Organochlorine Pesticides by Gas Chromatography
<b>Method 8082A:</b>	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
<b>Method 8091:</b>	Nitroaromatics and Cyclic Ketones by Gas Chromatography
<b>Method 8100:</b>	Polynuclear Aromatic Hydrocarbons
<b>Method 8111:</b>	Haloethers by Gas Chromatography
<b>Method 8121:</b>	Chlorinated Hydrocarbons by Gas Chromatography: Capillary Column Technique
<b>Method 8131:</b>	Aniline and Selected Derivatives by Gas Chromatography
<b>Method 8141B:</b>	Organophosphorus Compounds by Gas Chromatography
<b>Method 8151A:</b>	Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization

### 4.3 DETERMINATION OF ORGANIC ANALYTES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

#### 4.3.2 GAS CHROMATOGRAPHIC/MASS SPECTROMETRIC METHODS

The following methods are included in this section:

- Method 8260B:** Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
- Method 8270D:** Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
- Method 8275A:** Semivolatile Organic Compounds (PAHs and PCBs) in Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS)
- Method 8280B:** Polychlorinated Dibenzo-*p*-Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)
- Method 8290A:** Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)
- Appendix A:** Procedures for the Collection, Handling, Analysis, and Reporting of Wipe Tests Performed within the Laboratory

### 4.3 DETERMINATION OF ORGANIC ANALYTES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

#### 4.3.3 HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHODS

The following methods are included in this section:

<b>Method 8310:</b>	Polynuclear Aromatic Hydrocarbons
<b>Method 8315A:</b>	Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)
<b>Appendix A:</b>	Recrystallization of 2,4-Dinitrophenylhydrazine (DNPH)
<b>Method 8316:</b>	Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)
<b>Method 8318:</b>	N-Methylcarbamates by High Performance Liquid Chromatography (HPLC)
<b>Method 8321B:</b>	Solvent-Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection
<b>Method 8325:</b>	Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry (HPLC/PB/MS)
<b>Method 8330A:</b>	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)
<b>Method 8331:</b>	Tetrazene by Reverse Phase High Performance Liquid Chromatography (HPLC)
<b>Method 8332:</b>	Nitroglycerine by High Performance Liquid Chromatography

### 4.3 DETERMINATION OF ORGANIC ANALYTES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

#### 4.3.4 INFRARED METHODS

The following methods are included in this section:

- |                     |   |
|---------------------|---|
| <b>Method 8410:</b> | Gas Chromatography/Fourier Transform Infrared (GC/FT-IR) Spectrometry for Semivolatile Organics: Capillary Column |
| <b>Method 8430:</b> | Analysis of Bis(2-chloroethyl) Ether and Hydrolysis Products by Direct Aqueous Injection GC/FT-IR                 |
| <b>Method 8440:</b> | Total Recoverable Petroleum Hydrocarbons by Infrared Spectrophotometry  |

### 4.3 DETERMINATION OF ORGANIC ANALYTES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

#### 4.3.5 MISCELLANEOUS SPECTROMETRIC METHODS

The following method is included in this section:

**Method 8520:** Continuous Measurement of Formaldehyde in Ambient Air

#### 4.4 IMMUNOASSAY METHODS

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

The following methods are included in this section:

<b>Method 4000:</b>	Immunoassay
<b>Method 4010A:</b>	Screening for Pentachlorophenol by Immunoassay
<b>Method 4015:</b>	Screening for 2,4-Dichlorophenoxyacetic Acid by Immunoassay
<b>Method 4020:</b>	Screening for Polychlorinated Biphenyls by Immunoassay
<b>Method 4030:</b>	Soil Screening for Petroleum Hydrocarbons by Immunoassay
<b>Method 4035:</b>	Soil Screening for Polynuclear Aromatic Hydrocarbons by Immunoassay
<b>Method 4040:</b>	Soil Screening for Toxaphene by Immunoassay
<b>Method 4041:</b>	Soil Screening for Chlordane by Immunoassay
<b>Method 4042:</b>	Soil Screening for DDT by Immunoassay
<b>Method 4050:</b>	TNT Explosives in Soil by Immunoassay
<b>Method 4051:</b>	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in Soil by Immunoassay
<b>Method 4670:</b>	Triazine Herbicides as Atrazine in Water by Quantitative Immunoassay

#### 4.5 MISCELLANEOUS SCREENING METHODS

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

The following methods are included in this section:

<b>Method 3820:</b>	Hexadecane Extraction and Screening of Purgeable Organics
<b>Method 8515:</b>	Colorimetric Screening Method for Trinitrotoluene (TNT) in Soil
<b>Method 9074:</b>	Turbidmetric Screening Method for Total Recoverable Petroleum Hydrocarbons in Soil
<b>Method 9078:</b>	Screening Test Method for Polychlorinated Biphenyls in Soil
<b>Method 9079:</b>	Screening Test Method for Polychlorinated Biphenyls in Transformer Oil



# **APPENDIX D**

EPA Method  
Techniques

METHOD 8260B  
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/  
 MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

Compound	CAS No. <sup>b</sup>	Appropriate Preparation Technique <sup>a</sup>					Direct Inject.
		5030/ 5035	5031	5032	5021	5041	
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d <sub>5</sub> (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc

(continued)

Compound	CAS No. <sup>b</sup>	Appropriate Preparation Technique <sup>a</sup>					Direct Inject.
		5030/ 5035	5031	5032	5021	5041	
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d <sub>4</sub> (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d <sub>4</sub> (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (IS)	540-36-3	nd	nd	nd	nd	c	nd
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	l	nd	nd	nd	nd	c
Ethanol	64-17-5	l	c	c	nd	nd	c
Ethyl acetate	141-78-6	l	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	l	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	l	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	l	nd	nd	nd	c
Methanol	67-56-1	l	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c

(continued)

Compound	CAS No. <sup>b</sup>	Appropriate Preparation Technique <sup>a</sup>					Direct Inject.
		5030/ 5035	5031	5032	5021	5041	
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	l	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	l	nd	nd	nd	c
β-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	pc
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	l	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-3	c	nd	c	c	c	c
Toluene-d <sub>8</sub> (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluidine	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethene	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
o-Xylene	95-47-6	c	nd	c	c	c	c
m-Xylene	108-38-3	c	nd	c	c	c	c
p-Xylene	106-42-3	c	nd	c	c	c	c

<sup>a</sup> See Sec. 1.2 for other appropriate sample preparation techniques

<sup>b</sup> Chemical Abstract Service Registry Number

- c = Adequate response by this technique
- ht = Method analyte only when purged at 80°C
- nd = Not determined
- l = Inappropriate technique for this analyte
- pc = Poor chromatographic behavior
- pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
- surr = Surrogate
- IS = Internal Standard

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The more common techniques are listed in the table above. Purge-and-trap, by Methods 5030 (aqueous samples) and 5035 (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. These include direct injection following dilution with hexadecane (Method 3585) for waste oil samples; automated static headspace by Method 5021 for solid samples; direct injection of an aqueous sample (concentration permitting) or injection of a sample concentrated by azeotropic distillation (Method 5031); and closed system vacuum distillation (Method 5032) for aqueous, solid, oil and tissue samples. For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040). Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile, water soluble compounds can be included in this analytical technique by the use of azeotropic distillation or closed-system vacuum distillation. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented. The following compounds are also amenable to analysis by Method 8260:

Bromobenzene	1,3-Dichloropropane
n-Butylbenzene	2,2-Dichloropropane
sec-Butylbenzene	1,1-Dichloropropene
tert-Butylbenzene	p-Isopropyltoluene
Chloroacetonitrile	Methyl acrylate
1-Chlorobutane	Methyl-t-butyl ether
1-Chlorohexane	Pentafluorobenzene
2-Chlorotoluene	n-Propylbenzene
4-Chlorotoluene	1,2,3-Trichlorobenzene
Dibromofluoromethane	1,2,4-Trimethylbenzene
cis-1,2-Dichloroethene	1,3,5-Trimethylbenzene

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrupole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

## 2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column or cryofocused on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

## 3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explain this in text accompanying the uncorrected data.

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique (Method 5021) or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

#### 4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.

4.2 Purge-and-trap device for solid samples - Described in Method 5035.

4.3 Automated static headspace device for solid samples - Described in Method 5021.

4.4 Azeotropic distillation apparatus for aqueous and solid samples - Described in Method 5031.

4.5 Vacuum distillation apparatus for aqueous, solid and tissue samples - Described in Method 5032.

4.6 Desorption device for air trapping media for air samples - Described in Method 5041.

4.7 Air sampling loop for sampling from Tedlar® bags for air samples - Described in Method 0040.

4.8 Injection port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven (see illustration below). A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

#### 4.9 Gas chromatography/mass spectrometer/data system

4.9.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.9.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.9.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.9.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.9.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph, and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.9.1.5 During the cryofocussing step, the temperature of the fused-silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

#### 4.9.2 Gas chromatographic columns

4.9.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5- $\mu$ m film thickness, or equivalent.

4.9.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt<sub>x</sub>-502.2 (RESTEK), or VOCOL (Supelco), 3- $\mu$ m film thickness, or equivalent.

4.9.2.3 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt<sub>x</sub>-5, SPB-5, or equivalent), 1- $\mu$ m film thickness.

4.9.2.4 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624 (J&W Scientific), 1.8- $\mu$ m film thickness, or equivalent.



4.9.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at  $m/z$  49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.9.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

4.9.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

4.9.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.9.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.9.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.10 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000- $\mu$ L.

4.11 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.12 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.13 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.14 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

- 4.15 Vials - 2-mL, for GC autosampler.
- 4.16 Disposable pipets - Pasteur.
- 4.17 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.
- 4.18 Spatula - Stainless steel.

## 5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH<sub>3</sub>OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Hexadecane - Reagent hexadecane is defined as hexadecane in which interference is not observed at the method detection limit of compounds of interest. Hexadecane quality is demonstrated through the analysis of a solvent blank injected directly into the GC/MS. The results of such a blank analysis must demonstrate that all interfering volatiles have been removed from the hexadecane.

5.5 Polyethylene glycol, H(OCH<sub>2</sub>CH<sub>2</sub>)<sub>n</sub>OH - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100- $\mu$ L syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.7.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to

5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a septum. Attach PTFE tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.7.4 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

#### 5.7.5 Frequency of Standard Preparation

5.7.5.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.5.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

#### 5.7.6 Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially-available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichloro-difluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

- a) Connect either the 100- $\mu$ L or 500- $\mu$ L Luer syringe to the inlet fitting of the cylinder.
- b) Make sure the on/off valve on the syringe is in the open position.
- c) Slowly open the valve on the cylinder and withdraw a full syringe volume.
- d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.
- e) Expel the gas from the syringe into a well-ventilated area.
- f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is ~30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

**NOTE:** Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025  $\mu$ g/ $\mu$ L.

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

<u>Gas Volume</u>	<u>Calibration Concentration</u>
40 $\mu$ L	20 $\mu$ g/L
100 $\mu$ L	50 $\mu$ g/L
200 $\mu$ L	100 $\mu$ g/L
300 $\mu$ L	150 $\mu$ g/L
400 $\mu$ L	200 $\mu$ g/L

5.7.6.11 The following are the recommended gas volumes spiked into 25 mL of water to produce a typical 5-point calibration:

<u>Gas Volume</u>	<u>Calibration Concentration</u>
10 $\mu$ L	1 $\mu$ g/L
20 $\mu$ L	2 $\mu$ g/L
50 $\mu$ L	5 $\mu$ g/L
100 $\mu$ L	10 $\mu$ g/L
250 $\mu$ L	25 $\mu$ g/L

5.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7.4 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene- $d_8$ , 4-bromofluorobenzene, 1,2-dichloroethane- $d_4$ , and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250  $\mu$ g/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 10  $\mu$ L of the surrogate spiking solution prior to analysis. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene- $d_5$ , and 1,4-dichlorobenzene- $d_4$ . Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.7 and 5.8. It is recommended that the secondary dilution standard be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10  $\mu$ L of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50  $\mu$ g/L. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/ $\mu$ L of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards - There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

## 7.0 PROCEDURE

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and-trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40°C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.1.3 Vacuum distillation - this technique may be used for the introduction of volatile organics from aqueous, solid, or tissue samples (Method 5032) into the GC/MS system.

7.1.4 Automated static headspace - this technique may be used for the introduction of volatile organics from solid samples (Method 5021) into the GC/MS system.

7.1.5 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

## 7.2 Recommended chromatographic conditions

### 7.2.1 General conditions

Injector temperature:	200 - 225 °C
Transfer line temperature:	250 - 300 °C

7.2.2 Column 1 and Column 2 with cryogenic cooling (example chromatograms are presented in Figures 1 and 2)

Carrier gas (He) flow rate: 15 mL/min  
Initial temperature: 10°C, hold for 5 minutes  
Temperature program: 6°C/min to 70°C, then 15°C/min to 145°C  
Final temperature: 145°C, hold until all expected compounds have eluted.

#### 7.2.5 Direct injection - Column 2

Carrier gas (He) flow rate: 4 mL/min  
Column: J&W DB-624, 70m x 0.53 mm  
Initial temperature: 40°C, hold for 3 minutes  
Temperature program: 8°C/min  
Final temperature: 260°C, hold until all expected compounds have eluted.  
Column Bake out: 75 minutes  
Injector temperature: 200-225°C  
Transfer line temperature: 250-300°C

#### 7.2.6 Direct split interface - Column 4

Carrier gas (He) flow rate: 1.5 mL/min  
Initial temperature: 35°C, hold for 2 minutes  
Temperature program: 4°C/min to 50°C  
10°C/min to 220°C  
Final temperature: 220°C, hold until all expected compounds have eluted  
Split ratio: 100:1  
Injector temperature: 125°C

### 7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:

Mass range: 35 - 260 amu  
Scan time: 0.6 - 2 sec/scan  
Source temperature: According to manufacturer's specifications  
Ion trap only: Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene (2- $\mu$ L injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of



BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 4 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

**NOTE:** All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10  $\mu$ L of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.2.3 To prepare a calibration standard for direct injection analysis of waste oil, dilute standards in hexadecane.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2  $\mu$ L into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

$A_s$  = Peak area (or height) of the analyte or surrogate.

$A_{is}$  = Peak area (or height) of the internal standard.

$C_s$  = Concentration of the analyte or surrogate.

$C_{is}$  = Concentration of the internal standard.

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

$RF_i$  = RF for each of the calibration standards

$\overline{RF}$  = mean RF for each compound from the initial calibration

n = Number of calibration standards, e.g., 5

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

#### 7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

**NOTE:** Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

NOTE: The 20% RSD criteria in Method 8000 pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

#### 7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

#### 7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater

than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

## 7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Some of the screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate. Sample screening is particularly important when Method 8260 is used to achieve low detection levels.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 10 µL of the surrogate spiking solution and 10 µL of the internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard.

If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.9 Add 10  $\mu\text{L}$  of the matrix spike solution (Sec. 5.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50  $\mu\text{g/L}$  of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2  $\mu\text{L}$  into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2  $\mu\text{L}$  injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

**NOTE:** It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

## 7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within  $\pm 0.06$  RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library



searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

- (1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within  $\pm 20\%$ . (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

## 7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor ( $\overline{RF}$ ) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas  $A_x$  and  $A_{is}$  should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

## 8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6. Calculated MDLs are presented in Table 1.

9.3 The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocused narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7. MDL values were also calculated from these data and are presented in Table 2.

9.4 Direct injection (Method 3585) has been used for the analysis of waste motor oil samples using a wide-bore column. Single laboratory precision and accuracy data are presented in Tables 10 and 11 for TCLP volatiles in oil. The performance data were developed by spiking and analyzing seven replicates each of new and used oil. The oils were spiked at the TCLP regulatory concentrations for most analytes, except for the alcohols, ketones, ethyl acetate and chlorobenzene which are spiked at 5 ppm, well below the regulatory concentrations. Prior to spiking, the new oil (an SAE 30-weight motor oil) was heated at 80°C overnight to remove volatiles. The used oil (a mixture of used oil drained from passenger automobiles) was not heated and was contaminated with 20 - 300 ppm of BTEX compounds and isobutanol. These contaminants contributed to the extremely high recoveries of the BTEX compounds in the used oil. Therefore, the data from the deuterated analogs of these analytes represent more typical recovery values.

9.5 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each

sample was fortified with the analytes at a concentration of 4 µg/kg. These data are listed in Tables 17, 18, and 19. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.

9.5.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.

9.5.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene, are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in Table 19 include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

9.6 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 12 to 16.

9.7 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 20 to 27.

9.8 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in two soil matrices: sand and a surface garden soil. Replicate samples were fortified with the analytes at concentrations of 10 µg/kg. These data are listed in Table 30. All data were calculated using the internal standards listed for each analyte in Table 28. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

9.8.1 If a detector other than an MS is used for analysis, consideration must be given to the choice of internal standards and surrogates. They must not coelute with any other analyte and must have similar properties to the analytes. The recoveries of the analytes are 50% or higher for each matrix studied. The recoveries of the gases or very volatile compounds are greater than 100% in some cases. Also, results include high recoveries of some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection.

9.8.2 The method detection limits using Method 5021 listed in Table 29 were calculated from results of seven replicate analyses of the sand matrix. Sand was chosen because it demonstrated the least degree of matrix effect of the soils studied. These MDLs were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

9.9 The MDL concentrations listed in Table 31 were determined using Method 5041 in conjunction with Method 8260. They were obtained using cleaned blank VOST tubes and reagent water. Similar results have been achieved with field samples. The MDL actually achieved in a given analysis will vary depending upon instrument sensitivity and the effects of the matrix. Preliminary spiking studies indicate that under the test conditions, the MDLs for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.

9.10 The EQL of sample taken by Method 0040 and analyzed by Method 8260 is estimated to be in the range of 0.03 to 0.9 ppm (See Table 33). Matrix effects may cause the individual compound detection limits to be higher.

## 10.0 REFERENCES

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TABLE 1

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)  
FOR VOLATILE ORGANIC COMPOUNDS ON WIDE-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes)			MDL <sup>d</sup> (µg/L)
	Column 1 <sup>a</sup>	Column 2 <sup>b</sup>	Column 2 <sup>c</sup>	
Dichlorodifluoromethane	1.35	0.70	3.13	0.10
Chloromethane	1.49	0.73	3.40	0.13
Vinyl Chloride	1.56	0.79	3.93	0.17
Bromomethane	2.19	0.96	4.80	0.11
Chloroethane	2.21	1.02	--	0.10
Trichlorofluoromethane	2.42	1.19	6.20	0.08
Acrolein	3.19			
Iodomethane	3.56			
Acetonitrile	4.11			
Carbon disulfide	4.11			
Allyl chloride	4.11			
Methylene chloride	4.40	2.06	9.27	0.03
1,1-Dichloroethene	4.57	1.57	7.83	0.12
Acetone	4.57			
trans-1,2-Dichloroethene	4.57	2.36	9.90	0.06
Acrylonitrile	5.00			
1,1-Dichloroethane	6.14	2.93	10.80	0.04
Vinyl acetate	6.43			
2,2-Dichloropropane	8.10	3.80	11.87	0.35
2-Butanone	--			
cis-1,2-Dichloroethene	8.25	3.90	11.93	0.12
Propionitrile	8.51			
Chloroform	9.01	4.80	12.60	0.03
Bromochloromethane	--	4.38	12.37	0.04
Methacrylonitrile	9.19			
1,1,1-Trichloroethane	10.18	4.84	12.83	0.08
Carbon tetrachloride	11.02	5.26	13.17	0.21
1,1-Dichloropropene	--	5.29	13.10	0.10
Benzene	11.50	5.67	13.50	0.04
1,2-Dichloroethane	12.09	5.83	13.63	0.06
Trichloroethene	14.03	7.27	14.80	0.19
1,2-Dichloropropane	14.51	7.66	15.20	0.04
Bromodichloromethane	15.39	8.49	15.80	0.08
Dibromomethane	15.43	7.93	5.43	0.24
Methyl methacrylate	15.50			
1,4-Dioxane	16.17			
2-Chloroethyl vinyl ether	--			
4-Methyl-2-pentanone	17.32			
trans-1,3-Dichloropropene	17.47	--	16.70	--
Toluene	18.29	10.00	17.40	0.11
cis-1,3-Dichloropropene	19.38	--	17.90	--

TABLE 1 (cont.)

Compound	Retention Time (minutes)			MDL <sup>d</sup> (µg/L)
	Column 1 <sup>a</sup>	Column 2 <sup>b</sup>	Column 2 <sup>nc</sup>	
1,1,2-Trichloroethane	19.59	11.05	18.30	0.10
Ethyl methacrylate	20.01			
2-Hexanone	20.30			
Tetrachloroethene	20.26	11.15	18.60	0.14
1,3-Dichloropropane	20.51	11.31	18.70	0.04
Dibromochloromethane	21.19	11.85	19.20	0.05
1,2-Dibromoethane	21.52	11.83	19.40	0.06
1-Chlorohexane	--	13.29	--	0.05
Chlorobenzene	23.17	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	23.36	13.33	20.87	0.05
Ethylbenzene	23.38	13.39	21.00	0.06
p-Xylene	23.54	13.69	21.30	0.13
m-Xylene	23.54	13.68	21.37	0.05
o-Xylene	25.16	14.52	22.27	0.11
Styrene	25.30	14.60	22.40	0.04
Bromoform	26.23	14.88	22.77	0.12
Isopropylbenzene (Cumene)	26.37	15.46	23.30	0.15
cis-1,4-Dichloro-2-butene	27.12			
1,1,2,2-Tetrachloroethane	27.29	16.35	24.07	0.04
Bromobenzene	27.46	15.86	24.00	0.03
1,2,3-Trichloropropane	27.55	16.23	24.13	0.32
n-Propylbenzene	27.58	16.41	24.33	0.04
2-Chlorotoluene	28.19	16.42	24.53	0.04
trans-1,4-Dichloro-2-butene	28.26			
1,3,5-Trimethylbenzene	28.31	16.90	24.83	0.05
4-Chlorotoluene	28.33	16.72	24.77	0.06
Pentachloroethane	29.41			
1,2,4-Trimethylbenzene	29.47	17.70	31.50	0.13
sec-Butylbenzene	30.25	18.09	26.13	0.13
tert-Butylbenzene	30.59	17.57	26.60	0.14
p-Isopropyltoluene	30.59	18.52	26.50	0.12
1,3-Dichlorobenzene	30.56	18.14	26.37	0.12
1,4-Dichlorobenzene	31.22	18.39	26.60	0.03
Benzyl chloride	32.00			
n-Butylbenzene	32.23	19.49	27.32	0.11
1,2-Dichlorobenzene	32.31	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	35.30	21.08	--	0.26
1,2,4-Trichlorobenzene	38.19	23.08	31.50	0.04
Hexachlorobutadiene	38.57	23.68	32.07	0.11
Naphthalene	39.05	23.52	32.20	0.04
1,2,3-Trichlorobenzene	40.01	24.18	32.97	0.03



TABLE 1 (cont.)

Compound	Retention Time (minutes)			MDL <sup>d</sup> (µg/L)
	Column 1 <sup>a</sup>	Column 2 <sup>b</sup>	Column 2" <sup>c</sup>	
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene-d <sub>5</sub>	23.10			
1,4-Dichlorobenzene-d <sub>4</sub>	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene-d <sub>4</sub>	32.30	19.08	27.25	
Dichloroethane-d <sub>4</sub>	12.08			
Dibromofluoromethane	--			
Toluene-d <sub>8</sub>	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

<sup>a</sup> Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

<sup>b</sup> Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

<sup>c</sup> Column 2" - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10 °C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

<sup>d</sup> MDL based on a 25-mL sample volume.

TABLE 2

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)  
FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes) Column 3 <sup>a</sup>	MDL <sup>b</sup> (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06

TABLE 2 (cont.)

Compound	Retention Time (minutes) Column 3 <sup>a</sup>	MDL <sup>b</sup> (µg/L)
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

<sup>a</sup> Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

<sup>b</sup> MDL based on a 25-mL sample volume.

TABLE 3

ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES<sup>a</sup>

Estimated Quantitation Limits		
5-mL Ground Water Purge (µg/L)	25-mL Ground water Purge (µg/L)	Low Soil/Sediment <sup>b</sup> µg/kg
5	1	5

<sup>a</sup> Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following footnote for further guidance on matrix-dependent EQLs.

<sup>b</sup> EQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor <sup>c</sup>
Water miscible liquid waste	50
High concentration soil and sludge	125
Non-water miscible waste	500

<sup>c</sup> EQL = [EQL for low soil sediment (Table 3)] x [Factor].

For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 4

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA<sup>a</sup>

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

<sup>a</sup> Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 5

## CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d <sub>4</sub>	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
$\beta$ -Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d <sub>6</sub>	84	83
Bromobenzene-d <sub>5</sub>	82	162
Bromochloromethane-d <sub>2</sub>	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d <sub>5</sub>	117	
1,4-Dichlorobenzene-d <sub>4</sub>	152	115, 150
1,1,2-Trichloroethane-d <sub>3</sub>	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d <sub>1</sub>	84	
Dibromofluoromethane	113	



TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Dichloroethane-d <sub>4</sub>	102	
Toluene-d <sub>8</sub>	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

\* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

TABLE 6

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR  
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED  
WITH A WIDE-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3

TABLE 6 (cont.)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	RSD
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

<sup>a</sup> Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

<sup>b</sup> Standard deviation was calculated by pooling data from three concentrations.

TABLE 7

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR  
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED  
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. (µg/L)	Number of Samples	% Recovery <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.5	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3

TABLE 7 (cont.)

Compound	Conc. (µg/L)	Number of Samples	% Recovery <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	RSD
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

<sup>a</sup> Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 8

## SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene <sup>a</sup>	86-115	74-121
Dibromofluoromethane <sup>a</sup>	86-118	80-120
Toluene-d <sub>8</sub> <sup>a</sup>	88-110	81-117
Dichloroethane-d <sub>4</sub> <sup>a</sup>	80-120	80-120

<sup>a</sup> Single laboratory data, for guidance only.

TABLE 9

## QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SAMPLES

Approximate Concentration Range (µg/kg)	Volume of Extract <sup>a</sup>
500 - 10,000	100 µL
1,000 - 20,000	50 µL
5,000 - 100,000	10 µL
25,000 - 500,000	100 µL of 1/50 dilution <sup>b</sup>

Calculate appropriate dilution factor for concentrations exceeding this table.

<sup>a</sup> The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of solvent is necessary to maintain a volume of 100 µL added to the syringe.

<sup>b</sup> Dilute an aliquot of the solvent extract and then take 100 µL for analysis.

TABLE 10

## DIRECT INJECTION ANALYSIS OF NEW OIL AT 5 PPM (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone	91	14.8	1.9	5.0
Benzene	86	21.3	0.1	0.5
n-Butanol*,**	107	27.8	0.5	5.0
iso-Butanol*,**	95	19.5	0.9	5.0
Carbon tetrachloride	86	44.7	0.0	0.5
Carbon disulfide**	53	22.3	0.0	5.0
Chlorobenzene	81	29.3	0.0	5.0
Chloroform	84	29.3	0.0	6.0
1,4-Dichlorobenzene	98	24.9	0.0	7.5
1,2-Dichloroethane	101	23.1	0.0	0.5
1,1-Dichloroethene	97	45.3	0.0	0.7
Diethyl ether	76	24.3	0.0	5.0
Ethyl acetate	113	27.4	0.0	5.0
Ethylbenzene	83	30.1	0.2	5.0
Hexachloroethane	71	30.3	0.0	3.0
Methylene chloride	98	45.3	0.0	5.0
Methyl ethyl ketone	79	24.6	0.4	5.0
MIBK	93	31.4	0.0	5.0
Nitrobenzene	89	30.3	0.0	2.0
Pyridine	31	35.9	0.0	5.0
Tetrachloroethene	82	27.1	0.0	0.7
Trichlorofluoromethane	76	27.6	0.0	5.0
1,1,2-Trichlorotrifluoroethane	69	29.2	0.0	5.0
Toluene	73	21.9	0.6	5.0
Trichloroethene	66	28.0	0.0	0.5
Vinyl chloride	63	35.2	0.0	0.2
o-Xylene	83	29.5	0.4	5.0
m/p-Xylene	84	29.5	0.6	10.0

\* Alternate mass employed

\*\* IS quantitation

Data are taken from Reference 9.

TABLE 11

SINGLE LABORATORY PERFORMANCE  
DATA FOR THE DIRECT INJECTION METHOD - USED OIL (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone**	105	54	2.0	5.0
Benzene	3135	44	14	0.5
Benzene-d <sub>6</sub>	56	44	2.9	0.5
n-Butanol**	100	71	12	5.0
iso-Butanol*,**	132	27	0	5.0
Carbon tetrachloride	143	68	0	0.5
Carbon tetrachloride- <sup>13</sup> C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene-d <sub>5</sub>	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform-d <sub>1</sub>	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene-d <sub>4</sub>	53	44	3.4	7.5
1,2-Dichloroethane**	191	54	0	0.5
1,1-Dichloroethene*	155	51	0	0.7
1,1-Dichloroethene-d <sub>2</sub>	68	44	3.4	0.7
Diethyl ether**	95	66	0	5.0
Ethyl acetate*,**	126	39	0	5.0
Ethylbenzene	1298	44	54	5.0
Ethylbenzene-d <sub>10</sub>	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane- <sup>13</sup> C	54	45	3.5	3.0
Methylene chloride**	86	65	0.3	5.0
Methyl ethyl ketone**	107	64	0	5.0
4-Methyl-2-pentanone (MIBK)**	100	74	0.1	5.0
Nitrobenzene	111	80	0	2.0
Nitrobenzene-d <sub>5</sub>	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine-d <sub>5</sub>	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl <sub>3</sub> F <sub>3</sub> ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene-d <sub>8</sub>	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene-d <sub>1</sub>	55	44	2.8	0.5



TABLE 11 (cont.)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Vinyl chloride**	100	69	0	0.2
o-Xylene	2292	44	105	5.0
o-Xylene-d <sub>10</sub>	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene-d <sub>10</sub>	67	44	3.7	10.0

\* Alternate mass employed

\*\* IS quantitation

ND = Not Detected

Data are based on seven measurements and are taken from Reference 9.

TABLE 12  
METHOD DETECTION LIMITS (METHOD 5031)

Compound	MDL (µg/L)	Concentration Factor	
	Macro <sup>a</sup>	Macro	Micro
Acetone	31	25-500	-
Acetonitrile	57	25-500	200
Acrolein	-	-	100
Acrylonitrile	16	25-500	100
Allyl Alcohol	7	25-500	-
1-Butanol	-	-	250
Crotonaldehyde	12	25-500	-
1,4-Dioxane	12	25-500	150
Ethyl Acetate	-	-	100
Isobutyl alcohol	7	25-500	-
Methanol	38	25-500	140
Methyl Ethyl Ketone	16	25-500	-
2-Methyl-1-propanol	-	-	250
n-Nitroso-di-n-butylamine	14	25-500	-
Paraldehyde	10	25-500	-
2-Picoline	7	25-500	-
1-Propanol	-	-	240
Propionitrile	11	25-500	200
Pyridine	4	25-500	-
o-Toluidine	13	25-500	-

<sup>a</sup> Produced by analysis of seven aliquots of reagent water spiked at 25 ppb at the listed compounds; calculations based on internal standard technique and use of the following equation:

$$\text{MDL} = 3.134 \times \text{Std. Dev. of low concentration spike (ppb)}.$$

<sup>b</sup> When a 40-mL sample is used, and the first 100 µL of distillate are collected.

TABLE 13

## TARGET COMPOUNDS, SURROGATES, AND INTERNAL STANDARDS (METHOD 5031)

Target Compound	Surrogate	Internal Standard
Acetone	d <sub>6</sub> -Acetone	d <sub>8</sub> -Isopropyl alcohol
Acetonitrile	d <sub>3</sub> -Acetonitrile	d <sub>8</sub> -Isopropyl alcohol
Acrylonitrile	d <sub>8</sub> -Isopropyl alcohol	
Allyl alcohol	d <sub>7</sub> -Dimethyl formamide	
Crotonaldehyde	d <sub>8</sub> -Isopropyl alcohol	
1,4-Dioxane	d <sub>8</sub> -1,4-Dioxane	d <sub>7</sub> -Dimethyl formamide
Isobutyl alcohol	d <sub>7</sub> -Dimethyl formamide	
Methanol	d <sub>3</sub> -Methanol	d <sub>8</sub> -Isopropyl alcohol
Methyl ethyl ketone	d <sub>8</sub> -Isopropyl alcohol	
N-Nitroso-di-n-butylamine	d <sub>7</sub> -Dimethyl formamide	
Paraldehyde	d <sub>7</sub> -Dimethyl formamide	
2-Picoline	d <sub>7</sub> -Dimethyl formamide	
Propionitrile	d <sub>8</sub> -Isopropyl alcohol	
Pyridine	d <sub>5</sub> -Pyridine	d <sub>7</sub> -Dimethyl formamide
o-Toluidine	d <sub>7</sub> -Dimethyl formamide	

TABLE 14

## RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/μL)
<b>Internal Standards</b>	
d <sub>5</sub> -benzyl alcohol	10.0
d <sub>14</sub> -Diglyme	10.0
d <sub>7</sub> -Dimethyl formamide	10.0
d <sub>8</sub> -Isopropyl alcohol	10.0
<b>Surrogates</b>	
d <sub>6</sub> -Acetone	10.0
d <sub>3</sub> -Acetonitrile	10.0
d <sub>8</sub> -1,4-Dioxane	10.0
d <sub>3</sub> -Methanol	10.0
d <sub>5</sub> -Pyridine	10.0
<b>Target Compounds</b>	
Acetone	1.0, 5.0, 10.0, 25.0, 100.0
Acetonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Acrylonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Allyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Crotonaldehyde	1.0, 5.0, 10.0, 25.0, 100.0
1,4-Dioxane	1.0, 5.0, 10.0, 25.0, 100.0
Isobutyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Methanol	1.0, 5.0, 10.0, 25.0, 100.0
Methyl ethyl ketone	1.0, 5.0, 10.0, 25.0, 100.0
N-Nitroso-di-n-butylamine	1.0, 5.0, 10.0, 25.0, 100.0
Paraldehyde	1.0, 5.0, 10.0, 25.0, 100.0
2-Picoline	1.0, 5.0, 10.0, 25.0, 100.0
Propionitrile	1.0, 5.0, 10.0, 25.0, 100.0
Pyridine	1.0, 5.0, 10.0, 25.0, 100.0
o-Toluidine	1.0, 5.0, 10.0, 25.0, 100.0

TABLE 15

## CHARACTERISTIC IONS AND RETENTION TIMES FOR VOCs (METHOD 5031)

Compound	Quantitation Ion <sup>a</sup>	Secondary Ions	Retention Time (min) <sup>b</sup>
Internal Standards			
d <sub>8</sub> -Isopropyl alcohol	49		1.75
d <sub>14</sub> -Diglyme	66	98,64	9.07
d <sub>7</sub> -Dimethyl formamide	50	80	9.20
Surrogates			
d <sub>6</sub> -Acetone	46	64,42	1.03
d <sub>3</sub> -Methanol	33	35,30	1.75
d <sub>3</sub> -Acetonitrile	44	42	2.63
d <sub>8</sub> -1,4-Dioxane	96	64,34	3.97
d <sub>5</sub> -Pyridine	84	56,79	6.73
d <sub>5</sub> -Phenol <sup>c</sup>	99	71	15.43
Target Compounds			
Acetone	43	58	1.05
Methanol	31	29	1.52
Methyl ethyl ketone	43	72,57	1.53
Methacrylonitrile <sup>c</sup>	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone <sup>c</sup>	85	100,58	2.78
Propionitrile	54	52,55	3.13
Crotonaldehyde	41	70	3.43
1,4-Dioxane	58	88,57	4.00
Paraldehyde	45	89	4.75
Isobutyl alcohol	43	33,42	5.05
Allyl alcohol	57	39	5.63
Pyridine	79	50,52	6.70
2-Picoline	93	66	7.27
N-Nitroso-di-n-butylamine	84	116	12.82
Aniline <sup>c</sup>	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol <sup>c</sup>	94	66,65	15.43

<sup>a</sup> These ions were used for quantitation in selected ion monitoring.

<sup>b</sup> GC column: DB-Wax, 30 meter x 0.53 mm, 1 µm film thickness.  
Oven program: 45°C for 4 min, increased to 220°C at 12°C/min.

<sup>c</sup> Compound removed from target analyte list due to poor accuracy and precision.

TABLE 16

METHOD ACCURACY AND PRECISION BY MEAN PERCENT RECOVERY AND PERCENT RELATIVE STANDARD DEVIATION<sup>a</sup> (METHOD 5031 - MACRODISTILLATION TECHNIQUE)  
(Single Laboratory and Single Operator)

Compound	25 ppb Spike		100 ppb Spike		500 ppb Spike	
	Mean %R	%RSD	Mean %R	%RSD	Mean %R	%RSD
d <sub>6</sub> -Acetone	66	24	69	14	65	16
d <sub>3</sub> -Acetonitrile	89	18	80	18	70	10
d <sub>8</sub> -1,4-Dioxane	56	34	58	11	61	18
d <sub>3</sub> -Methanol	43	29	48	19	56	14
d <sub>5</sub> -Pyridine	83	6.3	84	7.8	85	9.0
Acetone	67	45	63	14	60	14
Acetonitrile	44	35	52	15	56	15
Acrylonitrile	49	42	47	27	45	27
Allyl alcohol	69	13	70	9.7	73	10
Crotonaldehyde	68	22	68	13	69	13
1,4-Dioxane	63	25	55	16	54	13
Isobutyl alcohol	66	14	66	5.7	65	7.9
Methanol	50	36	46	22	49	18
Methyl ethyl ketone	55	37	56	20	52	19
N-Nitroso-di- n-butylamine	57	21	61	15	72	18
Paraldehyde	65	20	66	11	60	8.9
Picoline	81	12	81	6.8	84	8.0
Propionitrile	67	22	69	13	68	13
Pyridine	74	7.4	72	6.7	74	7.3
o-Toluidine	52	31	54	15	58	12

<sup>a</sup> Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrapole mass spectrometer in the selected ion monitoring mode.

TABLE 17

RECOVERIES IN SAND SAMPLES FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng)					Mean	RSD	Mean Rec
	1	2	3	4	5			
Vinyl chloride	8.0	7.5	6.7	5.4	6.6	6.8	13.0	34.2
Trichlorofluoromethane	13.3	16.5	14.9	13.0	10.3	13.6	15.2	68.0
1,1-Dichloroethene	17.1	16.7	15.1	14.8	15.6	15.9	5.7	79.2
Methylene chloride	24.5	22.7	19.7	19.4	20.6	21.4	9.1	107
trans-1,2-Dichloroethene	22.7	23.6	19.4	18.3	20.1	20.8	0.7	104
1,2-Dichloroethane	18.3	18.0	16.7	15.6	15.9	16.9	6.4	84.4
cis-1,2-Dichloroethene	26.1	23.1	22.6	20.3	20.8	22.6	9.0	113
Bromochloromethane	24.5	25.4	20.9	20.1	20.1	22.2	10.2	111
Chloroform	26.5	26.0	22.1	18.9	22.1	23.1	12.2	116
1,1,1-Trichloroethane	21.5	23.0	23.9	16.7	31.2	23.4	21.2	117
Carbon tetrachloride	23.6	24.2	22.6	18.3	23.3	22.4	9.4	112
Benzene	22.4	23.9	20.4	17.4	19.2	20.7	11.2	103
Trichloroethene	21.5	20.5	19.2	14.4	19.1	18.9	12.7	94.6
1,2-Dichloropropane	24.9	26.3	23.1	19.0	23.3	23.3	10.5	117
Dibromomethane	25.4	26.4	21.6	20.4	23.6	23.5	9.6	117
Bromodichloromethane	25.7	26.7	24.1	17.9	23.0	23.5	13.1	117
Toluene	28.3	25.0	24.8	16.3	23.6	23.6	16.9	118
1,1,2-Trichloroethane	25.4	24.5	21.6	17.7	22.1	22.2	12.1	111
1,3-Dichloropropane	25.4	24.2	22.7	17.0	22.2	22.3	12.8	112
Dibromochloromethane	26.3	26.2	23.7	18.2	23.2	23.5	12.5	118
Chlorobenzene	22.9	22.5	19.8	14.6	19.4	19.9	15.0	99.3
1,1,1,2-Tetrachloroethane	22.4	27.7	25.1	19.4	22.6	23.4	12.0	117
Ethylbenzene	25.6	25.0	22.1	14.9	24.0	22.3	17.5	112
p-Xylene	22.5	22.0	19.8	13.9	20.3	19.7	15.7	98.5
o-Xylene	24.2	23.1	21.6	14.0	20.4	20.7	17.3	103
Styrene	23.9	21.5	20.9	14.3	20.5	20.2	15.7	101
Bromoform	26.8	25.6	26.0	20.1	23.5	24.4	9.9	122
iso-Propylbenzene	25.3	25.1	24.2	15.4	24.6	22.9	16.6	114
Bromobenzene	19.9	21.8	20.0	15.5	19.1	19.3	10.7	96.3
1,2,3-Trichloropropane	25.9	23.0	25.6	15.9	21.4	22.2	15.8	111
n-Propylbenzene	26.0	23.8	22.6	13.9	21.9	21.6	19.0	106
2-Chlorotoluene	23.6	23.8	21.3	13.0	21.5	20.6	19.2	103
4-Chlorotoluene	21.0	19.7	18.4	12.1	18.3	17.9	17.1	89.5
1,3,5-Trimethylbenzene	24.0	22.1	22.5	13.8	22.9	21.1	17.6	105
sec-Butylbenzene	25.9	25.3	27.8	16.1	28.6	24.7	18.1	124
1,2,4-Trimethylbenzene	30.6	39.2	22.4	18.0	22.7	26.6	28.2	133
1,3-Dichlorobenzene	20.3	20.6	18.2	13.0	17.6	17.9	15.2	89.7
p-iso-Propyltoluene	21.6	22.1	21.6	16.0	22.8	20.8	11.8	104
1,4-Dichlorobenzene	18.1	21.2	20.0	13.2	17.4	18.0	15.3	90.0
1,2-Dichlorobenzene	18.4	22.5	22.5	15.2	19.9	19.7	13.9	96.6
n-Butylbenzene	13.1	20.3	19.5	10.8	18.7	16.5	23.1	82.4
1,2,4-Trichlorobenzene	14.5	14.9	15.7	8.8	12.3	13.3	18.8	66.2
Hexachlorobutadiene	17.6	22.5	21.6	13.2	21.6	19.3	18.2	96.3
1,2,3-Trichlorobenzene	14.9	15.9	16.5	11.9	13.9	14.6	11.3	73.1

Data in Tables 17, 18, and 19 are from Reference 15.

TABLE 18  
RECOVERIES IN C-HORIZON SOILS FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng)					Mean	RSD	Mean Rec
	1	2	3	4	5			
Vinyl chloride	33.4	31.0	30.9	29.7	28.6	30.8	5.2	154
Trichlorofluoromethane	37.7	20.8	20.0	21.8	20.5	24.1	28.2	121
1,1-Dichloroethene	21.7	33.5	39.8	30.2	32.5	31.6	18.5	158
Methylene chloride	20.9	19.4	18.7	18.3	18.4	19.1	5.1	95.7
trans-1,2-Dichloroethene	21.8	18.9	20.4	17.9	17.8	19.4	7.9	96.8
1,1-Dichloroethane	23.8	21.9	21.3	21.3	20.5	21.8	5.2	109
cis-1,2-Dichloroethene	21.6	18.8	18.5	18.2	18.2	19.0	6.7	95.2
Bromochloromethane	22.3	19.5	19.3	19.0	19.2	20.0	6.0	100
Chloroform	20.5	17.1	17.3	16.5	15.9	17.5	9.2	87.3
1,1,1-Trichloroethane	16.4	11.9	10.7	9.5	9.4	11.6	22.4	57.8
Carbon tetrachloride	13.1	11.3	13.0	11.8	11.2	12.1	6.7	60.5
Benzene	21.1	19.3	18.7	18.2	16.9	18.8	7.4	94.1
Trichloroethene	19.6	16.4	16.5	16.5	15.5	16.9	8.3	84.5
1,2-Dichloropropane	21.8	19.0	18.3	18.8	16.5	18.9	9.0	94.4
Dibromomethane	20.9	17.9	17.9	17.2	18.3	18.4	6.9	92.1
Bromodichloromethane	20.9	18.0	18.9	18.2	17.3	18.6	6.6	93.2
Toluene	22.2	17.3	18.8	17.0	15.9	18.2	12.0	91.2
1,1,2-Trichloroethane	21.0	16.5	17.2	17.2	16.5	17.7	9.6	88.4
1,3-Dichloropropane	21.4	17.3	18.7	18.6	16.7	18.5	8.8	92.6
Dibromochloromethane	20.9	18.1	19.0	18.8	16.6	18.7	7.5	93.3
Chlorobenzene	20.8	18.4	17.6	16.8	14.8	17.7	11.2	88.4
1,1,1,2-Tetrachloroethane	19.5	19.0	17.8	17.2	16.5	18.0	6.2	90.0
Ethylbenzene	21.1	18.3	18.5	16.9	15.3	18.0	10.6	90.0
p-Xylene	20.0	17.4	18.2	16.3	14.4	17.3	10.9	86.3
o-Xylene	20.7	17.2	16.8	16.2	14.8	17.1	11.4	85.7
Styrene	18.3	15.9	16.2	15.3	13.7	15.9	9.3	79.3
Bromoform	20.1	15.9	17.1	17.5	16.1	17.3	8.6	86.7
iso-Propylbenzene	21.0	18.1	19.2	18.4	15.6	18.4	9.6	92.2
Bromobenzene	20.4	16.2	17.2	16.7	15.4	17.2	10.1	85.9
1,1,2,2-Tetrachloroethane	23.3	17.9	21.2	18.8	16.8	19.6	12.1	96.0
1,2,3-Trichloropropane	18.4	14.6	15.6	16.1	15.6	16.1	8.0	80.3
n-Propylbenzene	20.4	18.9	17.9	17.0	14.3	17.7	11.6	88.4
2-Chlorotoluene	19.1	17.3	16.1	16.0	14.4	16.7	9.2	83.6
4-Chlorotoluene	19.0	15.5	16.8	15.9	13.6	16.4	10.6	81.8
1,3,5-Trimethylbenzene	20.8	18.0	17.4	16.1	14.7	17.4	11.7	86.9
sec-Butylbenzene	21.4	18.3	18.9	17.0	14.9	18.1	11.8	90.5
1,2,4-Trimethylbenzene	20.5	18.6	16.8	15.3	13.7	17.0	14.1	85.0
1,3-Dichlorobenzene	17.6	15.9	15.6	14.2	14.4	15.6	7.9	77.8
p-iso-Propyltoluene	20.5	17.0	17.1	15.6	13.4	16.7	13.9	83.6
1,4-Dichlorobenzene	18.5	13.8	14.8	16.7	14.9	15.7	10.5	78.7
1,2-Dichlorobenzene	18.4	15.0	15.4	15.3	13.5	15.5	10.5	77.6
n-Butylbenzene	19.6	15.9	15.9	14.4	18.9	16.9	11.7	84.6
1,2,4-Trichlorobenzene	15.2	17.2	17.4	13.6	12.1	15.1	13.5	75.4
Hexachlorobutadiene	18.7	16.2	15.5	13.8	16.6	16.1	10.0	80.7
Naphthalene	13.9	11.1	10.2	10.8	11.4	11.5	11.0	57.4
1,2,3-Trichlorobenzene	14.9	15.2	16.8	13.7	12.7	14.7	9.5	73.2



TABLE 19  
RECOVERIES IN GARDEN SOIL FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng)					Mean	RSD	Mean Rec
	1	2	3	4	5			
Vinyl chloride	12.7	10.9	9.8	8.1	7.2	9.7	20.2	48.7
Trichlorofluoromethane	33.7	6.4	30.3	27.8	22.9	24.2	39.6	121
1,1-Dichloroethene	27.7	20.5	24.1	15.1	13.2	20.1	26.9	101
Methylene chloride	25.4	23.9	24.7	22.2	24.2	24.1	4.4	120
trans-1,2-Dichloroethene	2.8	3.0	3.3	2.2	2.4	2.7	15.0	13.6
1,1-Dichloroethane	24.1	26.3	27.0	20.5	21.2	23.8	11.0	119
cis-1,2-Dichloroethene	8.3	10.2	8.7	5.8	6.4	7.9	20.1	39.4
Bromochloromethane	11.1	11.8	10.2	8.8	9.0	10.2	11.2	50.9
Chloroform	16.7	16.9	17.0	13.8	15.0	15.9	7.9	79.3
1,1,1-Trichloroethane	24.6	22.8	22.1	16.2	20.9	21.3	13.4	107
Carbon tetrachloride	19.4	20.3	22.2	20.0	20.2	20.4	4.6	102
Benzene	21.4	22.0	22.4	19.6	20.4	21.2	4.9	106
Trichloroethene	12.4	16.5	14.9	9.0	9.9	12.5	22.9	62.7
1,2-Dichloropropane	19.0	18.8	19.7	16.0	17.6	18.2	7.1	91.0
Dibromomethane	7.3	8.0	6.9	5.6	6.8	6.9	11.3	34.6
Bromodichloromethane	14.9	15.9	15.9	12.8	13.9	14.7	8.3	73.3
Toluene	42.6	39.3	45.1	39.9	45.3	42.4	5.9	212
1,1,2-Trichloroethane	13.9	15.2	1.4	21.3	14.9	15.9	17.0	79.6
1,3-Dichloropropane	13.3	16.7	11.3	10.9	9.5	12.3	20.3	61.7
Dibromochloromethane	14.5	13.1	14.5	11.9	14.4	13.7	7.6	68.3
Chlorobenzene	8.4	10.0	8.3	6.9	7.8	8.3	12.1	41.3
1,1,1,2-Tetrachloroethane	16.7	16.7	15.6	15.8	15.7	16.1	3.2	80.4
Ethylbenzene	22.1	21.4	23.1	20.1	22.6	21.9	4.8	109
p-Xylene	41.4	38.4	43.8	38.3	44.0	41.2	6.1	206
o-Xylene	31.7	30.8	34.3	30.4	33.2	32.1	4.6	160
Styrene	0	0	0	0	0	0	0	0
Bromoform	8.6	8.9	9.1	7.0	7.7	8.3	9.4	41.4
iso-Propylbenzene	18.1	18.8	9.7	18.3	19.6	18.9	3.5	94.4
Bromobenzene	5.1	5.4	5.3	4.4	4.0	4.8	11.6	24.1
1,1,2,2-Tetrachloroethane	14.0	13.5	14.7	15.3	17.1	14.9	8.5	74.5
1,2,3-Trichloropropane	11.0	12.7	11.7	11.7	11.9	11.8	4.5	59.0
n-Propylbenzene	13.4	13.3	14.7	12.8	13.9	13.6	4.7	68.1
2-Chlorotoluene	8.3	9.0	11.7	8.7	7.9	9.1	14.8	45.6
4-Chlorotoluene	5.1	5.4	5.5	4.8	4.5	5.0	7.9	25.2
1,3,5-Trimethylbenzene	31.3	27.5	33.0	31.1	33.6	31.3	6.8	157
sec-Butylbenzene	13.5	13.4	16.4	13.8	15.4	14.5	8.3	72.5
1,2,4-Trimethylbenzene	38.7	32.4	40.8	34.1	40.3	37.3	9.1	186
1,3-Dichlorobenzene	3.6	3.6	3.7	3.0	3.2	3.4	8.0	17.2
p-iso-Propyltoluene	14.7	14.1	16.1	13.9	15.1	14.8	5.2	73.8
1,4-Dichlorobenzene	3.0	3.5	3.3	2.6	2.8	3.0	10.2	15.0
1,2-Dichlorobenzene	3.6	4.3	4.0	3.5	3.6	3.8	8.3	19.0
n-Butylbenzene	17.4	13.8	14.0	18.9	24.0	17.6	21.2	88.0
1,2,4-Trichlorobenzene	2.8	2.9	3.3	2.6	3.2	3.0	8.5	15.0
Hexachlorobutadiene	4.8	4.0	6.1	5.6	6.0	5.3	15.1	26.4
Naphthalene	5.5	5.1	5.5	4.7	5.6	5.3	6.2	26.5
1,2,3-Trichlorobenzene	2.2	2.3	2.4	2.2	2.3	2.3	3.5	11.4

Data in Table 19 are from Reference 15.

TABLE 20

VOLATILE ORGANIC ANALYTE RECOVERY FROM SOIL  
USING VACUUM DISTILLATION (METHOD 5032)<sup>a</sup>

Compound	Soil/H <sub>2</sub> O <sup>b</sup> Recovery		Soil/Oil <sup>c</sup> Recovery		Soil/Oil/H <sub>2</sub> O Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	61	20	40	18	108	68
Bromomethane	58	20	47	13	74	13
Vinyl chloride	54	12	46	11	72	20
Chloroethane	46	10	41	8	52	14
Methylene chloride	60	2	65	8	76	11
Acetone	INT <sup>e</sup>	INT	44	8		
Carbon disulfide	47	13	53	10	47	4
1,1-Dichloroethene	48	9	47	5	58	3
1,1-Dichloroethane	61	6	58	9	61	6
trans-1,2-Trichloroethane	54	7	60	7	56	5
cis-1,2-Dichloroethene	60	4	72	6	63	8
Chloroform	104	11	93	6	114	15
1,2-Dichloroethane	177	50	117	8	151	22
2-Butanone	INT	36	38	INT		
1,1,1-Trichloroethane	124	13	72	16	134	26
Carbon tetrachloride	172	122	INT	INT		
Vinyl acetate	88	11	INT			
Bromodichloromethane	93	4	91	23	104	23
1,1,2,2-Tetrachloroethane	96	13	50	12	104	7
1,2-Dichloropropane	105	8	102	6	111	6
trans-1,3-Dichloropropene	134	10	84	16	107	8
Trichloroethene	98	9	99	10	100	5
Dibromochloromethane	119	8	125	31	142	16
1,1,2-Trichloroethane	126	10	72	16	97	4
Benzene	99	7	CONT <sup>f</sup>	CONT		
cis-1,3-Dichloropropene	123	12	94	13	112	9
Bromoform	131	13	58	18	102	9
2-Hexanone	155	18	164	19	173	29
4-Methyl-2-pentanone	152	20	185	20	169	18
Tetrachloroethene	90	9	123	14	128	7
Toluene	94	3	CONT	CONT		
Chlorobenzene	98	7	93	18	112	5
Ethylbenzene	114	13	CONT	CONT		
Styrene	106	8	93	18	112	5
p-Xylene	97	9	CONT	CONT		
o-Xylene	105	8	112	12	144	13

TABLE 20 (cont.)

Compound	Soil/H <sub>2</sub> O <sup>b</sup> Recovery		Soil/Oil <sup>c</sup> Recovery		Soil/Oil/H <sub>2</sub> O Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Surrogates						
1,2-Dichloroethane	177	50	117	8	151	22
Toluene-d <sub>8</sub>	96	6	79	12	82	6
Bromofluorobenzene	139	13	37	13	62	5

<sup>a</sup> Results are for 10 min. distillations times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision value reflects the propagated errors. Each analyte was spiked at 50 ppb. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may introduce bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

<sup>b</sup> Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

<sup>c</sup> Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

<sup>d</sup> Soil samples + 1 g cod liver oil, spiked as above with 5 mL of water added to make slurry.

<sup>e</sup> Interference by co-eluting compounds prevented accurate measurement of analyte.

<sup>f</sup> Contamination of sample matrix by analyte prevented assessment of efficiency.

TABLE 21

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES  
IN FISH TISSUE (METHOD 5032)<sup>a</sup>

Compound	Efficiency	
	Mean (%)	RSD (%)
Chloromethane	N/A <sup>b</sup>	
Bromomethane	N/A <sup>b</sup>	
Vinyl chloride	N/A <sup>b</sup>	
Chloroethane	N/A <sup>b</sup>	
Methylene chloride	CONT <sup>c</sup>	
Acetone	CONT <sup>c</sup>	
Carbon disulfide	79	36
1,1-Dichloroethene	122	39
1,1-Dichloroethane	126	35
trans-1,2-Trichloroethene	109	46
cis-1,2-Dichloroethene	106	22
Chloroform	111	32
1,2-Dichloroethane	117	27
2-Butanone	INT <sup>d</sup>	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT <sup>d</sup>	
Bromodichloromethane	97	22
1,1,2,2-Tetrachloroethane	67	20
1,2-Dichloropropane	117	23
trans-1,3-Dichloropropene	92	22
Trichloroethene	98	31
Dibromochloromethane	71	19
1,1,2-Trichloroethane	92	20
Benzene	129	35
cis-1,3-Dichloropropene	102	24
Bromoform	58	19
2-Hexanone	INT <sup>d</sup>	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT <sup>c</sup>	
Chlorobenzene	65	19
Ethylbenzene	74	19
Styrene	57	14
p-Xylene	46	13
o-Xylene	83	20

TABLE 21 (cont.)

Compound	Efficiency	
	Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	115	27
Toluene-d <sub>8</sub>	88	24
Bromofluorobenzene	52	15

<sup>a</sup> Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicate 10-g aliquots of fish spiked at 25 ppb were analyzed using GC/MS external standard quantitation. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards were replicated and results reflect 1 sigma propagated standard deviation.

<sup>b</sup> No analyses.

<sup>c</sup> Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.

<sup>d</sup> Interfering by co-eluting compounds prevented accurate measurement of analyte.

TABLE 22

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES  
IN FISH TISSUE (METHOD 5032)<sup>a</sup>

Compound	Method Detection Limit (ppb)	
	External Standard Method	Internal Standard Method
Chloromethane	7.8	7.3
Bromomethane	9.7	9.8
Vinyl chloride	9.5	9.4
Chloroethane	9.2	10.0
Methylene chloride	CONT <sup>b</sup>	CONT <sup>b</sup>
Acetone	CONT <sup>b</sup>	CONT <sup>b</sup>
Carbon disulfide	5.4	4.9
1,1-Dichloroethene	4.0	5.7
1,1-Dichloroethane	4.0	3.5
trans-1,2-Dichloroethene	4.4	4.0
cis-1,2-Dichloroethene	4.7	4.1
Chloroform	5.6	5.0
1,2-Dichloroethane	3.3	3.2
2-Butanone	INT <sup>c</sup>	INT <sup>c</sup>
1,1,1-Trichloroethane	1.1	4.2
Carbon tetrachloride	3.2	3.5
Vinyl acetate	INT <sup>c</sup>	INT <sup>c</sup>
Bromodichloromethane	3.2	2.8
1,1,2,2-Tetrachloroethane	4.4	3.8
1,2-Dichloropropane	3.8	3.7
trans-1,3-Dichloropropene	3.4	3.0
Trichloroethene	3.1	4.0
Dibromochloromethane	3.5	3.2
1,1,2-Trichloroethane	4.4	3.3
Benzene	3.6	3.2
cis-1,3-Dichloropropene	3.5	3.0
Bromoform	4.9	4.0
2-Hexanone	7.7	8.0
4-Methyl-2-pentanone	7.5	8.0
Tetrachloroethene	4.3	4.0
Toluene	3.0	2.5
Chlorobenzene	3.3	2.8
Ethylbenzene	3.6	3.5
Styrene	3.5	3.3
p-Xylene	3.7	3.5
o-Xylene	3.3	4.7

Footnotes are on the following page.

TABLE 22 (cont.)

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- <sup>a</sup> Values shown are the average MDLs for studies on three non-consecutive days, involving seven replicate analyses of 10 g of fish tissue spiked a 5 ppb. Daily MDLs were calculated as three times the standard deviation. Quantitation was performed by GC/MS Method 8260 and separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.
- <sup>b</sup> Contamination of sample by analyte prevented determination.
- <sup>c</sup> Interference by co-eluting compounds prevented accurate quantitation.

TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER  
USING VACUUM DISTILLATION (METHOD 5032)<sup>a</sup>

Compound	5 mL H <sub>2</sub> O <sup>b</sup> Recovery		20 mL H <sub>2</sub> O <sup>c</sup> Recovery		20 mL H <sub>2</sub> O/Oil Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	114	27	116	29	176	67
Bromomethane	131	14	121	14	113	21
Vinyl chloride	131	13	120	16	116	23
Chloroethane	110	15	99	8	96	16
Methylene chloride	87	16	105	15	77	6
Acetone	83	22	65	34	119	68
Carbon disulfide	138	17	133	23	99	47
1,1-Dichloroethene	105	11	89	4	96	18
1,1-Dichloroethane	118	10	119	11	103	25
trans-1,2-Dichloroethene	105	11	107	14	96	18
cis-1,2-Dichloroethene	106	7	99	5	104	23
Chloroform	114	6	104	8	107	21
1,2-Dichloroethane	104	6	109	8	144	19
2-Butanone	83	50	106	31	INT <sup>c</sup>	
1,1,1-Trichloroethane	118	9	109	9	113	23
Carbon tetrachloride	102	6	108	12	109	27
Vinyl acetate	90	16	99	7	72	36
Bromodichloromethane	104	3	110	5	99	5
1,1,2,2-Tetrachloroethane	85	17	81	7	111	43
1,2-Dichloropropane	100	6	103	2	104	7
trans-1,3-Dichloropropene	105	8	105	4	92	4
Trichloroethene	98	4	99	2	95	5
Dibromochloroethane	99	8	99	6	90	25
1,1,2-Trichloroethane	98	7	100	4	76	12
Benzene	97	4	100	5	112	10
cis-1,3-Dichloropropene	106	5	105	4	98	3
Bromoform	93	16	94	8	57	21
2-Hexanone	60	17	63	16	78	23
4-Methyl-2-pentanone	79	24	63	14	68	15
Tetrachloroethene	101	3	97	7	77	14
Toluene	100	6	97	8	85	5
Chlorobenzene	98	6	98	4	88	16
Ethylbenzene	100	3	92	8	73	13
Styrene	98	4	97	9	88	16
p-Xylene	96	4	94	8	60	12
o-Xylene	96	7	95	6	72	14



TABLE 23 (cont.)

Compound	5 mL H <sub>2</sub> O <sup>b</sup> Recovery		20 mL H <sub>2</sub> O <sup>c</sup> Recovery		20 mL H <sub>2</sub> O/Oil Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Surrogates						
1,2-Dichloroethane	104	6	109	6	144	19
Toluene-d <sub>8</sub>	104	5	102	2	76	7
Bromofluorobenzene	106	6	106	9	40	8

<sup>a</sup> Results are for 10 min. distillation times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision values reflect the propagated errors. Concentrations of analytes were 50 ppb for 5-mL samples and 25 ppb for 20-mL samples. Recovery data generated with comparison to analyses of standards without the water matrix.

<sup>b</sup> Sample contained 1 gram cod liver oil and 20 mL water. An emulsion was created by adding 0.2 mL of water saturated with lecithin.

<sup>c</sup> Interference by co-eluting compounds prevented accurate assessment of recovery.

TABLE 24

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES  
USING VACUUM DISTILLATION (METHOD 5032) (INTERNAL STANDARD METHOD)<sup>a</sup>

Compound	Water <sup>b</sup> (µg/L)	Soil <sup>c</sup> (µg/kg)	Tissue <sup>d</sup> (µg/kg)	Oil <sup>e</sup> (mg/kg)
Chloromethane	3.2	8.0	7.3	N/A <sup>f</sup>
Bromomethane	2.8	4.9	9.8	N/A <sup>f</sup>
Vinyl chloride	3.5	6.0	9.4	N/A <sup>f</sup>
Chloroethane	5.9	6.0	10.0	N/A <sup>f</sup>
Methylene chloride	3.1	4.0	CONT <sup>g</sup>	0.05
Acetone	5.6	CONT <sup>g</sup>	CONT <sup>g</sup>	0.06
Carbon disulfide	2.5	2.0	4.9	0.18
1,1-Dichloroethene	2.9	3.2	5.7	0.18
1,1-Dichloroethane	2.2	2.0	3.5	0.14
trans-1,2-Dichloroethene	2.2	1.4	4.0	0.10
cis-1,2-Dichloroethene	2.0	2.3	4.1	0.07
Chloroform	2.4	1.8	5.0	0.07
1,2-Dichloroethane	1.7	1.5	3.2	0.06
2-Butanone	7.4	INT <sup>h</sup>	INT <sup>h</sup>	INT <sup>h</sup>
1,1,1-Trichloroethane	1.8	1.7	4.2	0.10
Carbon tetrachloride	1.4	1.5	3.5	0.13
Vinyl acetate	11.8	INT <sup>h</sup>	INT <sup>h</sup>	INT <sup>h</sup>
Bromodichloromethane	1.6	1.4	2.8	0.06
1,1,2,2-Tetrachloroethane	2.5	2.1	3.8	0.02
1,2-Dichloropropane	2.2	2.1	3.7	0.15
trans-1,3-Dichloropropene	1.5	1.7	3.0	0.05
Trichloroethene	1.6	1.7	4.0	0.04
Dibromochloromethane	1.7	1.5	3.2	0.07
1,1,2-Trichloroethane	2.1	1.7	3.3	0.05
Benzene	0.5	1.5	3.2	0.05
cis-1,3-Dichloropropene	1.4	1.7	3.0	0.04
Bromoform	1.8	1.5	4.0	0.05
2-Hexanone	4.6	3.6	8.0	INT <sup>h</sup>
4-Methyl-2-pentanone	3.5	4.6	8.0	INT <sup>h</sup>
Tetrachloroethene	1.4	1.6	4.0	0.10
Toluene	1.0	3.3	2.5	0.05
Chlorobenzene	1.4	1.4	2.8	0.06
Ethylbenzene	1.5	2.8	3.5	0.04
Styrene	1.4	1.4	3.3	0.18
p-Xylene	1.5	2.9	3.5	0.20
o-Xylene	1.7	3.4	4.7	0.07

Footnotes are found on the following page.

TABLE 24 (cont.)

- 
- a Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Method detection limits are the average MDLs for studies on three non-consecutive days.
  - b Method detection limits are the average MDLs for studies of three non-consecutive days. Daily studies were seven replicated analyses of 5 mL aliquots of 4 ppb soil. Daily MDLs were three times the standard deviation.
  - c Daily studies were seven replicated analyses of 10 g fish tissue spiked at 5 ppb. Daily MDLs were three times the standard deviation. Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.
  - d Method detection limits are estimated analyzing 1 g of cod liver oil samples spiked at 250 ppm. Five replicates were analyzed using Method 8260.
  - e No analyses.
  - f Contamination of sample by analyte prevented determination.
  - g Interference by co-eluting compounds prevented accurate quantitation.

TABLE 25

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES  
(METHOD 5032) (EXTERNAL STANDARD METHOD)<sup>a</sup>

Compound	Water <sup>b</sup> (µg/L)	Soil <sup>c</sup> (µg/kg)	Tissue <sup>d</sup> (µg/kg)	Oil <sup>e</sup> (mg/kg)
Chloromethane	3.1	8.6 <sup>f</sup>	7.8	N/A <sup>g</sup>
Bromomethane	2.5	4.9 <sup>f</sup>	9.7	N/A <sup>g</sup>
Vinyl chloride	4.0	7.1 <sup>f</sup>	9.5	N/A <sup>g</sup>
Chloroethane	6.1	7.5 <sup>f</sup>	9.2	N/A <sup>g</sup>
Methylene chloride	3.1	3.3	CONT <sup>h</sup>	0.08
Acetone	33.0 <sup>f</sup>	CONT <sup>h</sup>	CONT <sup>h</sup>	0.12
Carbon disulfide	2.5	3.2	5.4	0.19
1,1-Dichloroethene	3.4	3.8	4.0	0.19
1,1-Dichloroethane	2.3	1.7	4.0	0.13
trans-1,2-Dichloroethene	3.0	3.2	4.4	0.09
cis-1,2-Dichloroethene	2.4	2.7	4.7	0.08
Chloroform	2.7	2.6	5.6	0.06
1,2-Dichloroethane	1.6	1.7	3.3	0.06
2-Butanone	57.0 <sup>f</sup>	INT <sup>i</sup>	INT <sup>i</sup>	INT <sup>i</sup>
1,1,1-Trichloroethane	1.6	2.4	1.1	0.08
Carbon tetrachloride	1.5	1.7	3.2	0.15
Vinyl acetate	23.0 <sup>f</sup>	INT <sup>i</sup>	INT <sup>i</sup>	INT <sup>i</sup>
Bromodichloromethane	2.0	2.3	3.2	0.05
1,1,2,2-Tetrachloroethane	3.6	3.2	4.4	0.09
1,2-Dichloropropane	2.9	3.7	3.8	0.12
trans-1,3-Dichloropropene	2.3	2.4	3.8	0.08
Trichloroethene	2.5	3.0	3.1	0.06
Dibromochloromethane	2.1	2.9	3.5	0.04
1,1,2-Trichloroethane	2.7	2.8	4.4	0.07
Benzene	1.7	2.9	3.6	0.03
cis-1,3-Dichloropropene	2.1	2.5	3.5	0.06
Bromoform	2.3	2.5	4.9	0.10
2-Hexanone	4.6	4.6	7.7	INT <sup>i</sup>
4-Methyl-2-pentanone	3.8	3.9	7.5	INT <sup>i</sup>
Tetrachloroethene	1.8	2.6	4.3	0.12
Toluene	1.8	4.4	3.0	0.09
Chlorobenzene	2.4	2.6	3.3	0.07
Ethylbenzene	2.4	4.1	3.6	0.09
Styrene	2.0	2.5	3.5	0.16
p-Xylene	2.3	3.9	3.7	0.18
o-Xylene	2.4	4.1	3.3	0.08

TABLE 25 (cont.)

- 
- <sup>a</sup> Method detection limits are the average MDLs for studies on three non-consecutive days. Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb. Daily MDLs were three times the standard deviation.
- <sup>b</sup> Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb.
- <sup>c</sup> These studies were seven replicate analyses of 5-g aliquots of soil spiked at 4 ppb.
- <sup>d</sup> These studies were seven replicate analyses of 10-g aliquots of fish tissue spiked at 5 ppb.
- <sup>e</sup> Method detection limits were estimated by analyzing cod liver oil samples spiked at 250 ppb. Five replicates were analyzed using Method 8260.
- <sup>f</sup> Method detection limits were estimated by analyzing replicate 50 ppb standards five times over a single day.
- <sup>g</sup> No analyses.
- <sup>h</sup> Contamination of sample by analyte prevented determination.
- <sup>i</sup> Interference by co-eluting compound prevented accurate quantitation.

TABLE 26

VOLATILE ORGANIC ANALYTE RECOVERY FROM OIL  
USING VACUUM DISTILLATION (METHOD 5032)<sup>a</sup>

Compound	Recovery	
	Mean (%)	RSD (%)
Chloromethane	N/A <sup>b</sup>	
Bromomethane	N/A <sup>b</sup>	
Vinyl chloride	N/A <sup>b</sup>	
Chloroethane	N/A <sup>b</sup>	
Methylene chloride	62	32
Acetone	108	55
Carbon disulfide	98	46
1,1-Dichloroethene	97	24
1,1-Dichloroethane	96	22
trans-1,2-Trichloroethene	86	23
cis-1,2-Dichloroethene	99	11
Chloroform	93	14
1,2-Dichloroethane	138	31
2-Butanone	INT <sup>c</sup>	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT <sup>c</sup>	
Bromodichloromethane	106	14
1,1,2,2-Tetrachloroethane	205	46
1,2-Dichloropropane	107	24
trans-1,3-Dichloropropene	98	13
Trichloroethene	102	8
Dibromochloromethane	168	21
1,1,2-Trichloroethane	95	7
Benzene	146	10
cis-1,3-Dichloropropene	98	11
Bromoform	94	18
2-Hexanone	INT <sup>c</sup>	
4-Methyl-2-pentanone	INT <sup>c</sup>	
Tetrachloroethene	117	22
Toluene	108	8
Chlorobenzene	101	12
Ethylbenzene	96	10
Styrene	120	46
p-Xylene	87	23
o-Xylene	90	10

TABLE 26 (cont.)

Compound	Recovery	
	Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	137	30
Toluene-d <sub>8</sub>	84	6
Bromofluorobenzene	48	2

<sup>a</sup> Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish aliquots spiked at 25 ppb were analyzed. Quantitation was performed with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Standards and samples were replicated and precision value reflects the propagated errors. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

<sup>b</sup> Not analyzed.

<sup>c</sup> Interference by co-evaluating compounds prevented accurate measurement of analyte.

TABLE 27

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES  
IN OIL (METHOD 5032)<sup>a</sup>

Compound	Method Detection Limit (ppb)	
	External Standard Method	Internal Standard Method
Chloromethane	N/A <sup>b</sup>	N/A <sup>b</sup>
Bromomethane	N/A <sup>b</sup>	N/A <sup>b</sup>
Vinyl chloride	N/A <sup>b</sup>	N/A <sup>b</sup>
Chloroethane	N/A <sup>b</sup>	N/A <sup>b</sup>
Methylene chloride	80	50
Acetone	120	60
Carbon disulfide	190	180
1,1-Dichloroethene	190	180
1,1-Dichloroethane	130	140
trans-1,2-Dichloroethene	90	100
cis-1,2-Dichloroethene	80	70
Chloroform	60	70
1,2-Dichloroethane	60	60
2-Butanone	INT <sup>c</sup>	INT <sup>c</sup>
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT <sup>c</sup>	INT <sup>c</sup>
Bromodichloromethane	50	60
1,1,2,2-Tetrachloroethane	90	20
1,2-Dichloropropane	120	150
trans-1,3-Dichloropropene	80	50
Trichloroethene	60	40
Dibromochloromethane	40	70
1,1,2-Trichloroethane	70	50
Benzene	30	50
cis-1,3-Dichloropropene	60	40
Bromoform	100	50
2-Hexanone	INT <sup>c</sup>	INT <sup>c</sup>
4-Methyl-2-pentanone	INT <sup>c</sup>	INT <sup>c</sup>
Tetrachloroethene	120	100
Toluene	90	50
Chlorobenzene	70	60
Ethylbenzene	90	40
Styrene	160	180
p-Xylene	180	200
o-Xylene	80	70



TABLE 27 (cont.)

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- <sup>a</sup> Method detection limits are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MDLs were calculated as three times the standard deviation. Quantitation was performed using a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.
- <sup>b</sup> No analyses.
- <sup>c</sup> Interference by co-eluting compounds prevented accurate quantitation.

TABLE 28

INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS  
(METHOD 5021)

Chloroform-d <sub>1</sub>	1,1,2-TCA-d <sub>3</sub>	Bromobenzene-d <sub>5</sub>
Dichlorodifluoromethane	1,1,1-Trichloroethane	Chlorobenzene
Chloromethane	1,1-Dichloropropene	Bromoform
Vinyl chloride	Carbon tetrachloride	Styrene
Bromomethane	Benzene	iso-Propylbenzene
Chloroethane	Dibromomethane	Bromobenzene
Trichlorofluoromethane	1,2-Dichloropropane	n-Propylbenzene
1,1-Dichloroethene	Trichloroethene	2-Chlorotoluene
Methylene chloride	Bromodichloromethane	4-Chlorotoluene
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
1,1-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
cis-1,2-Dichloroethene	1,1,2-Trichloroethane	1,2,4-Trimethylbenzene
Bromochloromethane	Toluene	sec-Butylbenzene
Chloroform	1,3-Dichloropropane	1,3-Dichlorobenzene
2,2-Dichloropropane	Dibromochloromethane	1,4-Dichlorobenzene
1,2-Dichloroethane	1,2-Dibromoethane	p-iso-Propyltoluene
	Tetrachloroethene	1,2-Dichlorobenzene
	1,1,2-Trichloroethane	n-Butylbenzene
	Ethylbenzene	1,2-Dibromo-3-chloropropane
	m-Xylene	1,2,4-Trichlorobenzene
	p-Xylene	Naphthalene
	o-Xylene	Hexachlorobutadiene
	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
	1,2,3-Trichloropropane	

TABLE 29

PRECISION AND MDL DETERMINED FOR ANALYSIS OF FORTIFIED SAND<sup>a</sup> (METHOD 5021)

Compound	% RSD	MDL ( $\mu\text{g}/\text{kg}$ )
Benzene	3.0	0.34
Bromochloromethane	3.4	0.27
Bromodichloromethane	2.4	0.21
Bromoform	3.9	0.30
Bromomethane	11.6	1.3
Carbon tetrachloride	3.6	0.32
Chlorobenzene	3.2	0.24
Chloroethane	5.6	0.51
Chloroform	3.1	0.30
Chloromethane	4.1	3.5 <sup>b</sup>
1,2-Dibromo-3-chloropropane	5.7	0.40
1,2-Dibromoethane	3.2	0.29
Dibromomethane	2.8	0.20
1,2-Dichlorobenzene	3.3	0.27
1,3-Dichlorobenzene	3.4	0.24
1,4-Dichlorobenzene	3.7	0.30
Dichlorodifluoromethane	3.0	0.28
1,1-Dichloroethane	4.5	0.41
1,2-Dichloroethane	3.0	0.24
1,1-Dichloroethene	3.3	0.28
cis-1,2-Dichloroethene	3.2	0.27
trans-1,2-Dichloroethene	2.6	0.22
1,2-Dichloropropane	2.6	0.21
1,1-Dichloropropene	3.2	0.30
cis-1,3-Dichloropropene	3.4	0.27
Ethylbenzene	4.8	0.47
Hexachlorobutadiene	4.1	0.38
Methylene chloride	8.2	0.62 <sup>c</sup>
Naphthalene	16.8	3.4 <sup>c</sup>
Styrene	7.9	0.62
1,1,1,2-Tetrachloroethane	3.6	0.27
1,1,2,2-Tetrachloroethane	2.6	0.20
Tetrachloroethene	9.8	1.2 <sup>c</sup>
Toluene	3.5	0.38
1,2,4-Trichlorobenzene	4.2	0.44
1,1,1-Trichloroethane	2.7	0.27
1,1,2-Trichloroethane	2.6	0.20
Trichloroethene	2.3	0.19

TABLE 29 (cont.)

Compound	% RSD	MDL ( $\mu\text{g}/\text{kg}$ )
Trichlorofluoromethane	2.7	0.31
1,2,3-Trichloropropane	1.5	0.11
Vinyl chloride	4.8	0.45
m-Xylene/p-Xylene	3.6	0.37
o-Xylene	3.6	0.33

- <sup>a</sup> Most compounds spiked at 2 ng/g (2  $\mu\text{g}/\text{kg}$ )  
<sup>b</sup> Incorrect ionization due to methanol  
<sup>c</sup> Compound detected in unfortified sand at >1 ng

TABLE 30

RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5021)

Compound	Recovery per Replicate (ng)			Mean (ng)	RSD	Recovery (%)
	Sample 1	Sample 2	Sample 3			
Benzene	37.6	35.2	38.4	37.1	3.7	185 <sup>a</sup>
Bromochloromethane	20.5	19.4	20.0	20.0	2.3	100
Bromodichloromethane	21.1	20.3	22.8	21.4	4.9	107
Bromoform	23.8	23.9	25.1	24.3	2.4	121
Bromomethane	21.4	19.5	19.7	20.2	4.2	101
Carbon tetrachloride	27.5	26.6	28.6	27.6	3.0	138
Chlorobenzene	25.6	25.4	26.4	25.8	1.7	129
Chloroethane	25.0	24.4	25.3	24.9	1.5	125
Chloroform	21.9	20.9	21.7	21.5	2.0	108
Chloromethane	21.0	19.9	21.3	20.7	2.9	104 <sup>a</sup>
1,2-Dibromo-3-chloro- propane	20.8	20.8	21.0	20.9	0.5	104
1,2-Dibromoethane	20.1	19.5	20.6	20.1	2.2	100
Dibromomethane	22.2	21.0	22.8	22.0	3.4	110
1,2-Dichlorobenzene	18.0	17.7	17.1	17.6	2.1	88.0
1,3-Dichlorobenzene	21.2	21.0	20.1	20.8	2.3	104
1,4-Dichlorobenzene	20.1	20.9	19.9	20.3	2.1	102
Dichlorodifluoromethane	25.3	24.1	25.4	24.9	2.4	125
1,1-Dichloroethane	23.0	22.0	22.7	22.6	1.9	113
1,2-Dichloroethane	20.6	19.5	19.8	20.0	2.3	100
1,1-Dichloroethene	24.8	23.8	24.4	24.3	1.7	122
cis-1,2-Dichloroethene	21.6	20.0	21.6	21.1	3.6	105
trans-1,2-Dichloroethene	22.4	21.4	22.2	22.0	2.0	110
1,2-Dichloropropane	22.8	22.2	23.4	22.8	2.1	114
1,1-Dichloropropene	26.3	25.7	28.0	26.7	3.7	133
cis-1,3-Dichloropropene	20.3	19.5	21.1	20.3	3.2	102
Ethylbenzene	24.7	24.5	25.5	24.9	1.7	125
Hexachlorobutadiene	23.0	25.3	25.2	24.5	4.3	123
Methylene chloride	26.0	25.7	26.1	25.9	0.7	130 <sup>a</sup>
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8 <sup>a</sup>
Styrene	24.2	23.3	23.3	23.6	1.8	118
1,1,1,2-Tetrachloroethane	21.4	20.2	21.3	21.0	2.6	105
1,1,2,2-Tetrachloroethane	18.6	17.8	19.0	18.5	2.7	92.3
Tetrachloroethene	25.2	24.8	26.4	25.5	2.7	127
Toluene	28.6	27.9	30.9	29.1	4.4	146 <sup>a</sup>
1,2,4-Trichlorobenzene	15.0	14.4	12.9	14.1	6.3	70.5
1,1,1-Trichloroethane	28.1	27.2	29.9	28.4	4.0	142
1,1,2-Trichloroethane	20.8	19.6	21.7	20.7	4.2	104

TABLE 30 (cont.)

Compound	Recovery per Replicate (ng)			Mean (ng)	RSD	Recovery (%)
	Sample 1	Sample 2	Sample 3			
Trichloroethene	26.3	24.9	26.8	26.0	3.1	130
Trichlorofluoromethane	25.9	24.8	26.5	25.7	2.7	129
1,2,3-Trichloropropane	18.8	18.3	19.3	18.8	2.2	94.0
Vinyl chloride	24.8	23.2	23.9	24.0	2.7	120
m-Xylene/p-Xylene	24.3	23.9	25.3	24.5	2.4	123
o-Xylene	23.1	22.3	23.4	22.9	2.0	115

<sup>a</sup> Compound found in unfortified garden soil matrix at >5 ng.

TABLE 31

METHOD DETECTION LIMITS AND BOILING POINTS  
FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)<sup>a</sup>

Compound	Detection Limit (ng)	Boiling Point (°C)
Chloromethane	58	-24
Bromomethane	26	4
Vinyl chloride	14	-13
Chloroethane	21	13
Methylene chloride	9	40
Acetone	35	56
Carbon disulfide	11	46
1,1-Dichloroethene	14	32
1,1-Dichloroethane	12	57
trans-1,2-Dichloroethene	11	48
Chloroform	11	62
1,2-Dichloroethane	13	83
1,1,1-Trichloroethane	8	74
Carbon tetrachloride	8	77
Bromodichloromethane	11	88
1,1,2,2-Tetrachloroethane**	23	146
1,2-Dichloropropane	12	95
trans-1,3-Dichloropropene	17	112
Trichloroethene	11	87
Dibromochloromethane	21	122
1,1,2-Trichloroethane	26	114
Benzene	26	80
cis-1,3-Dichloropropene	27	112
Bromoform**	26	150
Tetrachloroethene	11	121
Toluene	15	111
Chlorobenzene	15	132
Ethylbenzene**	21	136
Styrene**	46	145
Trichlorofluoromethane	17	24
Iodomethane	9	43
Acrylonitrile	13	78
Dibromomethane	14	97
1,2,3-Trichloropropane**	37	157
total Xylenes**	22	138-144

Footnotes are found on the following page.

TABLE 31 (cont.)

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- \* The method detection limit (MDL) is defined in Chapter One. The detection limits cited above were determined according to 40 CFR, Part 136, Appendix B, using standards spiked onto clean VOST tubes. Since clean VOST tubes were used, the values cited above represent the best that the methodology can achieve. The presence of an emissions matrix will affect the ability of the methodology to perform at its optimum level.
- \*\* Boiling Point greater than 130°C. Not appropriate for quantitative sampling by Method 0030.



TABLE 32

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES  
ASSIGNED FOR QUANTITATION (METHOD 5041)

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Bromochloromethane

Acetone  
Acrylonitrile  
Bromomethane  
Carbon disulfide  
Chloroethane  
Chloroform  
Chloromethane  
1,1-Dichloroethane  
1,2-Dichloroethane  
1,2-Dichloroethane-d<sub>4</sub> (surrogate)  
1,1-Dichloroethene  
Trichloroethene  
trans-1,2-Dichloroethene  
Iodomethane  
Methylene chloride  
Trichlorofluoromethane  
Vinyl chloride

Chlorobenzene-d<sub>5</sub>

4-Bromofluorobenzene (surrogate)  
Chlorobenzene  
Ethylbenzene  
Styrene  
1,1,2,2-Tetrachloroethane  
Tetrachloroethene  
Toluene  
Toluene-d<sub>8</sub> (surrogate)  
1,2,3-Trichloropropane  
Xylenes

1,4-Difluorobenzene

Benzene  
Bromodichloromethane  
Bromoform  
Carbon tetrachloride  
Chlorodibromomethane  
Dibromomethane  
1,2-Dichloropropane  
cis-1,3-Dichloropropene  
trans-1,3-Dichloropropene  
1,1,1-Trichloroethane  
1,1,2-Trichloroethane

TABLE 33

## METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

Compound	Boiling Point (°C)	Condensation Point at 20°C (%)	Estimated Detection Limit <sup>a</sup> (ppm)
Dichlorodifluoromethane	-30	Gas	0.20
Vinyl chloride	-19	Gas	0.11
1,3-Butadiene	-4	Gas	0.90
1,2-Dichloro-1,1,2,2-tetrafluoroethane	4	Gas	0.14
Methyl bromide	4	Gas	0.14
Trichlorofluoromethane	24	88	0.18
1,1-Dichloroethene	31	22	0.07
Methylene chloride	40	44	0.05
1,1,2-Trichloro-trifluoroethane	48	37	0.13
Chloroform	61	21	0.04
1,1,1-Trichloroethane	75	13	0.03
Carbon tetrachloride	77	11	0.03
Benzene	80	10	0.16
Trichloroethene	87	8	0.04
1,2-Dichloropropane	96	5	0.05
Toluene	111	3	0.08
Tetrachloroethene	121	2	0.03

<sup>a</sup> Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack detection limits.

FIGURE 1  
GAS CHROMATOGRAM OF VOLATILE ORGANICS

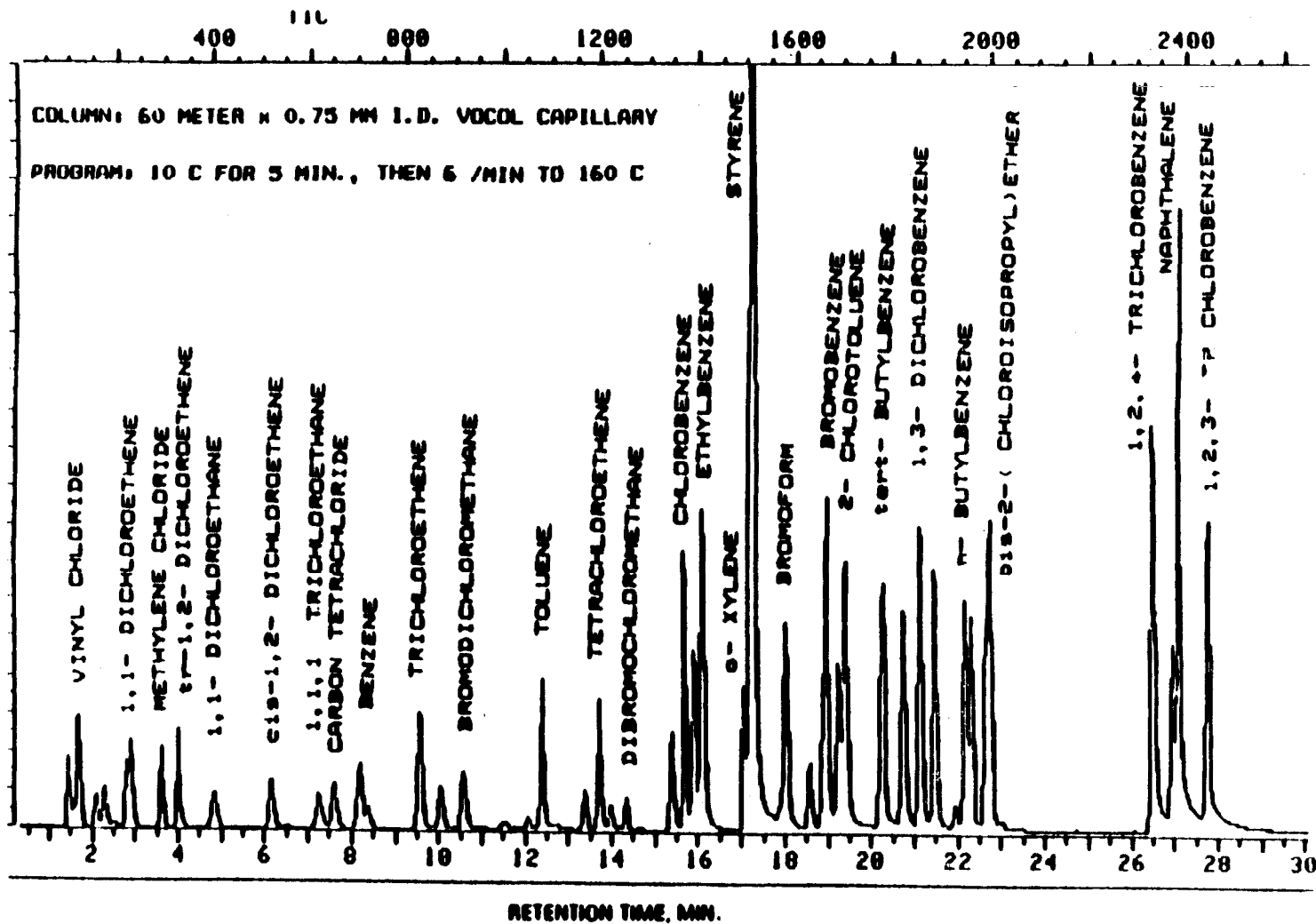


FIGURE 2  
GAS CHROMATOGRAM OF VOLATILE ORGANICS

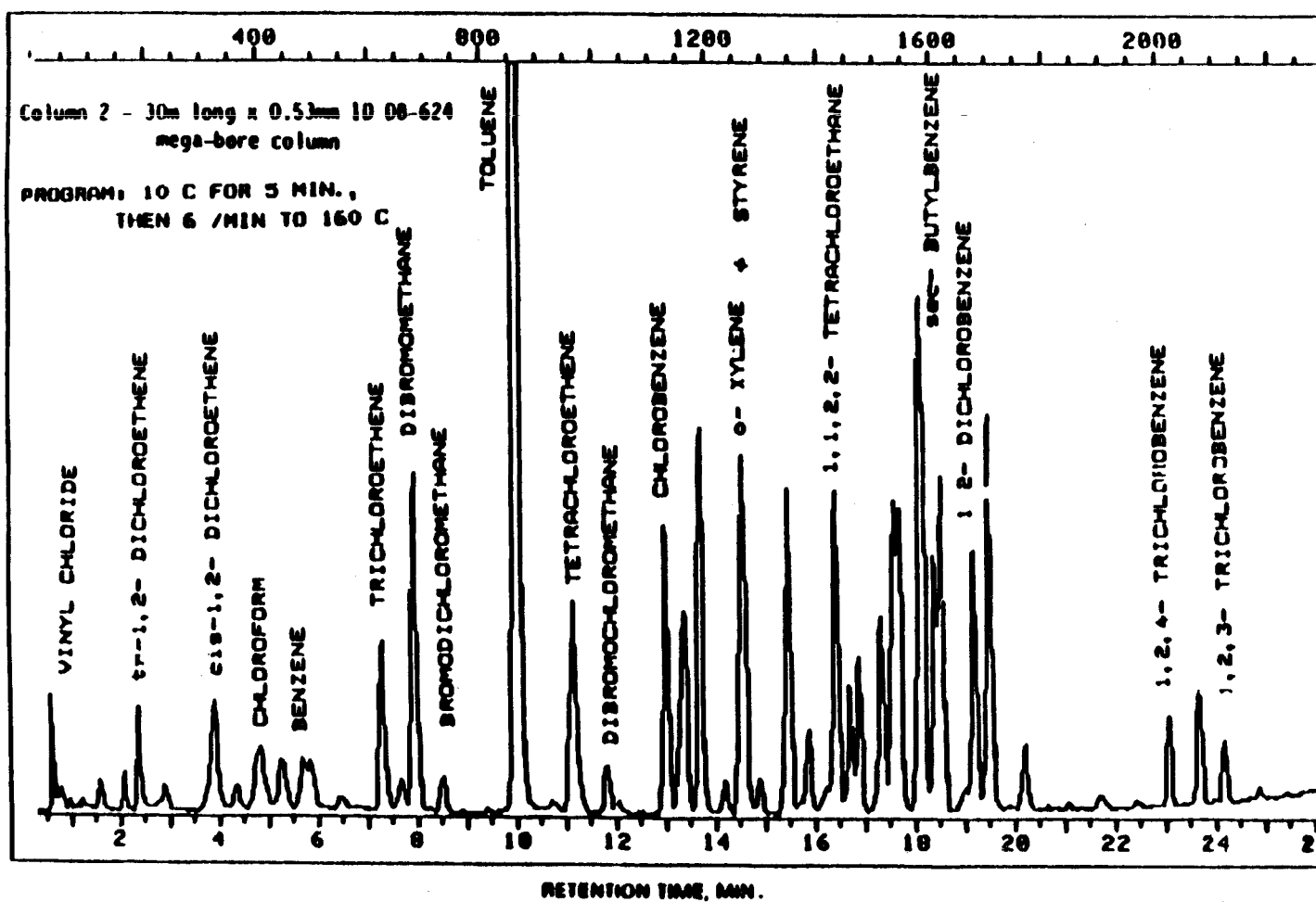
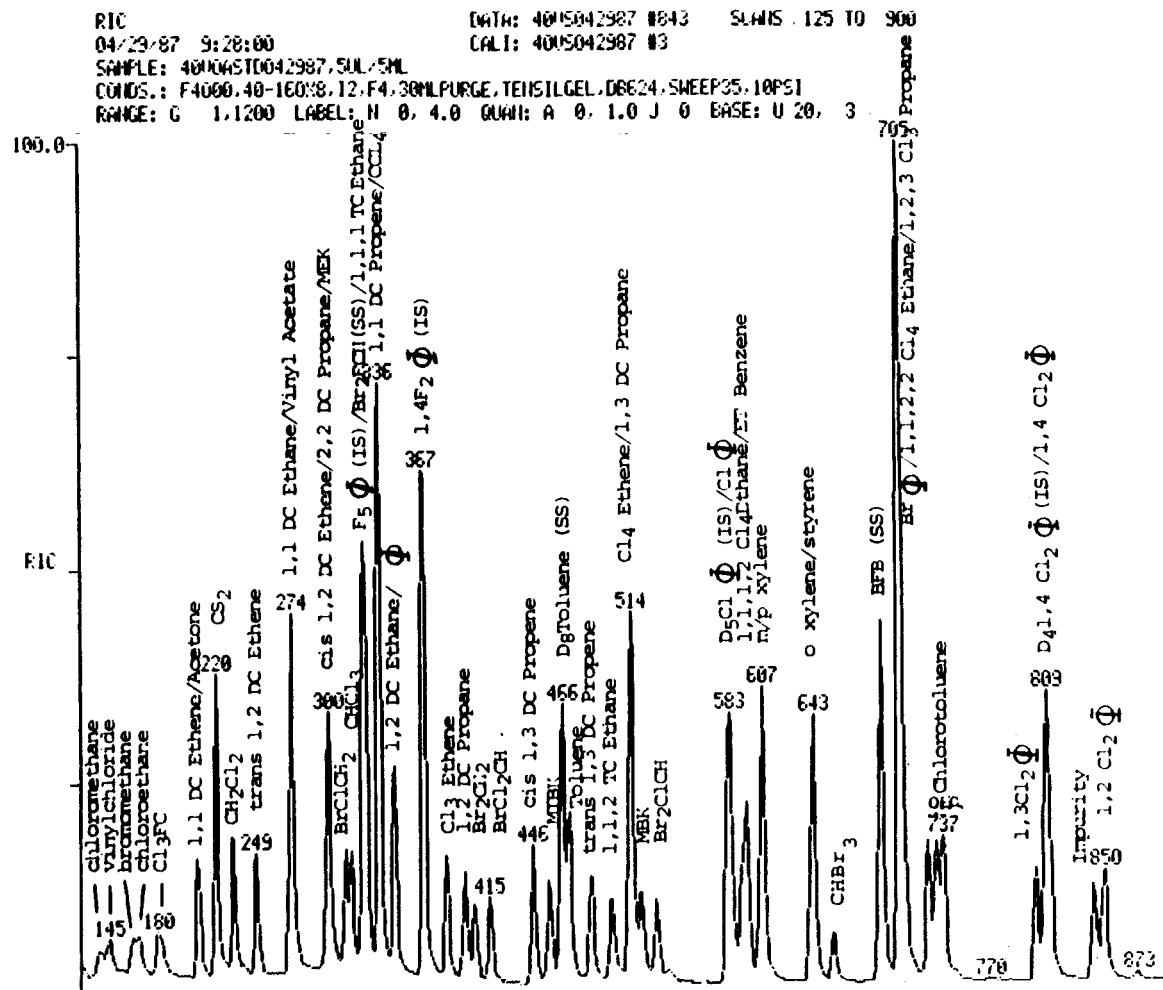
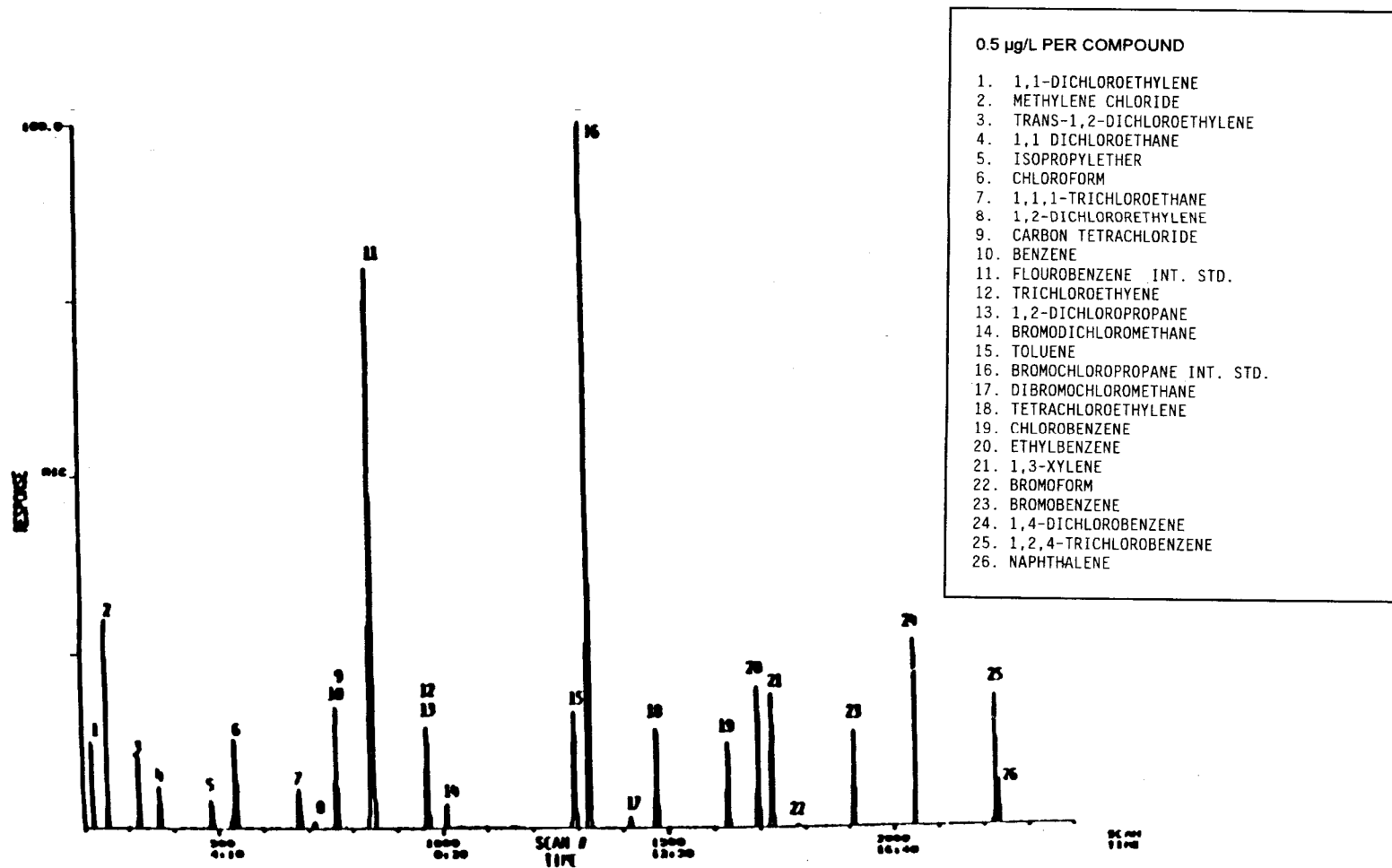


FIGURE 3  
GAS CHROMATOGRAM OF VOLATILE ORGANICS

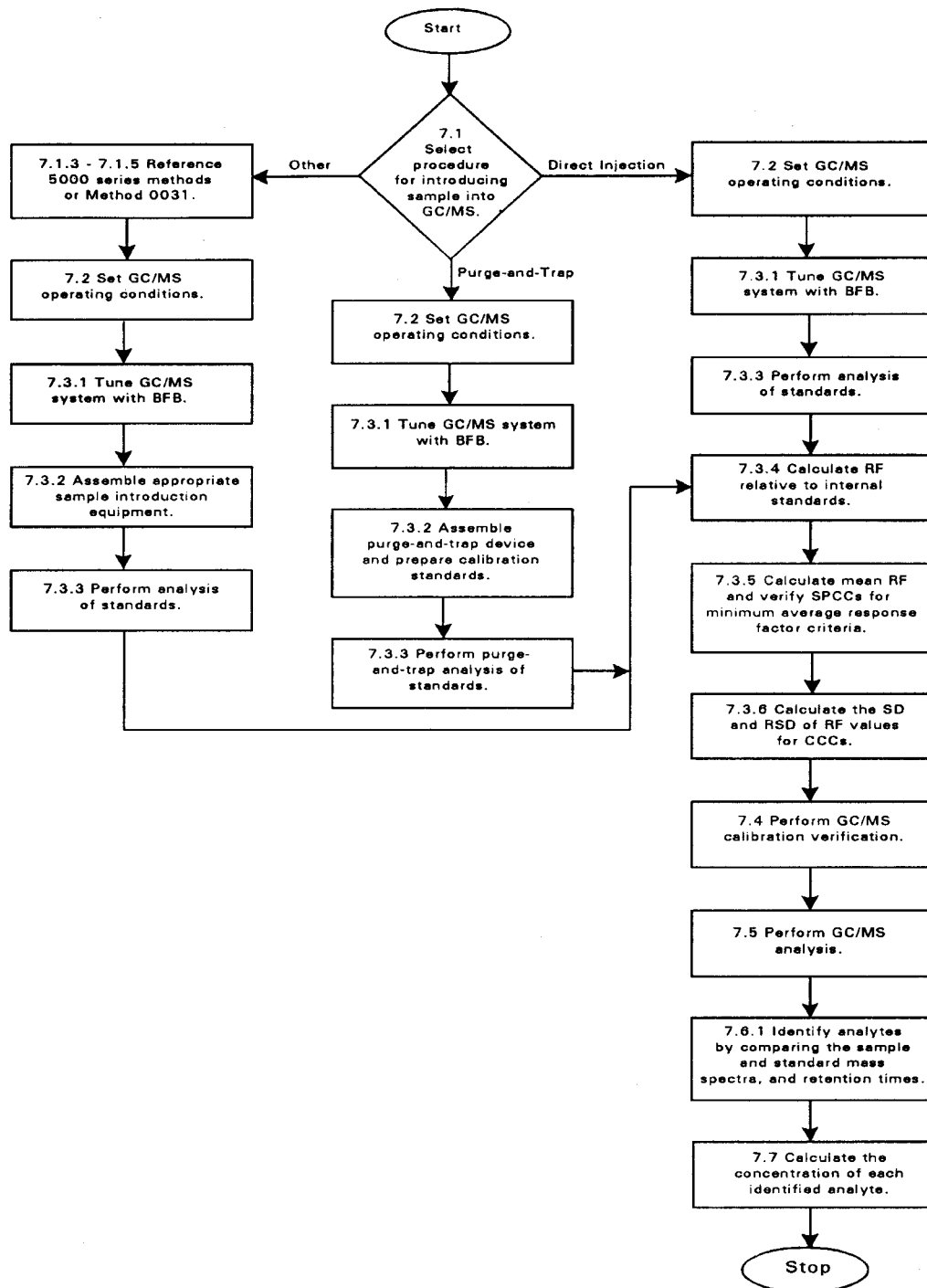


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FIGURE 4  
GAS CHROMATOGRAM OF TEST MIXTURE



METHOD 8260B  
 VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY  
 (GC/MS)



METHOD 8081A

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Method 8081 is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). When compared to the packed columns, these columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The compounds listed below may be determined by either a single- or dual-column analysis system.

Compound	CAS Registry No.
Aldrin	309-00-2
$\alpha$ -BHC	319-84-6
$\beta$ -BHC	319-85-7
$\gamma$ -BHC (Lindane)	58-89-9
$\delta$ -BHC	319-86-8
Chlorobenzilate	510-15-6
$\alpha$ -Chlordane	5103-71-9
$\gamma$ -Chlordane	5103-74-2
Chlordane - not otherwise specified	57-74-9
DBCP	96-12-8
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Diallate	2303-16-4
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
Isodrin	465-73-6
Methoxychlor	72-43-5
Toxaphene	8001-35-2

1.2 This revision of Method 8081 no longer includes the PCBs as Aroclors in the list of target analytes. The analysis of PCBs should be undertaken using Method 8082, which includes specific cleanup and quantitation procedures designed for PCB analysis. This change was made to obtain PCB data of better quality and to eliminate the complications inherent in a combined organochlorine pesticide and PCB method. Therefore, if the presence of PCBs is expected, use Method 8082 for



PCB analyses, and this method (8081) for the organochlorine pesticides. If there is no information of the likely presence of PCBs, either employ a PCB-specific screening procedure such as an immunoassay (e.g., Method 4020), or split the sample extract *prior to* any cleanup steps, and process part of the extract for organochlorine pesticide analysis and the other portion for PCB analysis using Method 8082.

1.3 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.).

1.4 Although performance data are presented for many of the target analytes, it is unlikely that all of them could be determined in a single analysis. The chemical and chromatographic behaviors of many of these chemicals can result in co-elution of some target analytes. Several cleanup/fractionation schemes are provided in this method and in Method 3600.

1.5 Several multi-component mixtures (i.e., Chlordane and Toxaphene) are listed as target analytes. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" multi-component mixtures that may have significant differences in peak patterns than those of standards.

1.6 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS Method 8270 is also recommended as a confirmation technique, if sensitivity permits (Sec. 8.0).

1.7 This method includes a dual-column option. The option allows a hardware configuration of two analytical columns joined to a single injection port. The option allows one injection to be used for dual-column analysis. Analysts are cautioned that the dual-column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period, or when contaminated samples are analyzed.

1.8 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.9 Extracts suitable for analysis by this method may also be analyzed for organophosphorus pesticides (Method 8141). Some extracts may also be suitable for triazine herbicide analysis, if low recoveries (normally samples taken for triazine analysis must be preserved) are not a problem.

1.10 The following compounds may also be determined using this method:

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Compound	CAS Registry No.
Alachlor	15972-60-8
Captafol	2425-06-1
Chloroneb	2675-77-6

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Compound	CAS Registry No.
Chloropropylate	99516-95-7
Chlorothalonil	1897-45-6
DCPA	1861-32-1
Dichlone	117-80-6
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Mirex	2385-85-5
Nitrofen	1836-75-5
PCNB	82-68-8
Permethrin	51877-74-8
Perthane	72-56-0
Propachlor	1918-16-7
Strobane	8001-50-1
<i>trans</i> -Nonachlor	39765-80-5
Trifluralin	1582-09-8

1.11 Kepone extracted from samples or standards exposed to water or methanol may produce peaks with broad tails that elute later than the standard by up to 1 minute. This shift is presumably the result of the formation of a hemi-acetal from the ketone functionality. As a result, Method 8081 is not recommended for determining Kepone. Method 8270 may be more appropriate for the analysis of Kepone.

## 2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

2.2 Liquid samples are extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), or other appropriate technique.

2.3 Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3550 (ultrasonic extraction), or other appropriate technique.

2.4 A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Suggested cleanups include alumina (Method 3610), Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660).

2.5 After cleanup, the extract is analyzed by injecting a 1- $\mu$ L sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).

### 3.0 INTERFERENCES

3.1 Refer to Methods 3500 (Sec. 3.0, in particular), 3600, and 8000, for a discussion of interferences.

3.2 Sources of interference in this method can be grouped into three broad categories.

3.2.1 Contaminated solvents, reagents, or sample processing hardware.

3.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

3.2.3 Compounds extracted from the sample matrix to which the detector will respond.

3.2.4 Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

3.3.1 These materials may be removed prior to analysis using Method 3640 (Gel Permeation Cleanup) or Method 3630 (Silica Gel Cleanup).

3.3.2 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

3.3.3 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

3.3.4 Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.4 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry it in an oven at 130°C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

3.5 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

3.6 Waxes, lipids, and other high molecular weight materials can be removed by Method 3640 (gel-permeation cleanup).

3.7 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorus pesticides are eliminated by Method 3640 (gel permeation cleanup - pesticide option). Co-eluting chlorophenols may be eliminated by using Method 3630 (silica gel), Method 3620 (florisil), or Method 3610 (alumina). Polychlorinated biphenyls (PCBs) also may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as Chlordane, Toxaphene, and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult Methods 3620 and 3630 for techniques that may be used to separate the pesticides from the PCBs.

3.8 Co-elution among the many target analytes in this method can cause interference problems. The following target analytes may coelute on the GC columns listed, when using the single-column analysis scheme:

DB 608	Trifluralin/Diallate isomers PCNP/Dichlone/Isodrin
DB 1701	Captafol/Mirex Methoxychlor/Endosulfan sulfate

3.9 The following compounds may coelute using the dual-column analysis scheme. In general, the DB-5 column resolves fewer compounds than the DB-1701.

DB-5	Permethrin/Heptachlor epoxide Endosulfan I/ $\alpha$ -Chlordane Perthane/Endrin Endosulfan II/Chloropropylate/Chlorobenzilate 4,4'-DDT/Endosulfan sulfate Methoxychlor/Dicofol
DB-1701	Chlorothalonil/ $\beta$ -BHC $\delta$ -BHC/DCPA/Permethrin $\alpha$ -Chlordane/ <i>trans</i> -Nonachlor

Nitrofen, Dichlone, Carbophenothion, Dichloran exhibit extensive peak tailing on both columns. Simazine and Atrazine give poor responses on the ECD detector. Triazine compounds should be analyzed using Method 8141 (NPD option).

#### 4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: an analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system.

#### 4.2 GC columns

This method describes procedures for both single-column and dual-column analyses. The single-column approach involves one analysis to determine that a compound is present, followed by a second analysis to confirm the identity of the compound (Sec. 8.4 describes how GC/MS

confirmation techniques may be employed). The single-column approach may employ either narrow-bore ( $\leq 0.32$  mm ID) columns or wide-bore (0.53 mm ID) columns. The dual-column approach involves a single injection that is split between two columns that are mounted in a single gas chromatograph. The dual-column approach employs only wide-bore (0.53 mm ID) columns.

The columns listed in this section were the columns used to develop the method performance data. The mention of these columns in this method is not intended to exclude the use of other columns that may be developed. Laboratories may use other capillary columns provided that they document method performance data (e.g., chromatographic resolution, analyte breakdown, and MDLs) that equals or exceeds the performance described in this method, or as appropriate for the intended application.

4.2.1 Narrow-bore columns for single-column analysis (use both columns to confirm compound identifications unless another confirmation technique such as GC/MS is employed).

4.2.1.1 30 m x 0.25 or 0.32 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1  $\mu$ m film thickness.

4.2.1.2 30 m x 0.25 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent), 2.5  $\mu$ m coating thickness, 1  $\mu$ m film thickness.

4.2.1.3 Narrow bore columns should be installed in split/splitless (Grob-type) injectors.

4.2.2 Wide-bore columns for single-column analysis (use two of the three columns listed to confirm compound identifications unless another confirmation technique such as GC/MS is employed).

4.2.2.1 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5  $\mu$ m or 0.83  $\mu$ m film thickness.

4.2.2.2 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0  $\mu$ m film thickness.

4.2.2.3 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 95 percent dimethyl - 5 percent diphenyl polysiloxane (DB-5, SPB-5, RTx-5, or equivalent), 1.5  $\mu$ m film thickness.

4.2.2.4 Wide-bore columns should be installed in 1/4 inch injectors, with deactivated liners designed specifically for use with these columns.

4.2.3 Wide-bore columns for dual-column analysis (choose one of the two pairs of columns listed below).

4.2.3.1 Column pair 1

30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5  $\mu$ m film thickness.

30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0 µm film thickness.

Column pair 1 is mounted in a press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733) or a Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.

#### 4.2.3.2 Column pair 2

30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 0.83 µm film thickness.

30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0 µm film thickness.

Column pair 2 is mounted in an 8 in. deactivated glass injection tee (Supelco, Catalog No. 2-3665M), or equivalent.

4.3 Column rinsing kit: Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000), or equivalent.

4.4 Volumetric flasks, 10-mL and 25-mL, for preparation of standards.

## 5.0 REAGENTS

5.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

**NOTE:** Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, it is recommended that aliquots of that lot be stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests (Sec. 8.0) indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC (Sec. 8.0) indicates a problem.

5.2 Solvents used in the extraction and cleanup procedures (appropriate 3500 and 3600 series methods) include n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane) and must be exchanged to n-hexane or isooctane prior to analysis.

Therefore, n-hexane and isooctane will be required in this procedure. Acetone or toluene may be required for the preparation of some standard solutions (see Sec. 5.4.2). All solvents should be pesticide quality or equivalent, and each lot of solvent should be determined to be phthalate free.

5.3 Organic-free reagent water - All references to water in this method refer to organic-free reagent water as defined in Chapter One.

5.4 Stock standard solutions (1000 mg/L) - May be prepared from pure standard materials or can be purchased as certified solutions.

5.4.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially prepared stock standard solutions can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.4.2  $\beta$ -BHC, Dieldrin, and some other standards may not be adequately soluble in isooctane. A small amount of acetone or toluene should be used to dissolve these compounds during the preparation of the stock standard solutions.

5.5 Composite stock standard - May be prepared from individual stock solutions.

5.5.1 For composite stock standards containing less than 25 components, take exactly 1 mL of each individual stock solution at a concentration of 1000 mg/L, add solvent, and mix the solutions in a 25-mL volumetric flask. For example, for a composite containing 20 individual standards, the resulting concentration of each component in the mixture, after the volume is adjusted to 25 mL, will be 1 mg/25 mL. This composite solution can be further diluted to obtain the desired concentrations.

5.5.2 For composite stock standards containing more than 25 components, use volumetric flasks of the appropriate volume (e.g., 50 mL, 100 mL), and follow the procedure described above.

5.6 Calibration standards should be prepared at a minimum of five different concentrations by dilution of the composite stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector.

5.6.1 Although all single component analytes can be resolved on a new 35 percent phenyl methyl silicone column (e.g., DB-608), two calibration mixtures should be prepared for the single component analytes of this method. This procedure is established to minimize potential resolution and quantitation problems on confirmation columns or on older 35 percent phenyl methyl silicone (e.g. DB-608) columns and to allow determination of Endrin and DDT breakdown for method QC (Sec. 8.0).

5.6.2 Separate calibration standards are required for each multi-component target analyte (e.g., Toxaphene and Chlordane). Analysts should evaluate the specific Toxaphene standard carefully. Some Toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results.

5.7 Internal standard (optional)

5.7.1 Pentachloronitrobenzene is suggested as an internal standard for the single-column analysis, when it is not considered to be a target analyte. 1-bromo-2-nitrobenzene may also be used. Prepare a solution of 5000 mg/L (5000 ng/ $\mu$ L) of pentachloronitrobenzene or 1-bromo-2-nitrobenzene. Spike 10  $\mu$ L of this solution into each 1 mL sample extract.

5.7.2 1-bromo-2-nitrobenzene is suggested as an internal standard for the dual-column analysis. Prepare a solution of 5000 mg/L (5000 ng/μL) of 1-bromo-2-nitrobenzene. Spike 10 μL of this solution into each 1 mL of sample extract.

## 5.8 Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. The following compounds are recommended as possible surrogates.

5.8.1 Decachlorobiphenyl and tetrachloro-m-xylene have been found to be a useful pair of surrogates for both the single-column and dual-column configurations. Method 3500, Sec. 5.0, describes the procedures for preparing these surrogates.

5.8.2 4-Chloro-3-nitrobenzotrifluoride may also be useful as a surrogate if the chromatographic conditions of the dual-column configuration cannot be adjusted to preclude co-elution of a target analyte with either of the surrogates in Sec. 5.8.1. However, this compound elutes early in the chromatographic run and may be subject to other interference problems. A recommended concentration for this surrogate is 500 ng/μL. Use a spiking volume of 100 μL for a 1-L aqueous sample.

5.8.3 Store surrogate spiking solutions at 4°C in PTFE-sealed containers in the dark.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Four, Organic Analytes, Sec. 4.0, for sample collection and preservation instructions.

6.2 Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

## 7.0 PROCEDURE

### 7.1 Sample extraction

Refer to Chapter Two and Method 3500 for guidance in choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520), or other appropriate technique. Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using one of the Soxhlet extraction (Method 3540 or 3541), pressurized fluid extraction (Method 3545), ultrasonic extraction (Method 3550), or other appropriate technique.

**NOTE:** Hexane-acetone (1:1) may be more effective as an extraction solvent for organochlorine pesticides in some environmental and waste matrices than is methylene chloride-acetone (1:1). Relative to the methylene chloride-acetone mixture, use of hexane-acetone generally reduces the amount of interferences that are extracted and improves signal-to-noise.



Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. Each sample type must be spiked with the compounds of interest to determine the percent recovery and the limit of detection for that sample (see Chapter One). See Method 8000 for guidance on demonstration of initial method proficiency as well as guidance on matrix spikes for routine sample analysis.

## 7.2 Extract cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. General guidance for sample extract cleanup is provided in this section and in Method 3600.

7.2.1 If a sample is of biological origin, or contains high molecular weight materials, the use of Method 3640 (GPC cleanup - pesticide option) is recommended. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel, or florisil) may also be required following the GPC cleanup.

7.2.2 Method 3610 (alumina) may be used to remove phthalate esters.

7.2.3 Method 3620 (florisil) may be used to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

7.2.4 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

7.2.5 Elemental sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in Method 3660.

## 7.3 GC conditions

This method allows the analyst to choose between a single-column or a dual-column configuration in the injector port. Either wide- or narrow-bore columns may be used. Identifications based on retention times from a single-column must be confirmed on a second column or with an alternative qualitative technique.

### 7.3.1 Single-column analysis

This capillary GC/ECD method allows the analyst the option of using 0.25-0.32 mm ID capillary columns (narrow-bore) or 0.53 mm ID capillary columns (wide-bore). Performance data are provided for both options. Figures 1-6 provide example chromatograms.

7.3.1.1 The use of narrow-bore ( $\leq 0.32$  mm ID) columns is recommended when the analyst requires greater chromatographic resolution. Use of narrow-bore columns is suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53 mm ID) are suitable for more complex environmental and waste matrices.

7.3.1.2 Table 1 lists average retention times and method detection limits (MDLs) for the target analytes in water and soil matrices, using wide-bore capillary

columns. Table 2 lists average retention times and method detection limits (MDLs) for the target analytes in water and soil matrices, using narrow-bore capillary columns. The MDLs for the components of a specific sample are dependent upon the nature of interferences in the sample matrix and may differ from those listed in Tables 1 and 2. Table 3 lists the Estimated Quantitation Limits (EQLs) for other matrices.

7.3.1.3 Table 4 lists the GC operating conditions for the single-column method of analysis.

### 7.3.2 Dual-column analysis

The dual-column/dual-detector approach involves the use of two 30 m x 0.53 mm ID fused-silica open-tubular columns of different polarities, thus, different selectivities towards the target analytes. The columns are connected to an injection tee and separate electron capture detectors.

7.3.2.1 Retention times for the organochlorine analytes on dual-columns are in Table 6. The GC operating conditions for the compounds in Table 6 are given in Table 7.

7.3.2.2 Multi-component mixtures of Toxaphene and Strobane were analyzed separately (Figures 5 and 6) using the GC operating conditions found in Table 7.

7.3.2.3 Figure 6 is a sample chromatogram for a mixture of organochlorine pesticides. The retention times of the individual components detected in these mixtures are given in Tables 6 and 7.

7.3.2.4 Operating conditions for a more heavily loaded DB-5/DB-1701 pair are given in Table 8. This column pair was used for the detection of multi-component organochlorine compounds.

7.3.2.5 Operating conditions for a DB-5/DB-1701 column pair with thinner films, a different type of splitter, and a slower temperature programming rate are provided in Table 7. These conditions gave better peak shapes for Nitrofen and Dicofol. Table 6 lists the retention times for the compounds detected on this column pair.

## 7.4 Calibration

7.4.1 Prepare calibration standards using the procedures in Sec. 5.0. Refer to Method 8000 (Sec. 7.0) for proper calibration techniques for both initial calibration and calibration verification. The procedure for either internal or external calibration may be used. In most cases, external standard calibration is used with Method 8081 because of the sensitivity of the electron capture detector and the probability of the internal standard being affected by interferences. Because several of the pesticides may co-elute on any single-column, analysts should use two calibration mixtures (see Sec. 3.8). The specific mixture should be selected to minimize the problem of peak overlap.

**NOTE:** Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration.

7.4.1.1 Unless otherwise necessary for a specific project, the analysis of the multi-component analytes employs a single-point calibration. A single calibration standard near the mid-point of the expected calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column.

7.4.1.2 For calibration verification (each 12-hour shift) all target analytes required in the project plan must be injected.

7.4.2 Establish the GC operating conditions appropriate for the configuration (single-column or dual column, Sec. 7.3) using Tables 4, 5, 7, or 8 as guidance. Optimize the instrumental conditions for resolution of the target analytes and sensitivity. An initial oven temperature  $\leq 140$  -  $150^{\circ}\text{C}$  is required to resolve the four BHC isomers. A final temperature of  $240$  -  $270^{\circ}\text{C}$  is required to elute decachlorobiphenyl. Use of injector pressure programming will improve the chromatography of late eluting peaks.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

7.4.3 A  $2\ \mu\text{L}$  injection volume of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.

7.4.4 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column should be primed (or deactivated) by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

CAUTION: Several analytes, including Aldrin, may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

#### 7.4.5 Calibration factors

When external standard calibration is employed, calculate the calibration factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, using the formulae below. If internal standard calibration is employed, refer to Method 8000 for the calculation of response factors.

7.4.5.1 Calculate the calibration factor for each analyte at each concentration as:

$$\text{CF} = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

7.4.5.2 Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{\text{CF}} = \frac{\sum_{i=1}^n \text{CF}_i}{n}$$

where n is the number of standards analyzed.

7.4.5.3 Calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$\text{SD} = \sqrt{\frac{\sum_{i=1}^n (\text{CF}_i - \overline{\text{CF}})^2}{n-1}} \quad \text{RSD} = \frac{\text{SD}}{\overline{\text{CF}}} \times 100$$

If the RSD for each analyte is  $\leq 20\%$ , then the response of the instrument is considered linear and the mean calibration factor can be used to quantitate sample results. If the RSD is greater than 20%, then linearity through the origin cannot be assumed. The analyst must use a calibration curve or a non-linear calibration model (e.g., a polynomial equation) for quantitation. See Method 8000 for information on non-linear calibrations.

#### 7.4.6 Retention time windows

Absolute retention times are used for compound identification. Retention time windows are crucial to the identification of target compounds, and should be established by one of the approaches described in Method 8000.

7.4.6.1 Before establishing the retention time windows, make sure the gas chromatographic system is operating within optimum conditions.

7.4.6.2 The widths of the retention time windows are defined as described in Method 8000. However, the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

### 7.5 Gas chromatographic analysis of sample extracts

7.5.1 The same GC operating conditions used for the initial calibration must be employed for samples analyses.

7.5.2 Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. Analysts should alternate the use of high and low concentration mixtures of single-component analytes and multi-component analytes for calibration verification. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is *recommended* to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of the analysis sequence. See Sec. 8.4.4 for additional guidance on the frequency of the standard injections.

7.5.2.1 The calibration factor for each analyte should not exceed a  $\pm 15$  percent difference from the mean calibration factor calculated for the initial calibration. If a non-

linear calibration model or a linear model not through the origin has been employed for the initial calibration, consult Sec. 7 of Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{CF - \overline{CF}_v}{\overline{CF}} \times 100$$

7.5.2.2 If this criterion is exceeded for any analyte, use the approach described in Sec. 7 of Method 8000 to calculate the average percent difference across all analytes. If the average of the responses for all analytes is within  $\pm 15\%$ , then the calibration has been verified. However, the conditions in Sec. 7 of Method 8000 also apply, e.g., the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the  $\pm 15\%$  limit.

7.5.2.3 If the calibration does not meet the  $\pm 15\%$  limit (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within  $\pm 15\%$ , then a new initial calibration must be prepared. The effects of a failing calibration verification standard on sample results are discussed in Sec. 7.5.7.

7.5.3 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in Sec. 7.4.6. As described in Method 8000, the center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

7.5.4 Inject a 2- $\mu\text{L}$  aliquot of the concentrated sample extract. Record the volume injected to the nearest 0.05  $\mu\text{L}$  and the resulting peak size in area units.

7.5.5 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS (see Sec. 7.7).

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Sec. 7 of Method 8000 for a discussion of such a comparison. Unless otherwise specified in an approved project plan, the higher result should be reported, as this is a conservative approach relative to protection of the environment. If the relative percent difference of the results exceeds 40%, consult Method 8000 for steps that may be taken to address the discrepancy.

7.5.6 When using the external calibration procedure (Method 8000), determine the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes, as follows. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined.

#### 7.5.6.1 For aqueous samples

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(\overline{\text{CF}})(V_i)(V_s)}$$

where:

$A_x$  = Area (or height) of the peak for the analyte in the sample.

$V_t$  = Total volume of the concentrated extract ( $\mu\text{L}$ ).

$D$  = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made,  $D = 1$ . The dilution factor is always dimensionless.

$\overline{\text{CF}}$  = Mean calibration factor from the initial calibration (area/ng).

$V_i$  = Volume of the extract injected ( $\mu\text{L}$ ). The injection volume for samples and calibration standards must be the same. For purge-and-trap analysis,  $V_i$  is not applicable and therefore is set at 1.

$V_s$  = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to  $\mu\text{g/L}$ .

#### 7.5.6.2 For non-aqueous samples

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_x)(V_t)(D)}{(\overline{\text{CF}})(V_i)(W_s)}$$

where  $A_x$ ,  $V_t$ ,  $D$ ,  $\overline{\text{CF}}$ , and  $V_i$  are the same as for aqueous samples, and

$W_s$  = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to  $\mu\text{g/kg}$ .

7.5.6.3 See Method 8000 for the equation used for internal standard quantitation.

7.5.6.4 If the responses exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.5.6.5 If partially overlapping or coeluting peaks are found, change GC columns or try GC/MS quantitation (see Sec. 8.0 and Method 8270).

7.5.7 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples.

Although analysis of a single mid-concentration standard (standard mixture or multi-component analyte) will satisfy the minimum requirements, analysts are urged to use different calibration verification standards during organochlorine pesticide analyses. Also, multi-level standards (mixtures or multi-component analytes) are highly recommended to ensure that the detector response remains stable for all the analytes over the calibration range.

The results from these bracketing standards must meet the calibration verification criteria in Sec. 7.5.2. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be re-injected if the QC limits are violated for the standard analysis.

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present (i.e., a false negative result).

7.5.8 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.5.9 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

#### 7.5.10 Validation of GC system qualitative performance

7.5.10.1 Use the calibration standards analyzed during the sequence to evaluate retention time stability. The retention time windows are established using the absolute retention time of each analyte as described in Method 8000.

7.5.10.2 Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

7.5.11 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using either internal or external calibration procedures.

7.5.12 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

7.6 Quantitation of multi-component analytes - Multi-component analytes present problems in measurement. Suggestions are offered in the following sections for handling Toxaphene, Strobane, Chlordane, BHC, and DDT.

7.6.1 Toxaphene and Strobane - Toxaphene is manufactured by the chlorination of camphenes, whereas Strobane results from the chlorination of a mixture of camphenes and pinenes. Quantitation of Toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate Toxaphene from GC/ECD results:

7.6.1.1. Adjust the sample size so that the major Toxaphene peaks are 10-70% of full-scale deflection (FSD).

7.6.1.2 Inject a Toxaphene standard that is estimated to be within  $\pm 10$  ng of the sample amount.

7.6.1.3 Quantitate Toxaphene using the total area of the Toxaphene pattern or using 4 to 6 major peaks.

7.6.1.3.1 While Toxaphene contains a large number of compounds that will produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. This unresolved complex mixture results in the "hump" in the chromatogram that is characteristic of this mixture. Although the resolved peaks are important for the identification of the mixture, the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

7.6.1.3.2 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

7.6.1.3.3 Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. A collaborative study of a series of Toxaphene residues evaluated several approaches to quantitation of this compound, including the use of the total area of the peaks in the Toxaphene chromatogram and the use of a subset of 4 to 6 peaks. That study indicated that the use of 4 to 6 peaks provides



results that agree well with the total peak area approach and may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT. Whichever approach is employed should be documented and available to the data user, if necessary.

7.6.1.3.4 When Toxaphene is determined using the 4 to 6 peaks approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionately larger or smaller in the sample compared to the standard.

7.6.1.3.5 The heights or areas of the 4 to 6 peaks should be summed together and used to determine the Toxaphene concentration. Alternatively, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for the sample.

7.6.2 Chlordane - Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The CAS Registry number for Technical Chlordane is properly given as 12789-03-6. *Trans*-Chlordane (or  $\alpha$ -Chlordane, CAS RN 5103-71-9) and *cis*-Chlordane ( $\gamma$ -Chlordane, CAS RN 5103-74-2), are the two most prevalent major components of Technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch. Moreover, changes may occur when the technical material is used to prepare specific pesticide formulations. The approach used for evaluating and reporting Chlordane results will often depend on the end use of the results and the analyst's skill in interpreting this multi-component pesticide residue. The following sections discuss three specific options: reporting Technical Chlordane (12789-03-6), reporting Chlordane (not otherwise specified, 57-74-9), and reporting the individual Chlordane components that can be identified under their individual CAS numbers.

7.6.2.1 When the GC pattern of the residue resembles that of Technical Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using three to five major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation of the residue. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area.

**NOTE:** Octachloro epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.

To measure the total area of the Chlordane chromatogram, inject an amount of a Technical Chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms. Construct the baseline of Technical Chlordane in the standard chromatogram between the retention times of the first and last eluting toxaphene components. Use this area and the mass of Technical Chlordane in the standard to calculate a calibration factor.

Construct a similar baseline in the sample chromatogram, measure the area, and use the calibration factor to calculate the concentration in the sample.

7.6.2.2 The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the pesticide active ingredient Technical Chlordane. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane (n.o.s.)" under the CAS number 57-74-9.

7.6.2.3 The third option is to quantitate the peaks of  $\alpha$ -Chlordane,  $\gamma$ -Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers.

7.6.2.4 To measure the total area of the Chlordane chromatogram, inject an amount of a Technical Chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms.

7.6.3 Hexachlorocyclohexane - Hexachlorocyclohexane is also known as BHC, from the former name, benzene hexachloride. Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor. It consists of a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes. Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. Quantitate each isomer ( $\alpha$ ,  $\beta$ ,  $\gamma$ , and  $\delta$ ) separately against a standard of the respective pure isomer.

7.6.4 DDT - Technical DDT consists primarily of a mixture of 4,4'-DDT (approximately 75%) and 2,4'-DDT (approximately 25%). As DDT weathers, 4,4'-DDE, 2,4'-DDE, 4,4'-DDD, and 2,4'-DDD are formed. Since the 4,4'-isomers of DDT, DDE, and DDD predominate in the environment, these are the isomers normally regulated by EPA. Therefore, sample extracts should be quantitated against standards of the respective pure isomers of 4,4'-DDT, 4,4'-DDE, and 4,4'-DDD.

7.7 GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

7.7.1 Full-scan GC/MS will normally require a concentration of approximately 10 ng/ $\mu$ L in the final extract for each single-component compound. Ion trap or selected ion monitoring will normally require a concentration of approximately 1 ng/ $\mu$ L.

7.7.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

7.7.3 GC/MS may not be used for confirmation when concentrations are below 1 ng/ $\mu$ L in the extract.

7.7.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis and the extract of the associated method blank.

7.7.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated

that the analyte is stable during acid/base partitioning. However, if the compounds are *not* detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

7.7.6 A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

7.8 Suggested chromatographic system maintenance - When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

7.8.1 Splitter connections - For dual-columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters (up to 30 cm) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

7.8.2 GC injector ports can be of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD, or DDE. When such breakdown is observed, clean and deactivate the injector port, break off at least 30 cm of the column and remount it. Check the injector temperature and lower it to 205°C, if required. Endrin and DDT breakdown are less of a problem when ambient on-column injectors are used.

7.8.3 Metal injector body - Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

7.8.3.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene, catching the rinsate in the beaker.

7.8.3.2 Prepare a solution of a deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

7.8.4 Column rinsing - The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

## 8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation techniques can be found in Method 3500. If an extract cleanup procedure was performed, refer to Method 3600 for the appropriate quality control procedures. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification, and chromatographic analysis of samples.

### 8.3 Initial Demonstration of Proficiency

8.3.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

8.3.2 It is suggested that the quality control (QC) reference sample concentrate (as discussed in Section 8.0 of Methods 8000 and 3500) contain each analyte of interest at 10 mg/L. If this method is to be used for analysis of Chlordane or Toxaphene only, the QC reference sample concentrate should contain the most representative multi-component mixture at a suggested concentration of 50 mg/L in acetone. See Method 8000, Sec. 8.0 for additional information on how to accomplish this demonstration.

8.3.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, a matrix spike, a duplicate, a laboratory control sample (LCS), and the addition of surrogates to each field sample and QC sample.

8.4.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.2 In-house method performance criteria should be developed using the guidance found in Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same

concentrations as the matrix spike. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 Include a calibration standard after each group of 20 samples (it is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. Thus, injections of method blank extracts, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. The response factors for the calibration should be within  $\pm 15\%$  of the initial calibration (see Sec. 7.5.2). When this calibration verification standard falls out of this acceptance window, the laboratory should stop analyses and take corrective action.

8.4.5 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed.

8.4.6 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

8.4.6.1 Calculate percent breakdown as follows:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

$$\% \text{ breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde + ketone)}}{\text{sum of all peak areas (Endrin + aldehyde + ketone)}} \times 100$$

8.4.6.2 The breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound (Sec. 7.8.2).

8.4.7 Whenever silica gel (Method 3630) or Florisil (Method 3620) cleanups are used, the analyst must demonstrate that the fractionation scheme is reproducible. Batch to batch variation in the composition of the silica gel or Florisil or overloading the column may cause a change in the distribution patterns of the organochlorine pesticides. When compounds are found in two fractions, add the concentrations found in the fractions, and correct for any additional dilution.

8.4.8 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries: The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method

8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined in Chapter One. A laboratory should develop its own matrix-specific MDLs using the guidance found in Chapter One.

9.2 The chromatographic separations in this method have been tested in a single laboratory by using clean hexane and liquid and solid waste extracts that were spiked with the test compounds at three concentrations. Single-operator precision, overall precision, and method accuracy were found to be related to the concentration of the compound and the type of matrix.

9.3 This method has been applied in a variety of commercial laboratories for environmental and waste matrices. Performance data were obtained for a limited number of target analytes spiked into sewage sludge and dichloroethene stillbottoms at high concentrations. These data are provided in Tables 9 and 10.

9.4 The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

9.5 Tables 9 and 10 contain precision and recovery data generated for sewage sludge and dichloroethane stillbottoms. Table 11 contains recovery data for a clay soil, taken from Reference 10. The spiking concentration was for the clay soil was 500 µg/kg. The spiking solution was mixed into the soil and then immediately transferred to the extraction device and immersed in the extraction solvent. The spiked sample was then extracted by Method 3541 (Automated Soxhlet). The data represent a single determination. Analysis was by capillary column gas chromatography/electron capture detector following Method 8081 for the organochlorine pesticides.

## 10.0 REFERENCES

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TABLE 1

GAS CHROMATOGRAPHIC RETENTION TIMES  
FOR THE ORGANOCHLORINE PESTICIDES  
USING WIDE-BORE CAPILLARY COLUMNS  
SINGLE-COLUMN METHOD OF ANALYSIS

Compound	Retention Time (min)	
	DB 608 <sup>a</sup>	DB 1701 <sup>a</sup>
Aldrin	11.84	12.50
α-BHC	8.14	9.46
β-BHC	9.86	13.58
δ-BHC	11.20	14.39
γ-BHC (Lindane)	9.52	10.84
α-Chlordane	15.24	16.48
γ-Chlordane	14.63	16.20
4,4'-DDD	18.43	19.56
4,4'-DDE	16.34	16.76
4,4'-DDT	19.48	20.10
Dieldrin	16.41	17.32
Endosulfan I	15.25	15.96
Endosulfan II	18.45	19.72
Endosulfan Sulfate	20.21	22.36
Endrin	17.80	18.06
Endrin aldehyde	19.72	21.18
Heptachlor	10.66	11.56
Heptachlor epoxide	13.97	15.03
Methoxychlor	22.80	22.34
Toxaphene	MR	MR

NA = Data not available.

MR = Multiple response compound.

<sup>a</sup> See Table 4 for GC operating conditions.



TABLE 2

GAS CHROMATOGRAPHIC RETENTION TIMES  
FOR THE ORGANOCHLORINE PESTICIDES  
USING NARROW-BORE CAPILLARY COLUMNS  
SINGLE-COLUMN METHOD OF ANALYSIS

Compound	Retention Time (min)	
	DB 608 <sup>a</sup>	DB 5 <sup>a</sup>
Aldrin	14.51	14.70
$\alpha$ -BHC	11.43	10.94
$\beta$ -BHC	12.59	11.51
$\delta$ -BHC	13.69	12.20
$\gamma$ -BHC (Lindane)	12.46	11.71
$\alpha$ -Chlordane	NA	NA
$\gamma$ -Chlordane	17.34	17.02
4,4'-DDD	21.67	20.11
4,4'-DDE	19.09	18.30
4,4'-DDT	23.13	21.84
Dieldrin	19.67	18.74
Endosulfan I	18.27	17.62
Endosulfan II	22.17	20.11
Endosulfan sulfate	24.45	21.84
Endrin	21.37	19.73
Endrin aldehyde	23.78	20.85
Heptachlor	13.41	13.59
Heptachlor epoxide	16.62	16.05
Methoxychlor	28.65	24.43
Toxaphene	MR	MR

NA = Data not available.

MR = Multiple response compound.

<sup>a</sup> See Table 4 for GC operating conditions.

TABLE 3

FACTORS FOR DETERMINATION OF ESTIMATED QUANTITATION LIMITS<sup>a</sup> (EQLs)  
FOR VARIOUS MATRICES

Matrix	Factor
Ground water	10
Low-concentration soil by sonication with GPC cleanup	670
High-concentration soil and sludges by sonication	10,000
Non-water miscible waste	100,000

<sup>a</sup> Laboratories may estimate the quantitation limits of the target analytes in environmental and waste media by generating MDLs in organic-free reagent water and using the following equation (see Sec. 5.0 of Chapter One for information on generating MDL data):

$$\text{EQL} = [\text{MDL in water}] \text{ times } [\text{factor in this table}]$$

For nonaqueous samples, the factor is on a wet-weight basis. Sample EQLs are highly matrix-dependent. EQLs determined using these factors are provided as guidance and may not always be achievable.

TABLE 4

GC OPERATING CONDITIONS FOR ORGANOCHLORINE COMPOUNDS  
SINGLE-COLUMN ANALYSIS USING NARROW-BORE COLUMNS

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Column 1 - 30 m x 0.25 or 0.32 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1  $\mu\text{m}$  film thickness.

Carrier gas	Helium
Carrier gas pressure	16 psi
Injector temperature	225°C
Detector temperature	300°C
Initial temperature	100°C, hold 2 minutes
Temperature program	100°C to 160°C at 15°C/min, followed by 160°C to 270°C at 5°C/min
Final temperature	270°C

Column 2 - 30 m x 0.25 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent), 25  $\mu\text{m}$  coating thickness, 1  $\mu\text{m}$  film thickness.

Carrier gas	Nitrogen
Carrier gas pressure	20 psi
Injector temperature	225°C
Detector temperature	300°C
Initial temperature	160°C, hold 2 minutes
Temperature program	160°C to 290°C at 5°C/min
Final temperature	290°C, hold 1 min

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TABLE 5

GC OPERATING CONDITIONS FOR ORGANOCHLORINE COMPOUNDS  
SINGLE-COLUMN ANALYSIS USING WIDE-BORE COLUMNS

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Column 1 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5  $\mu\text{m}$  or 0.83  $\mu\text{m}$  film thickness.

Column 2 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0  $\mu\text{m}$  film thickness.

Both Column 1 and Column 2 use the same GC operating conditions.

Carrier gas	Helium
Carrier gas flow rate	5-7 mL/minute
Makeup gas	argon/methane (P-5 or P-10) or nitrogen
Makeup gas flow rate	30 mL/min
Injector temperature	250°C
Detector temperature	290°C
Initial temperature	150°C, hold 0.5 minute
Temperature program	150°C to 270°C at 5°C/min
Final temperature	270°C, hold 10 min

Column 3 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5  $\mu\text{m}$  film thickness.

Carrier gas	Helium
Carrier gas flow rate	6 mL/minute
Makeup gas	argon/methane (P-5 or P-10) or nitrogen
Makeup gas flow rate	30 mL/min
Injector temperature	205°C
Detector temperature	290°C
Initial temperature	140°C, hold 2 min
Temperature program	140°C to 240°C at 10°C/min, hold 5 minutes at 240°C, 240°C to 265°C at 5°C/min
Final temperature	265°C, hold 18 min

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TABLE 6

RETENTION TIMES OF THE ORGANOCHLORINE PESTICIDES<sup>a</sup>  
DUAL-COLUMN METHOD OF ANALYSIS

Compound	DB-5 RT (min)	DB-1701 RT (min)
DBCP	2.14	2.84
Hexachlorocyclopentadiene	4.49	4.88
Etridiazole	6.38	8.42
Chloroneb	7.46	10.60
Hexachlorobenzene	12.79	14.58
Diallate	12.35	15.07
Propachlor	9.96	15.43
Trifluralin	11.87	16.26
$\alpha$ -BHC	12.35	17.42
PCNB	14.47	18.20
$\gamma$ -BHC	14.14	20.00
Heptachlor	18.34	21.16
Aldrin	20.37	22.78
Alachlor	18.58	24.18
Chlorothalonil	15.81	24.42
Alachlor	18.58	24.18
$\beta$ -BHC	13.80	25.04
Isodrin	22.08	25.29
DCPA	21.38	26.11
$\delta$ -BHC	15.49	26.37
Heptachlor epoxide	22.83	27.31
Endosulfan-I	25.00	28.88
$\gamma$ -Chlordane	24.29	29.32
$\alpha$ -Chlordane	25.25	29.82
<i>trans</i> -Nonachlor	25.58	30.01
4,4'-DDE	26.80	30.40
Dieldrin	26.60	31.20
Perthane	28.45	32.18
Endrin	27.86	32.44
Chloropropylate	28.92	34.14
Chlorobenzilate	28.92	34.42
Nitrofen	27.86	34.42
4,4'-DDD	29.32	35.32
Endosulfan II	28.45	35.51
4,4'-DDT	31.62	36.30
Endrin aldehyde	29.63	38.08
Mirex	37.15	38.79
Endosulfan sulfate	31.62	40.05
Methoxychlor	35.33	40.31
Captafol	32.65	41.42

(continued)

TABLE 6 (continued)

RETENTION TIMES OF THE ORGANOCHLORINE PESTICIDES<sup>a</sup>  
DUAL-COLUMN METHOD OF ANALYSIS

Compound	DB-5 RT (min)	DB-1701 RT (min)
Endrin ketone	33.79	42.26
Permethrin	41.50	45.81
Kepone	31.10	<sup>b</sup>
Dicofol	35.33	<sup>b</sup>
Dichlone	15.17	<sup>b</sup>
$\alpha,\alpha'$ -Dibromo-m-xylene	9.17	11.51
2-Bromobiphenyl	8.54	12.49

<sup>a</sup> See Table 7 for GC operating conditions.

<sup>b</sup> Not detected at 2 ng per injection.

TABLE 7

GC OPERATING CONDITIONS FOR ORGANOCHLORINE PESTICIDES  
FOR DUAL-COLUMN METHOD OF ANALYSIS  
LOW TEMPERATURE, THIN FILM

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Column 1:	DB-1701 or equivalent 30 m x 0.53 mm ID 1.0 µm film thickness
Column 2:	DB-5 or equivalent 30 m x 0.53 mm ID 0.83 µm film thickness
Carrier gas	Helium
Carrier gas flow rate	6 mL/minute
Makeup gas	Nitrogen
Makeup gas flow rate	20 mL/min
Injector temperature	250°C
Detector temperature	320°C
Initial temperature	140°C, hold 2 minutes
Temperature program	140°C to 270°C at 2.8°C/min
Final temperature	270°C, hold 1 minute

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TABLE 8

GC OPERATING CONDITIONS FOR ORGANOCHLORINE PESTICIDES  
FOR THE DUAL COLUMN METHOD OF ANALYSIS  
HIGH TEMPERATURE, THICK FILM

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Column 1:	DB-1701 (J&W) or equivalent 30 m x 0.53 mm ID 1.0 $\mu$ m
Column 2:	DB-5 (J&W) or equivalent 30 m x 0.53 mm ID 1.5 $\mu$ m
Carrier gas:	Helium
Carrier gas flowrate:	6 mL/minute
Makeup gas:	Nitrogen
Makeup gas flowrate:	20 (mL/min)
Injector temperature:	250°C
Detector temperature:	320°C
Initial temperature:	150°C, hold 0.5 min
Temperature program:	150°C to 190°C at 12°C/min, hold 2 min 190°C to 275°C at 4°C/min
Final temperature	275°C, hold 10 min

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TABLE 9  
ANALYTE RECOVERY FROM SEWAGE SLUDGE

Compound	Ultrasonic Extraction		Soxhlet	
	%Recovery	%RSD	%Recovery	%RSD
Hexachloroethane	80	7	79	1
2-Chloronaphthalene	50	56	67	8
4-Bromodiphenyl ether	118	4	nd	nd
$\alpha$ -BHC	88	25	265	18
$\gamma$ -BHC	55	9	155	29
Heptachlor	60	13	469	294
Aldrin	92	33	875	734
$\beta$ -BHC	351	71	150	260
$\delta$ -BHC	51	11	57	2
Heptachlor epoxide	54	11	70	3
Endosulfan I	52	11	70	4
$\gamma$ -Chlordane	50	9	65	1
$\alpha$ -Chlordane	49	8	66	0
DDE	52	11	74	1
Dieldrin	89	19	327	7
Endrin	56	10	92	15
Endosulfan II	52	10	88	11
DDT	57	10	95	17
Endrin aldehyde	45	6	42	10
DDD	57	11	99	8
Tetrachloro-m-xylene	71	19	82	1
Decachlorobiphenyl	26	23	28	48

Concentration spiked in the sample: 500-1000 ng/g, three replicates analyses.

Soxhlet extraction by Method 3540 with methylene chloride.

Ultrasonic extraction by Method 3550 with methylene chloride/acetone (1:1).

Cleanup by Method 3640.

GC column: DB-608, 30 m x 0.53 mm ID.

TABLE 10

## ANALYTE RECOVERY FROM DICHLOROETHANE STILLBOTTOMS

Compound	Ultrasonic Extraction		Soxhlet	
	%Recovery	%RSD	%Recovery	%RSD
Hexachloroethane	70	2	50	30
2-Chloronaphthalene	59	3	35	35
4-Bromodiphenyl ether	159	14	128	137
$\alpha$ -BHC	55	7	47	25
$\beta$ -BHC	43	6	30	30
Heptachlor	48	6	55	18
Aldrin	48	5	200	258
$\beta$ -BHC	51	7	75	42
$\delta$ -BHC	43	4	119	129
Heptachlor epoxide	47	6	66	34
Endosulfan I	47	4	41	18
$\gamma$ -Chlordane	48	5	47	13
$\alpha$ -Chlordane	45	5	37	21
DDE	45	4	70	40
Dieldrin	45	5	58	24
Endrin	50	6	41	23
Endosulfan II	49	5	46	17
DDT	49	4	40	29
Endrin aldehyde	40	4	29	20
DDD	48	5	35	21
Tetrachloro-m-xylene	49	2	176	211
Decachlorobiphenyl	17	29	104	93

Concentration spiked in the sample: 500-1000 ng/g, three replicates analyses.

Soxhlet extraction by Method 3540 with methylene chloride.

Ultrasonic extraction by Method 3550 with methylene chloride/acetone (1:1).

Cleanup by Method 3640.

GC column: DB-608, 30 m x 0.53 mm ID.

TABLE 11

SINGLE LABORATORY ACCURACY DATA FOR THE EXTRACTION OF  
ORGANOCHLORINE PESTICIDES FROM SPIKED CLAY SOIL BY METHOD 3541  
(AUTOMATED SOXHLET)<sup>a</sup>

Compound Name	% Recovery	
	DB-5	DB-1701
α-BHC	89	94
β-BHC	86	ND
Heptachlor	94	95
Aldrin	ND	92
Heptachlor epoxide	97	97
trans-Chlordane	94	95
Endosulfan I	92	92
Dieldrin	ND	113
Endrin	111	104
Endosulfan II	104	104
4,4'-DDT	ND	ND
Mirex	108	102

<sup>a</sup> The operating conditions for the automated Soxhlet were:

Immersion time 45 min; extraction time 45 min; 10 g sample size; extraction solvent, 1:1 acetone/hexane. No equilibration time following spiking.

ND = Not able to determine because of interference.

All compounds were spiked at 500 µg/kg.

Data taken from Reference 10.

TABLE 12

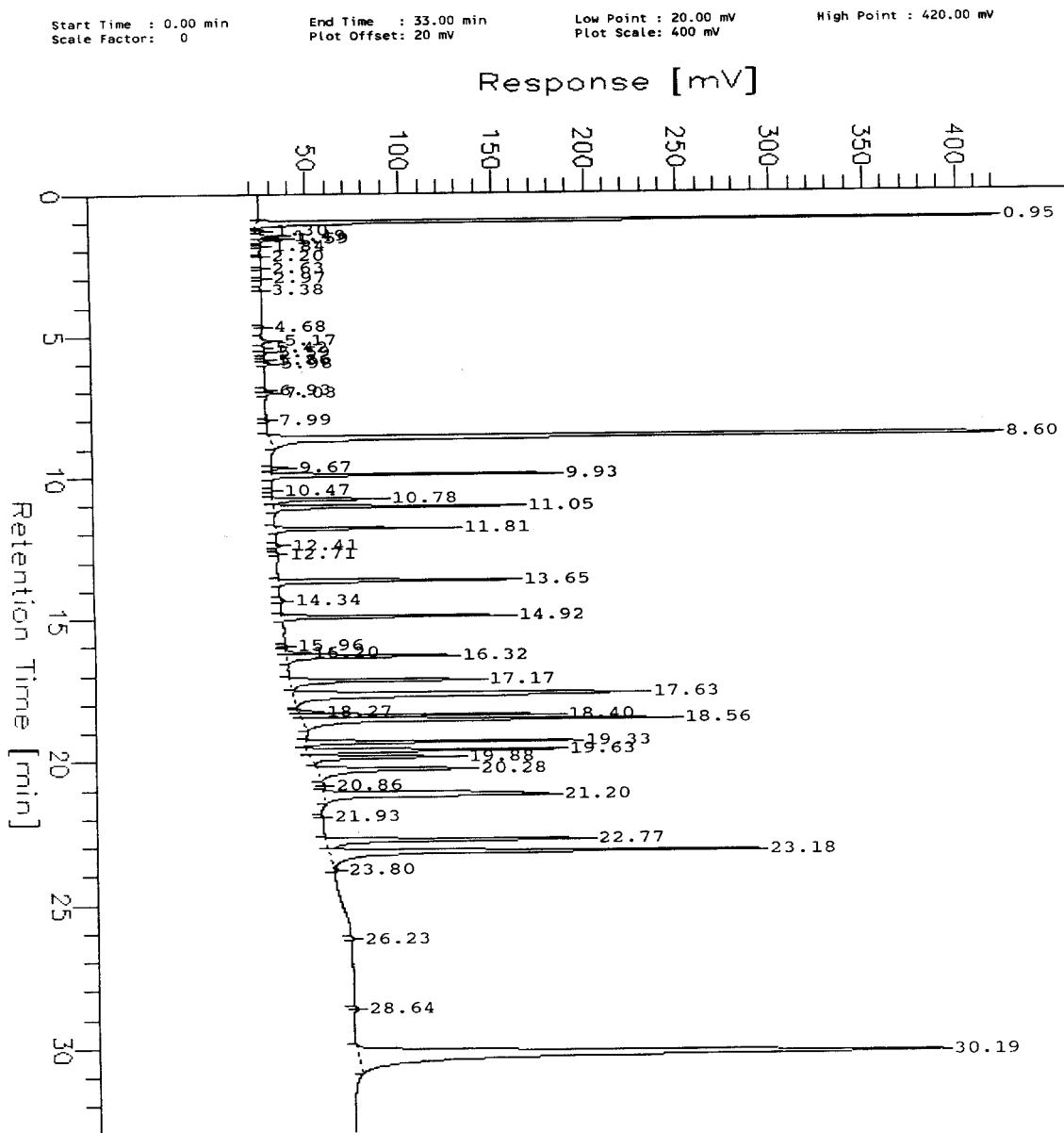
SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR SOLID PHASE EXTRACTION BY METHOD 3535<sup>1</sup>

Compound	Bias (%)				Precision (%)				MDL ( µg/L)	
	Ground water (low)	Ground water (high)	Waste water (low)	Waste water (high)	Ground water (low)	Ground water (high)	Waste water (low)	Waste water (high)	Ground water	Waste water
Aldrin	37.3	93.5	79.3	94.0	23.7	5.5	6.7	3.4	1.4	0.83
β-BHC	89.2	107.8	79.7	82.3	6.5	2.5	1.6	4.2	0.91	0.20
δ-BHC	106.2	86.0	88.9	83.4	5.6	2.4	2.5	4.2	0.93	0.35
α-Chlordane	75.4	112.3	78.9	89.5	12.8	2.7	4.7	2.4	1.5	0.58
γ-Chlordane	70.7	98.9	79.9	93.9	15.8	2.7	4.6	2.9	1.8	0.58
Dieldrin	83.4	96.1	81.2	93.3	7.1	2.3	3.8	3.6	0.9	0.49
Endosulfan I	79.6	99.1	79.6	87.9	10.6	2.3	4.1	3.8	1.3	0.51
Endosulfan II	94.5	101.6	82.7	93.5	5.8	2.8	4.2	4.1	0.9	0.54
Endrin	88.3	98.4	85.1	89.6	6.2	2.3	3.1	2.9	1.7	0.82
Endrin Aldehyde	87.5	99.9	69.0	80.2	6.0	4.0	3.3	5.9	0.8	0.36
Heptachlor	43.1	95.4	71.8	78.6	19.2	3.9	5.0	2.8	1.3	0.56
Heptachlor Epoxide	76.4	97.6	75.3	83.4	12.1	2.4	2.9	3.3	1.5	0.34
Lindane	81.3	115.2	82.1	85.3	11.1	3.2	2.4	3.1	1.4	0.32
p,p'-DDE	80.3	96.0	85.1	97.9	8.3	2.5	4.4	2.4	1.0	0.59
p,p'-DDT	86.6	105.4	105	111	4.4	2.7	4.3	4.7	0.6	0.71
p,p'-TDE (DDD)	90.5	101.1	74.9	79.6	4.8	2.4	4.6	2.9	1.4	0.85

<sup>1</sup>All results determined from seven replicates of each sample type. Two spiking levels were used. "Low" samples were spiked at 5-10 µg/L for each analyte, while "high" samples were spiked at 250 - 500 µg/L. MDL values were determined from the "low" samples without further consideration of the spiking level.

FIGURE 1

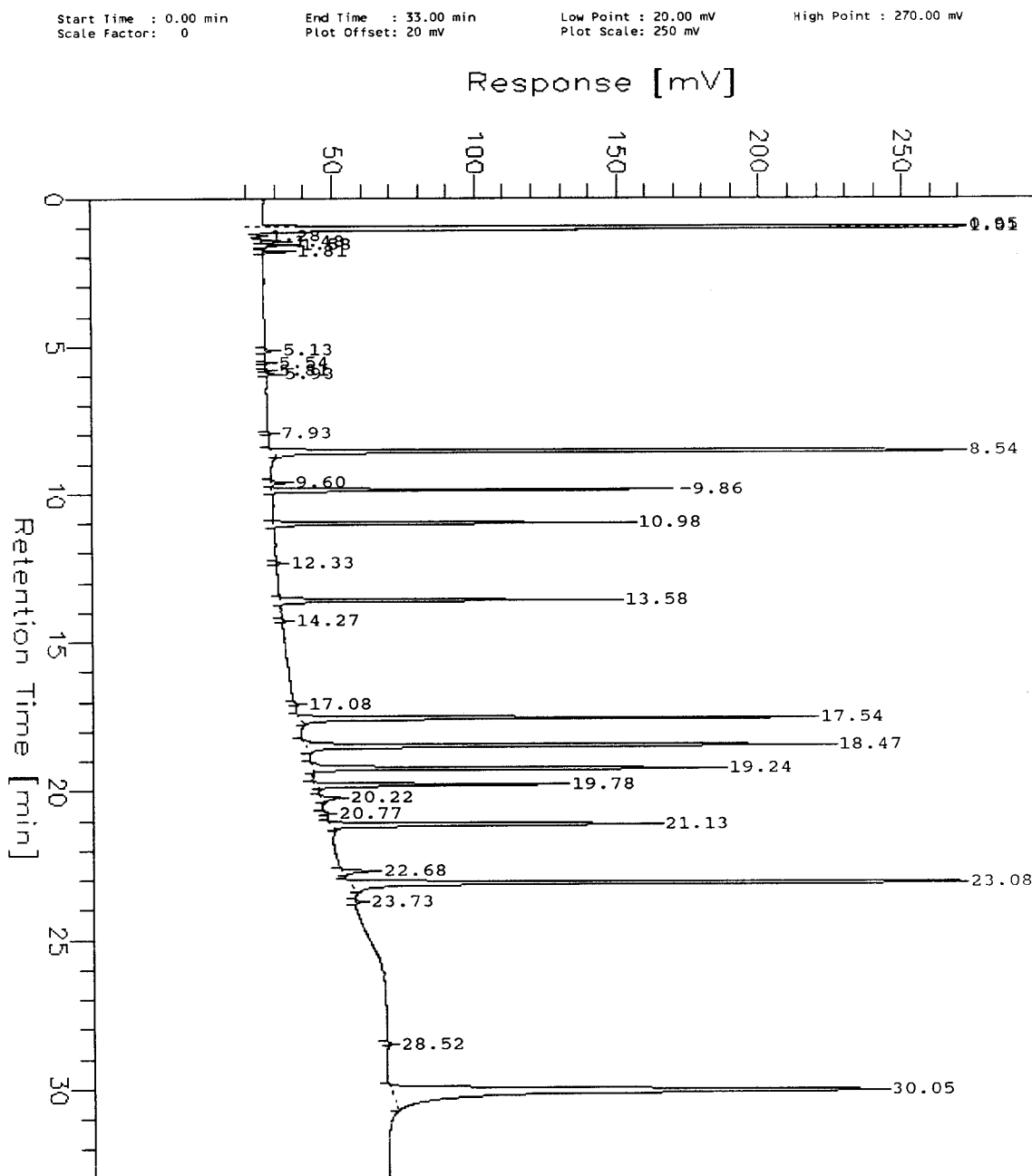
GAS CHROMATOGRAM OF THE MIXED ORGANOCHLORINE PESTICIDE STANDARD



Column: 30 m x 0.25 mm ID, DB-5  
 Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C;  
 carrier He at 16 psi.

FIGURE 2

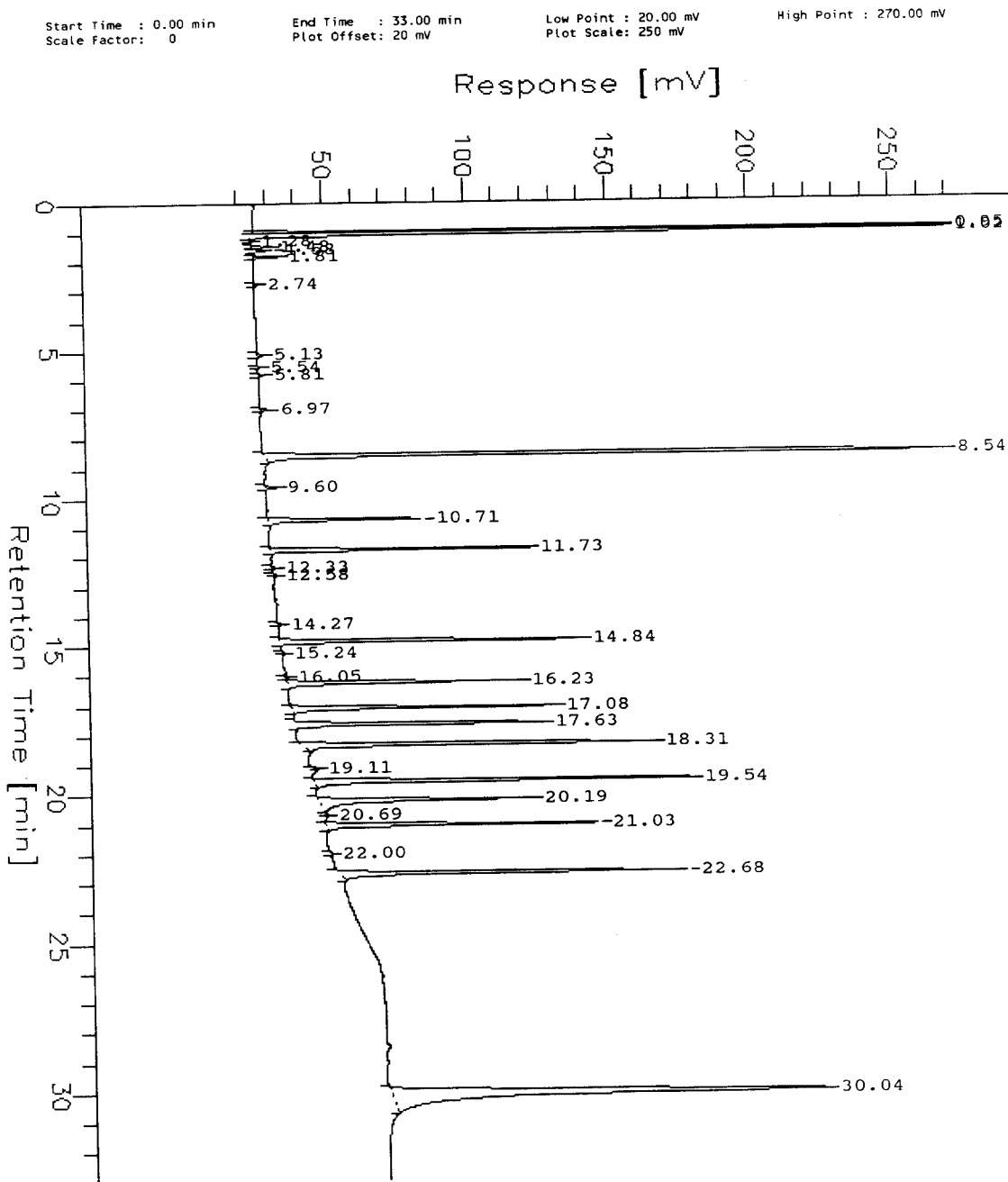
GAS CHROMATOGRAM OF INDIVIDUAL ORGANOCHLORINE PESTICIDE STANDARD MIX A



Column: 30 m x 0.25 mm ID, DB-5  
 Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C;  
 carrier He at 16 psi.

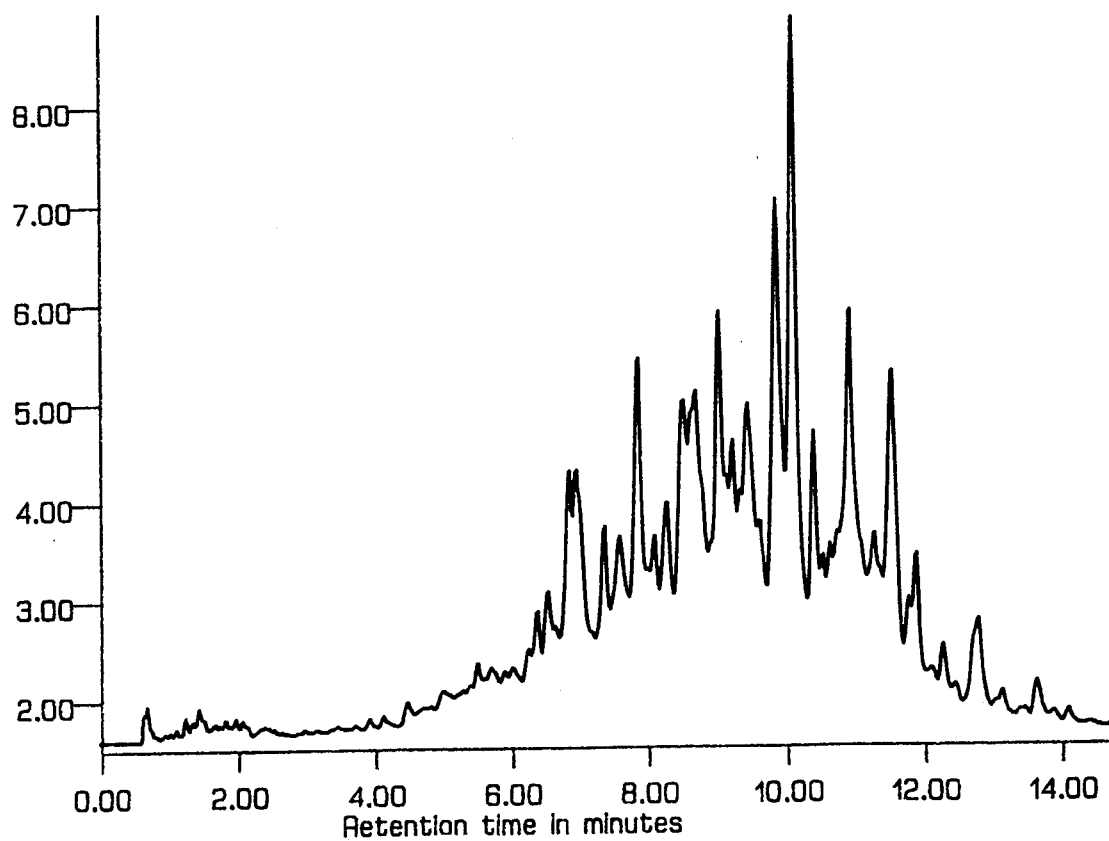
FIGURE 3

GAS CHROMATOGRAM OF INDIVIDUAL ORGANOCHLORINE PESTICIDE STANDARD MIX B



Column: 30 m x 0.25 mm ID, DB-5  
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C;  
carrier He at 16 psi.

FIGURE 4  
GAS CHROMATOGRAM OF TOXAPHENE

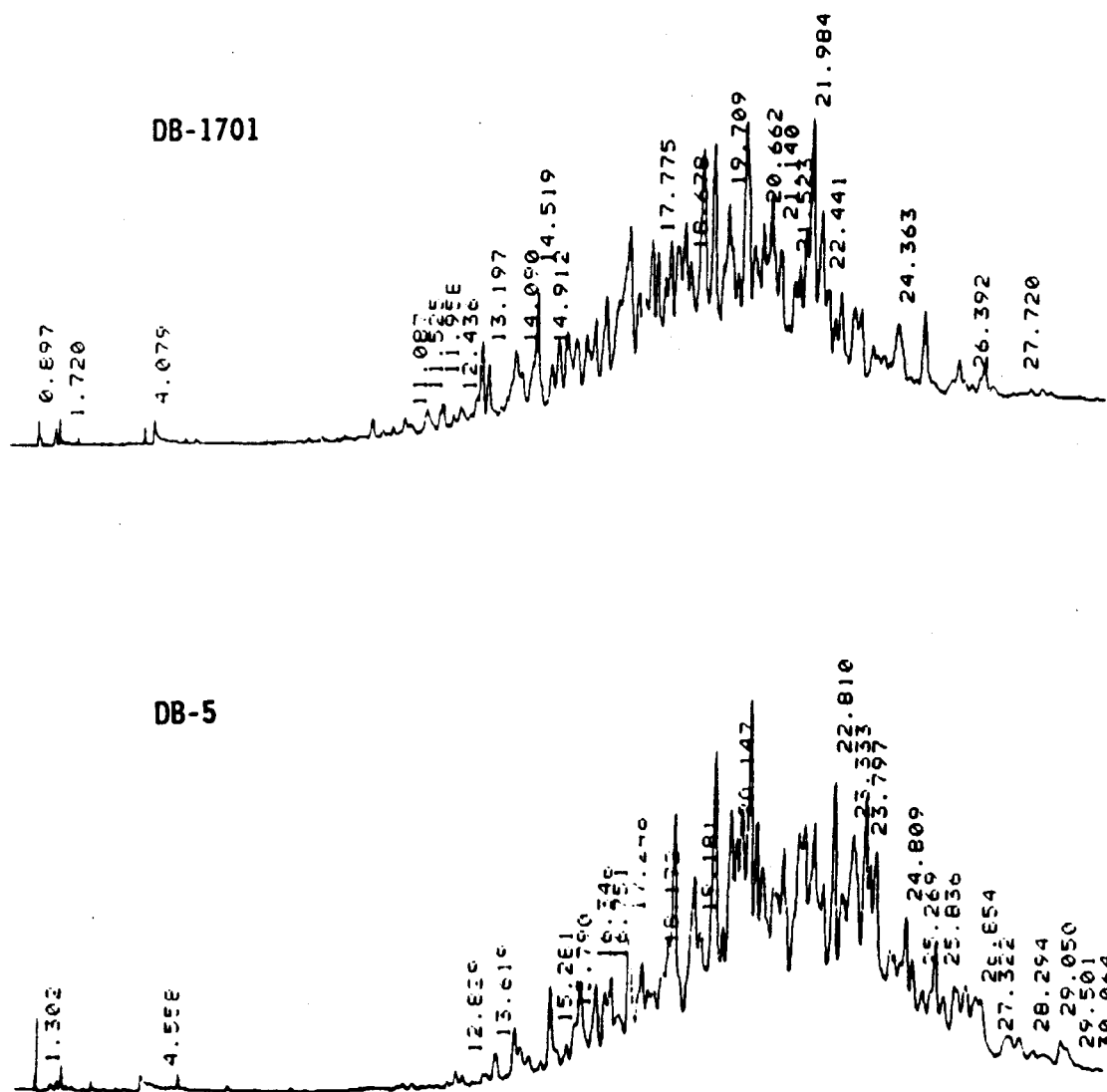


Toxaphene analyzed on an SPB-608 fused-silica open-tubular column. The GC operating conditions were as follows: 30 m x 0.53 mm ID SPB-608. Temperature program: 200°C (2 min hold) to 290°C at 6°C/min.



FIGURE 5

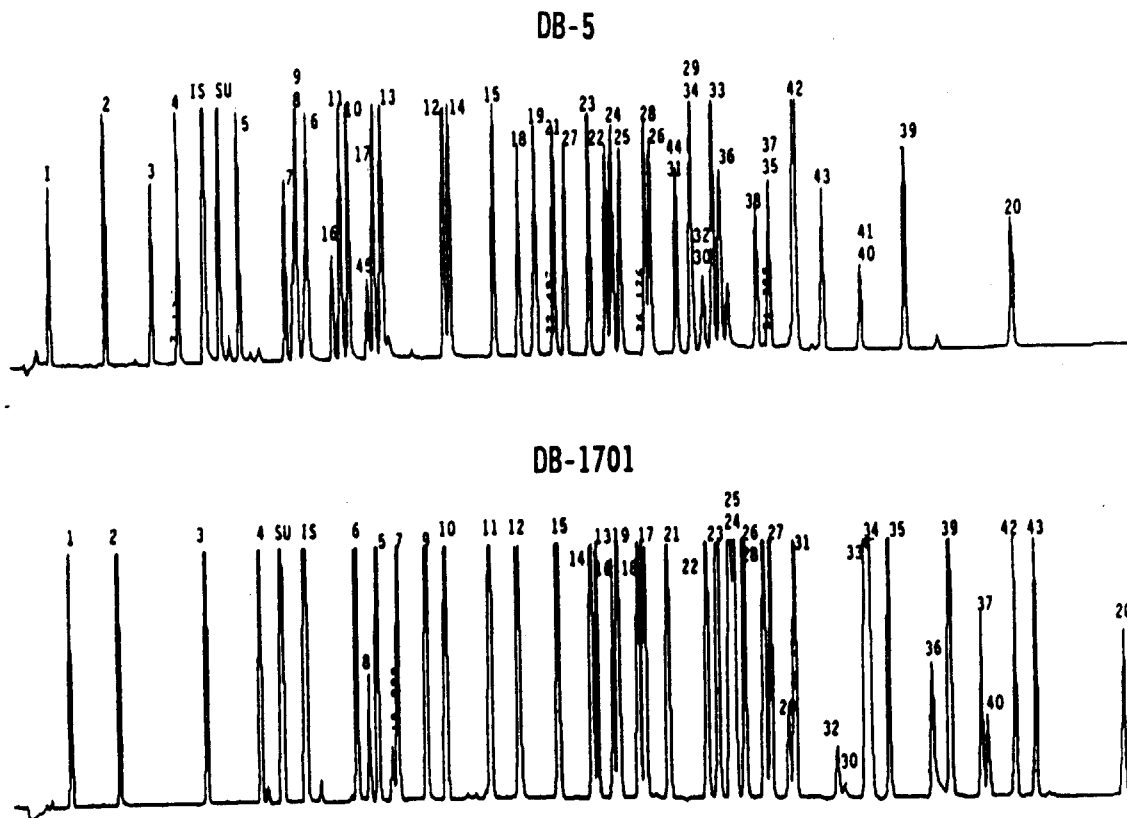
GAS CHROMATOGRAM OF STROBANE



Strobane analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5- $\mu$ m film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0- $\mu$ m film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

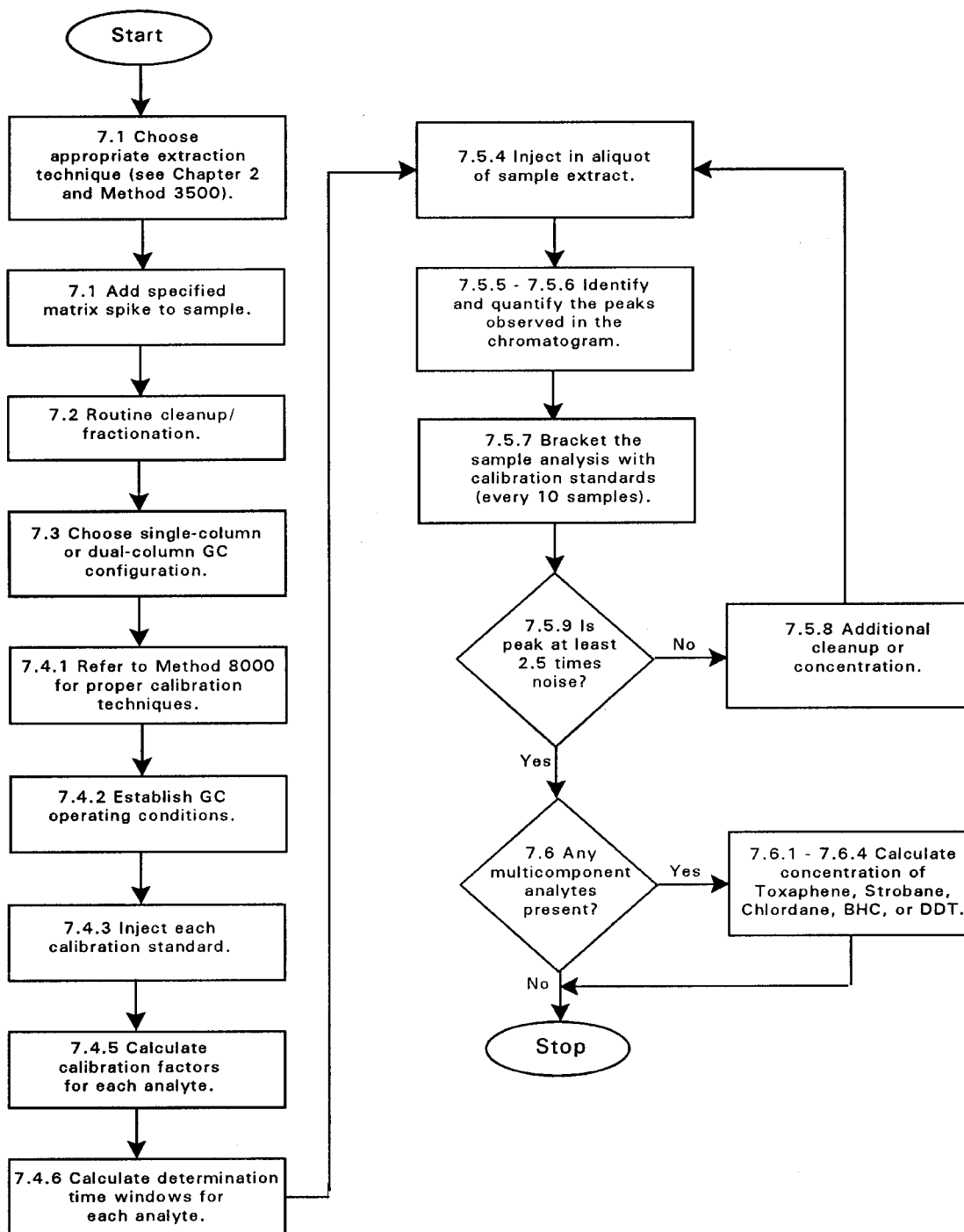
FIGURE 6

GAS CHROMATOGRAM OF ORGANOCHLORINE PESTICIDES



Organochlorine pesticides analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (0.83- $\mu$ m film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0- $\mu$ m film thickness) connected to an 8 in. injection tee (Supelco Inc.). Temperature program: 140°C (2 min hold) to 270°C (1 min hold) at 2.8°C/min.

METHOD 8081A  
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY



## METHOD 8015B

NONHALOGENATED ORGANICS USING GC/FID

## 1.0 SCOPE AND APPLICATION

1.1 Method 8015 is used to determine the concentration of various nonhalogenated volatile organic compounds and semivolatile organic compounds by gas chromatography. The following compounds can be determined quantitatively by this method:

Compound Name	CAS No. <sup>a</sup>	Appropriate Technique		
		Purge-and-Trap	Direct Injection	Solvent Extraction
Acetone	67-64-1	pp	b,d	l
Acetonitrile	75-05-8	pp	b,d	l
Acrolein	107-02-8	pp	b,d	l
Acrylonitrile	107-13-1	pp	b,d	l
Allyl alcohol	107-18-6	ht	b,d	l
1-Butanol (n-Butyl alcohol)	71-36-3	ht	b,d	l
t-Butyl alcohol	75-65-0	pp	b,d	l
2-Chloroacrylonitrile (I.S.)	920-37-6	NA	d	NA
Crotonaldehyde	123-73-9	pp	b,d	l
Diethyl ether	60-29-7	b	b	l
1,4-Dioxane	123-91-1	pp	b,d	l
Ethanol	64-17-5	l	b,d	l
Ethyl acetate	141-78-6	l	b,d	l
Ethylene glycol	107-21-1	l	b	l
Ethylene oxide	75-21-8	l	b,d	l
Hexafluoro-2-propanol (I.S.)	920-66-1	NA	d	NA
Hexafluoro-2-methyl-2-propanol (I.S.)	515-14-6	NA	d	NA
Isobutyl alcohol	78-83-1	pp	b,d	l
Isopropyl alcohol	67-63-0	pp	b,d	l
Methanol	67-56-1	l	b,d	l
Methyl ethyl ketone (MEK)	78-93-3	pp	b,d	l
Methyl isobutyl ketone (MIBK)	108-10-1	pp	b,d	l
N-Nitroso-di-n-butylamine	924-16-3	pp	b,d	b
Paraldehyde	123-63-7	pp	b,d	l
2-Pentanone	107-87-9	pp	b,d	l
2-Picoline	109-06-8	pp	b,d	l
1-Propanol	71-23-8	pp	b,d	l
Propionitrile	107-12-0	ht	d	l

Compound Name	CAS No. <sup>a</sup>	Appropriate Technique		
		Purge-and-Trap	Direct Injection	Solvent Extraction
Pyridine	110-86-1	I	b,d	b
o-Toluidine	95-53-4	I	b,d	b

- <sup>a</sup> Chemical Abstract Services Registry Number.  
b Adequate response using this technique  
d Amenable to concentration by azeotropic distillation (Method 5031)  
ht Method analyte only when purged at 80°C  
I Inappropriate technique for this analyte  
pp Poor purging efficiency, resulting in high EQLs  
NA Not available  
I.S. Internal standard appropriate for Method 5031

1.2 This method may also be applicable to the analysis of petroleum hydrocarbons, including gasoline range organics (GROs) and diesel range organics (DROs). GROs correspond to the range of alkanes from C<sub>6</sub> to C<sub>10</sub> and covering a boiling point range of approximately 60°C - 170°C (Reference 6). DROs correspond to the range of alkanes from C<sub>10</sub> to C<sub>28</sub> and covering a boiling point range of approximately 170°C - 430°C (Reference 6). The identification of specific fuel types may be complicated by environmental processes such as evaporation, biodegradation, or when more than one fuel type is present. Methods from other sources may be more appropriate for GROs and DROs, since these hydrocarbons are not regulated under RCRA. Consult State and local regulatory authorities for specific requirements.

1.3 This method is restricted for use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled in the interpretation of gas chromatograms. In addition, if this method is used for the analysis of petroleum hydrocarbons, it is limited to analysts experienced in the interpretation of hydrocarbon data. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 The method can also be used as a screening tool (for both volatile and semivolatile organics) to obtain semiquantitative data for the prevention of sample overload during quantitative analysis on a GC/MS system. This may be accomplished using an automated (Method 5021) headspace method or by direct injection if a solvent extraction method has been utilized for sample preparation. Single point calibration would be acceptable in this situation. Performance data are not provided for screening.

## 2.0 SUMMARY OF METHOD

2.1 Method 8015 provides gas chromatographic conditions for the detection of certain nonhalogenated volatile and semivolatile organic compounds.

2.1.1 Samples may be introduced into the GC:

- following solvent extraction (Methods 3510, 3520, 3540, 3541, 3545, 3550, or 3560)

- by direct injection (aqueous samples) including the concentration of analytes by azeotropic distillation (Method 5031)
- by purge-and-trap (Methods 5030 or 5035), or
- by vacuum distillation (Method 5032)

2.1.2 Ground or surface water samples must generally be analyzed in conjunction with Methods 5030, 5031, 5032, 3510, 3520, or other appropriate preparatory methods to obtain the necessary quantitation limits. Method 3535 (solid-phase extraction) may also be applicable to the target analytes, but has not yet been validated by EPA in conjunction with Method 8015.

2.1.3 Diesel range organics (DROs) may be prepared by an appropriate solvent extraction method.

2.1.4 Gasoline range organics (GROs) may be introduced into the GC/FID by purge-and-trap, automated headspace, vacuum distillation, or other appropriate technique.

2.2 An appropriate column and temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a flame ionization detector (FID).

2.3 The method allows the use of packed or capillary columns for the analysis and confirmation of the non-halogenated individual analytes. Columns and conditions listed have been demonstrated to provide separation of those target analytes. Analysts may change these conditions as long as they demonstrate adequate performance.

2.4 Fused silica capillary columns are necessary for the analysis of petroleum hydrocarbons.

### 3.0 INTERFERENCES

3.1 When analyzing for volatile organics, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and subsequent storage and handling must serve as a check on such contamination.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a solvent blank to check for cross contamination.

3.2.1 Clean purging vessels with a detergent solution, rinse with distilled water, and then dry in a 105°C oven between analyses. Clean syringes or autosamplers by flushing all surfaces that contact samples using appropriate solvents.

3.2.2 All glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130°C for several hours or rinse with methanol and drain. Store dry glassware in a clean environment.

3.3 The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis.

## 4.0 APPARATUS AND MATERIALS

### 4.1 Gas chromatograph

4.1.1 Gas Chromatograph - Analytical system complete with gas chromatograph suitable for solvent injections or purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

#### 4.1.2 Recommended GC Columns

4.1.2.1 Column 1 - 8 ft x 0.1 in. ID stainless steel or glass column packed with 1% SP-1000 on Carbopack-B 60/80 mesh or equivalent.

4.1.2.2 Column 2 - 6 ft x 0.1 in. ID stainless steel or glass column packed with n-octane on Porasil-C 100/120 mesh (Durapak) or equivalent.

4.1.2.3 Column 3 - 30 m x 0.53 mm ID fused silica capillary column bonded with DB-Wax (or equivalent), 1- $\mu$ m film thickness.

4.1.2.4 Column 4 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 5% methyl silicone (DB-5, SPB-5, RTx, or equivalent), 1.5- $\mu$ m film thickness.

4.1.2.4.1 Capillary columns are needed for petroleum hydrocarbon analyses. Laboratories may use other capillary columns (e.g. 0.25-0.32 mm ID capillary columns) if they document method performance data (e.g. chromatographic resolution and MDLs) if appropriate for the intended use of the data.

4.1.2.4.2 Wide-bore columns should be installed in 1/4-inch injectors, with deactivated liners designed specifically for use with these columns.

#### 4.1.3 Detector - Flame ionization (FID)

### 4.2 Sample introduction and preparation apparatus

4.2.1 Refer to the 5000 series sample preparation methods for the appropriate apparatus.

4.2.2 Samples may also be introduced into the GC via injection of solvent extracts or direct injection of aqueous samples.

### 4.3 Syringes

4.3.1 A 5-mL Luer-Lok glass hypodermic and a 5-mL gas-tight syringe with shutoff valve for volatile analytes.

4.3.2 Microsyringes - 10- and 25- $\mu$ L with a 0.006 in. ID needle (Hamilton 702N or equivalent) and 100- $\mu$ L.

4.4 Volumetric flasks, Class A - Appropriate sizes with ground glass stoppers.

4.5 Analytical balance - 0 - 160 g capacity, capable of measuring differences of 0.0001 g.

## 5.0 REAGENTS

5.1 Reagent grade chemicals shall be used whenever possible. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH<sub>3</sub>OH. Pesticide quality or equivalent. Store away from other solvents.

5.4 Fuels, e.g., gasoline or diesel. Purchase from a commercial source. Low boiling components in fuel evaporate quickly. If available, obtain fuel from the leaking tank on site.

5.5 Alkane standard. A standard containing a homologous series of n-alkanes for establishing retention times (e.g., C<sub>10</sub>-C<sub>32</sub> for diesel).

5.6 Stock standards - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. When methanol is a target analyte or when using azeotropic distillation for sample preparation, standards should not be prepared in methanol. Standards must be replaced after 6 months or sooner, if comparison with check standards indicates a problem.

5.7 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sec. 5.8 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.8 Calibration standards - Calibration standards at a minimum of five different concentrations are prepared in water (purge-and-trap or direct injection) or in methylene chloride (solvent injection) from the secondary dilution of the stock standards. One of the standards should be at or below the concentration equivalent to the appropriate quantitation limit for the project. The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for detection by this method (e.g., some or all of the compounds listed in Sec. 1.1 may be included). Volatile organic standards are prepared in organic-free reagent water. In order to prepare accurate aqueous standard solutions, the following precautions must be observed:

5.8.1 Do not inject more than 20  $\mu$ L of methanolic standards into 100 mL of water.



5.8.2 Use a 25- $\mu$ L Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

5.8.3 Rapidly inject the primary standard into the filled volumetric flask. Remove the needle as fast as possible after injection.

5.8.4 Mix diluted standards by inverting the flask three times only.

5.8.5 Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use any solution contained in the neck of the flask).

5.8.6 Never use pipets to dilute or transfer samples or aqueous standards when diluting volatile organic standards.

5.8.7 Aqueous standards used for purge-and-trap analyses (Method 5030) are not stable and should be discarded after 1 hour, unless held in sealed vials with zero headspace. If so stored, they may be held for up to 24 hours. Aqueous standards used for azeotropic distillation (Method 5031) may be stored for up to a month in polytetrafluoroethylene (PTFE)-sealed screw-cap bottles with minimal headspace, at 4°C, and protected from light.

5.9 Internal standards (if internal standard calibration is used) - To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The following internal standards are recommended when preparing samples by azeotropic distillation: 2-chloroacrylonitrile, hexafluoro-2-propanol and hexafluoro-2-methyl-2-propanol.

5.10 Surrogate standards - Whenever possible, the analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and blank with one or two surrogate compounds which are not affected by method interferences.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

## 7.0 PROCEDURE

### 7.1 Introduction/preparation methods

Various alternate methods are provided for sample introduction. All internal standards, surrogates, and matrix spikes (when applicable) must be added to samples before introduction into the GC/FID system. Follow the introduction method on when to add standards.

7.1.1 Direct injection - This involves direct syringe injection into the GC injection port.

7.1.1.1 Volatile organics (includes gasoline range organics [GROs])

This may involve injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation for nonpurgeable volatile organics); and injection of an organic solvent waste. Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only permitted for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020) to determine if alcohol is present at > 24%.

7.1.1.2 Semivolatile organics (includes diesel range organics [DROs])

This may involve syringe injection of extracts of aqueous samples prepared by Methods 3510 or 3520 or extracts of soil/solids prepared by Methods 3540, 3541, 3545, 3550 or 3560.

**WARNING:** Ultrasonic extraction (Method 3550) is not as rigorous a method as the other extraction methods for soil/solids. This means it is very critical that the method be followed explicitly to achieve extraction efficiency which approaches that of Soxhlet extraction. Consult Method 3550 for information on the critical aspects of this extraction procedure.

7.1.2 Purge and trap - this includes purge and trap for aqueous samples (Method 5030) and purge and trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of solid and oily waste samples by methanol (and other water miscible solvents) with subsequent purge and trap from an aqueous matrix using Method 5030. Normally purge and trap for aqueous samples is performed at ambient temperatures while soil/solid samples utilize a 40°C purge to improve extraction efficiency. Occasionally, there may be a need to perform a heated purge for aqueous samples to lower detection limits; however, a 25-mL sample should provide the sensitivity needed in most situations.

7.1.3 Vacuum distillation - this is a device for the introduction of volatile organics from aqueous, solid or tissue samples (Method 5032) into the GC/FID system.

7.1.4 Automated static headspace - this is a device for the introduction of volatile organics from solid samples (Method 5021) into the GC/FID system.

7.2 Chromatographic conditions (recommended)

7.2.1 Column 1

Carrier gas (Helium) flow rate: 40 mL/min  
Temperature program:  
Initial temperature: 45°C, hold for 3 minutes  
Program: 45°C to 220°C at 8°C/min  
Final temperature: 220°C, hold for 15 minutes.

### 7.2.2 Column 2

Carrier gas (Helium) flow rate: 40 mL/min  
Temperature program:  
Initial temperature: 50°C, hold for 3 minutes  
Program: 50°C to 170°C at 6°C/min  
Final temperature: 170°C, hold for 4 minutes.

### 7.2.3 Column 3

Carrier gas (Helium) flow rate: 15 mL/min  
Temperature program:  
Initial temperature: 45°C, hold for 4 minutes  
Program: 45°C to 220°C at 12°C/min  
Final temperature: 220°C, hold for 3 minutes.

### 7.2.4 Column 4 (DROs)

Carrier gas (Helium) flow rate: 5-7 mL/minute  
Makeup gas (Helium) flow rate: 30 mL/min  
Injector temperature: 200°C  
Detector temperature: 340°C  
Temperature program:  
Initial temperature: 45°C, hold 3 minute  
Program: 45°C to 275°C at 12°C/min  
Final temperature: 275°C, hold 12 min

### 7.2.5 Column 4 (GROs)

Carrier gas (Helium) flow rate: 5-7 mL/minute  
Makeup gas (Helium) flow rate: 30 mL/min  
Injector temperature: 200°C  
Detector temperature: 340°C  
Temperature program:  
Initial temperature: 45°C, hold 1 minute  
Program: 45°C to 100°C at 5°C/min  
Final temperature: 100°C to 275°C, at 8°C/min  
Final hold: 5 min

## 7.3 Initial calibration

7.3.1 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each sample introduction mode because of the differences in conditions and equipment. Establish chromatographic operating parameters that provide instrument performance equivalent to that documented in this method. Prepare calibration standards using the procedures described above (Sec. 5.8). The external standard technique is described below. Analysts wishing to use the internal standard technique are referred to Method 8000. Recommended internal standards for the non-purgeable volatiles include hexafluoro-2-propanol, hexafluoro-2-methyl-2-propanol, and 2-chloroacrylonitrile.

### 7.3.2 External standard calibration procedure for single component analytes

7.3.2.1 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent. One of the external standards should be at a concentration at or below the quantitation limit necessary for the project (based on the concentration in the final volume specified in the preparation method, with no dilutions). The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2.2 Introduce each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Tabulate peak height or area responses against the mass injected. Calculate the calibration factor (CF) for each single component analyte as described in Method 8000.

### 7.3.3 External standard calibration procedure for DROs and GROs

The calibration of DROs and GROs is markedly different from that for single component analytes. In particular, the response used for calibration must represent the entire area of the chromatogram within the retention time range for the fuel type (DROs or GROs), including the unresolved complex mixture that lies below the individual peaks. See Sec. 7.7.2 for information on calculating this area.

7.3.3.1 For each fuel type, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent. One of the external standards should be at a concentration at or below the quantitation limit necessary for the project (based on the concentration in the final volume specified in the preparation method, with no dilutions). The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

NOTE: Whenever possible, the calibration should be performed using the specific fuel that is contaminating the site (e.g., a sample of the fuel remaining in the tank suspected of leaking). Where such samples are not available or not known, use recently purchased commercially-available fuel. A qualitative screening injection and GC run may be performed to identify unknown fuels.

7.3.3.2 Introduce each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Determine the area of the response as described in Sec. 7.7.2. Calculate the calibration factor (CF) for each fuel type as shown below:

$$\text{Calibration Factor} = \frac{\text{Total Area within Retention Time Range}}{\text{Mass injected (in nanograms)}}$$

### 7.3.4 Calibration linearity

The linearity of the calibration must be assessed. This applies to both the single component analytes and the fuel types.

7.3.4.1 If the percent relative standard deviation (%RSD) of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve.

7.3.4.2 If the % RSD is more than 20% over the working range, linearity through the origin cannot be assumed. See Method 8000 for other calibration options that may be employed.

#### 7.4 Retention time windows

Single component target analytes (see Sec. 1.1) are identified on the basis of retention time windows. GROs and DROs are distinguished on the basis of the ranges of retention times for characteristic components in each type of fuel.

7.4.1 Before establishing retention time windows, make sure that the chromatographic system is functioning reliably and that the operating parameters have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. Establish the retention time windows for single component target analytes using the procedure described in Sec. 7.0 of Method 8000.

7.4.2 The retention time range for GROs is defined during initial calibration. Two specific gasoline components are used to establish the range, 2-methylpentane and 1,2,4-trimethylbenzene. Use the procedure described in Sec. 7.0 of Method 8000 to establish the retention time windows for these two components. The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component.

7.4.3 The retention time range for DROs is defined during initial calibration. The range is established from the retention times of the C<sub>10</sub> and C<sub>28</sub> alkanes. Use the procedure described in Sec. 7.0 of Method 8000 to establish the retention time windows for these two components. The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component.

#### 7.5 Calibration verification

7.5.1 The working calibration curve, and retention times must be verified at the beginning of each 12-hour work shift as a minimum requirement. Verification is accomplished by the measurement of one or more calibration standards (normally mid-concentration) that contain all of the target analytes and surrogates when individual target analytes are being analyzed. Verification is accomplished by the measurement of the fuel standard and the hydrocarbon retention time standard when petroleum hydrocarbons are being analyzed. Additional analyses of the verification standard(s) throughout a 12-hour shift are strongly recommended, especially for samples that contain visible concentrations of oily material. See Sec. 7.0 "calibration verification" of Method 8000 for more detailed information.

7.5.2 Calculate the % difference as detailed in Sec. 7.0 of Method 8000. If the response for any analyte is within  $\pm 15\%$  of the response obtained during the initial calibration, then the initial calibration is considered still valid, and analyst may continue to use the mean CF or RF values from the initial calibration to quantitate sample results. For analyses employing azeotropic distillation as the sample introduction technique, the % difference may be up to  $\pm 20\%$ . If the response for any analyte varies from the predicted response by more

than  $\pm 15\%$  ( $\pm 20\%$  for azeotropic distillation), corrective action must be taken to restore the system or a new calibration curve must be prepared for that compound.

7.5.3 All target analytes and surrogates or n-alkanes in the calibration verification analyses must fall within previously established retention time windows. If the retention time of any analyte does not fall within the  $\pm 3\sigma$  window, corrective action must be taken to restore the system or a new calibration curve must be prepared for that compound.

7.5.4 Solvent blanks and any method blanks should be run with calibration verification analyses to confirm that laboratory contamination does not cause false positives.

## 7.6 Gas chromatographic analysis

7.6.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with calibration verification followed by sample extract analyses. Additional analyses of the verification standard(s) throughout a 12-hour shift are strongly recommended, especially for samples that contain visible concentrations of oily material. A verification standard is also necessary at the end of a set. The sequence ends when the set of samples has been injected or when retention time and/or % difference QC criteria are exceeded.

If the criteria are exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with sample analysis. All sample analyses performed using external standard calibration must be bracketed with acceptable data quality analyses (e.g., calibration and retention time criteria). Therefore, all samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.

7.6.2 Samples are analyzed with the same instrument configuration as is used during calibration. When using Method 5030 for sample introduction, analysts are cautioned that opening a sample vial or drawing an aliquot from a sealed vial (thus creating headspace) will compromise samples analyzed for volatiles. Therefore, it is recommended that analysts prepare two samples for purge-and-trap analysis. The second sample can be stored for 24 hours to ensure that an uncompromised sample is available for analysis or dilution, if the analysis of the first sample is unsuccessful or if results exceed the calibration range of the instrument. Distillates from Method 5031 may be split into two portions and held at  $4^{\circ}\text{C}$  prior to analysis. It is recommended that the distillate be analyzed within 24 hours of distillation. Distillates must be analyzed within 7 days of distillation.

7.6.3 Sample concentrations are calculated by comparing sample response data with the initial calibration of the system (Sec. 7.3). Therefore, if sample response exceeds the limits of the initial calibration range, a dilution of the sample must be analyzed. For volatile organic aqueous samples, the dilution must be performed on a second aliquot of the sample which has been properly sealed and stored prior to use and reanalysis. Extracts should be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable as long as calibration limits are not exceeded. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.6.4 Tentative identification of a single component analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is required on a second column or by GC/MS. Since the flame ionization detector is non-specific, it is highly

recommended that GC/MS confirmation be performed on single component analytes unless historical data are available to support the identification(s).

7.6.5 Second column confirmation is generally not necessary for petroleum hydrocarbon analysis. However, if analytical interferences are indicated, analysis using the second GC column is required. Also, the analyst must ensure that the sample hydrocarbons fall within the retention time range established during the initial calibration.

NOTE: Identification of fuels, especially gasoline, is complicated by their inherent volatility. The early eluting compounds in fuels are obviously the most volatile and the most likely to have weathered unless sampled immediately following a spill. The most highly volatile fraction of gasoline constitutes 50% of the total peak area of a gasoline chromatogram. This fraction is least likely to be present in an environmental sample or present in only very low concentration in relation to the remainder of a gasoline chromatogram.

7.6.6 The performance of the entire analytical system should be checked every 12 hours, using data gathered from analyses of blanks, standards, and replicate samples. Significant peak tailing must be corrected. Tailing problems are generally traceable to active sites on the column, cold spots in a GC, the detector operation, or leaks in the system. See Sec. 7.9 for GC/FID system maintenance. Follow manufacturer's instructions for maintenance of the introduction device.

## 7.7 Calculations

7.7.1 The concentration of each analyte in the sample may be determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the mean CF or RF from the initial curve.

7.7.2 While both diesel fuel and gasoline contain a large number of compounds that will produce well resolved peaks in a GC/FID chromatogram, both fuels contain many other components that are not chromatographically resolved. This unresolved complex mixture results in the "hump" in the chromatogram that is characteristic of these fuels. In addition, although the resolved peaks are important for the identification of the specific fuel type, the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

7.7.2.1 For the analysis of DROs, sum the area of all peaks eluting between  $C_{10}$  and  $C_{28}$ . This area is generated by projecting a horizontal baseline between the retention times of  $C_{10}$  and  $C_{28}$ .

7.7.2.2 Because the chromatographic conditions employed for DRO analysis can result in significant column bleed and a resulting rise in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the DRO chromatogram. In order to accomplish this subtraction, a methylene chloride blank should be analyzed during each 12-hour analytical shift during which samples are analyzed for DROs. The area of this chromatogram is measured in the same fashion as is used for samples (see Sec. 7.7.2.1), by projecting a horizontal baseline across the retention time range for DROs. This area is then subtracted from the area measured for the sample and the difference in areas is used to calculate the DRO concentration, using the equations in Method 8000.

7.7.2.3 For the analysis of GROs, sum the area of all peaks eluting between 2-methylpentane and 1,2,4-trimethyl benzene. This area is used to calculate the GRO concentration, using the equations in Method 8000. Column bleed subtraction is not generally required for GRO analysis.

7.7.3 Refer to Method 8000, Sec. 7.0 for calculation formulae. The formulae cover external and internal standard calibration, aqueous and non-aqueous samples and linear and non-linear calibration curves.

## 7.8 Screening

7.8.1 Method 8015 with single-point calibration can also be used for GC/FID screening in order to reduce instrument down-time when highly contaminated samples are analyzed using GC/MS (e.g., Methods 8260 and 8270).

7.8.2 The same configuration of introduction device interfaced to the GC/MS may be utilized for the GC/FID or alternative configurations are acceptable.

7.8.3 Establish that the system response and chromatographic retention times are stable. Analyze the high-point GC/MS calibration standard.

7.8.4 Analyze samples or sample extracts. Compare peak heights in the sample chromatograms with the high-point standard to establish that no compound with the same retention time as a target analyte exceeds the calibration range. However, the FID is much less sensitive to halogenated compounds than the GC/MS system, therefore, the above comparison is not an absolute certainty.

7.8.5 It is recommended that the high-point standard should be run at least every 12 hours to confirm the stability of instrument response and chromatographic retention times. However, there is no QC requirement for screening.

## 7.9 Instrument Maintenance

7.9.1 Injection of sample extracts from waste sites often leaves a high boiling residue in: the injection port area, splitters when used, and the injection port end of the chromatographic column. This residue effects chromatography in many ways (i.e., peak tailing, retention time shifts, analyte degradation, etc.) and, therefore, instrument maintenance is very important. Residue buildup in a splitter may limit flow through one leg and therefore change the split ratios. If this occurs during an analytical run, the quantitative data may be incorrect. Proper cleanup techniques will minimize the problem and instrument QC will indicate when instrument maintenance is required.

### 7.9.2 Suggested chromatograph maintenance

Corrective measures may require any one or more of the following remedial actions. Also see Sec. 7.0 in Method 8000 for additional guidance on corrective action for capillary columns and the injection port.

7.9.2.1 Splitter connections - For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter or replace with a cleaned and deactivated splitter. Break off the first few inches (up to one foot) of the injection port side of the column. Remove the



columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

7.9.2.2 Column rinsing - The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone; methylene chloride is a satisfactory final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to remain flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen passing through the column.

## 8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.2 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a

potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.3 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 9.0 METHOD PERFORMANCE

9.1 Specific method performance information for non-purgeable volatiles prepared using the azeotropic microdistillation technique from Method 5031 is included in Tables 1, 3 and 4 for aqueous matrices and in Tables 2 and 5 for solid matrices.

9.2 Specific method performance information is provided for diesel fuel spiked into soil in Tables 6 and 7.

## 10.0 REFERENCES

1. Bellar, T.A., and J.J. Lichtenberg. "Determining Volatile Organics at Microgram-per-Liter Levels by Gas Chromatography", J. Amer. Water Works Assoc., 66(12), pp. 739-744 (1974).
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3. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters: Category 11 - Purgeables and Category 12 - Acrolein, Acrylonitrile, and Dichlorodifluoromethane, Report for EPA Contract 68-03-2635.
4. Bruce, M.L., R.P. Lee, and M.W. Stevens. "Concentration of Water Soluble Volatile Organic Compounds from Aqueous Samples by Azeotropic Microdistillation", Environ. Sci. Technol. 1992, 26, 160-163.
5. Tsang, S.F., N. Chau, P.J. Marsden, and K.R. Carter. "Evaluation of the EnSys PETRO RISc kit for TPH", Report for Ensys, Inc., Research Triangle Park, NC, 27709, 1992.
6. "Interlaboratory Study of Three Methods for Analyzing Petroleum Hydrocarbons in Soils," API Publication Number 4599, American Petroleum Institute, March 1994.

TABLE 1

METHOD DETECTION LIMITS FOR NON-PURGEABLE VOLATILE COMPOUNDS  
IN AQUEOUS MATRICES BY AZEOTROPIC MICRODISTILLATION (METHOD 5031)

Analyte	MDL ( $\mu\text{g/L}$ ) <sup>a</sup>		
	Reagent Water	Ground Water	TCLP Leachate
Acetone <sup>b</sup>	48	16	63
Acetonitrile	15	6	14
Acrolein	13	15	7
Acrylonitrile	8	9	14
1-Butanol	14	8	7
t-Butyl alcohol	8	7	17
1,4-Dioxane	12	15	16
Ethanol	18	12	13
Ethyl acetate	9	8	16
Ethylene oxide	8	9	10
Isobutyl alcohol	11	8	4
Isopropyl alcohol	18	17	7
Methanol	21	21	22
Methyl ethyl ketone	4	5	9
Methyl isobutyl ketone	4	2	8
2-Pentanone	2	2	7
1-Propanol	--	7	--
Propionitrile	10	6	13
Pyridine	11	9	21

<sup>a</sup> Produced by analysis of 7 aliquots of water spiked at 25  $\mu\text{g/L}$ , using internal standard calibration.

<sup>b</sup> Problematic due to transient laboratory contamination.

TABLE 2

METHOD DETECTION LIMITS FOR NON-PURGEABLE VOLATILE COMPOUNDS  
IN SOLID MATRICES BY AZEOTROPIC MICRODISTILLATION (METHOD 5031)

Analyte	MDL (mg/kg)	
	Incinerator Ash	Kaolin
Acrylonitrile	0.42	0.09
1-Butanol	0.23	0.09
t-Butyl alcohol	0.34	0.13
1,4-Dioxane	0.31	0.16
Ethanol	0.47	0.19
Ethyl acetate	0.18	0.07
Isopropyl alcohol	0.40	0.19
Methanol	0.46	0.31
Methyl ethyl ketone	0.27	0.12
Methyl isobutyl ketone	0.12	0.05
2-Pentanone	0.16	0.07
Pyridine	0.20	0.08

The MDLs calculated for this table were produced by the analysis of 7 replicates spiked at 0.50 mg/kg, using internal standard calibration.

TABLE 3

## METHOD PERFORMANCE DATA FOR NON-PURGEABLE VOLATILES IN GROUND WATER BY AZEOTROPIC MICRODISTILLATION (METHOD 5031)

Compound	<u>Low Conc.<sup>a</sup></u>		<u>Medium Conc.<sup>b</sup></u>		<u>High Conc.<sup>c</sup></u>	
	Average <sup>d</sup> %Rec	%RSD	Average <sup>d</sup> %Rec	%RSD	Average <sup>d</sup> %Rec	%RSD
Acetone <sup>e</sup>	126	17	N/A	--	N/A	--
Acetonitrile	147	5	105	8	92	9
Acrolein	146	13	120	27	80	20
Acrylonitrile	179	7	143	28	94	21
1-Butanol	127	8	86	8	90	9
t-Butyl alcohol	122	7	N/A	--	N/A	--
1,4-Dioxane	124	16	96	10	99	8
Ethanol	152	10	N/A	--	N/A	--
Ethyl Acetate	142	7	135	33	92	25
Ethylene oxide	114	10	N/A	--	N/A	--
Isobutyl alcohol	122	8	87	13	89	13
Isopropyl alcohol	167	13	N/A	--	N/A	--
Methanol	166	14	94	9	95	7
Methyl ethyl ketone	105	6	N/A	--	N/A	--
Methyl isobutyl ketone	66	4	N/A	--	N/A	--
2-Pentanone	94	3	N/A	--	N/A	--
1-Propanol	N/A	--	91	7	91	7
Propionitrile	135	5	102	14	90	14
Pyridine	92	12	N/A	--	N/A	--

- <sup>a</sup> 25 µg/L spikes, using internal calibration.  
<sup>b</sup> 100 µg/L spikes, using internal calibration.  
<sup>c</sup> 750 µg/L spikes, using internal calibration.  
<sup>d</sup> Average of 7 replicates  
<sup>e</sup> Problematic due to transient laboratory contamination.

N/A Data not available

TABLE 4

METHOD PERFORMANCE DATA FOR NON-PURGEABLE VOLATILES IN TCLP  
LEACHATE BY AZEOTROPIC MICRODISTILLATION (METHOD 5031)

Compound	Low Conc. <sup>a</sup>		Medium Conc. <sup>b</sup>		High Conc. <sup>c</sup>	
	Average <sup>d</sup> %Rec	%RSD	Average <sup>d</sup> %Rec	%RSD	Average <sup>d</sup> %Rec	%RSD
Acetone <sup>e</sup>	99	91	N/A	--	N/A	--
Acetonitrile	107	17	111	10	95	11
Acrolein	88	10	109	29	87	41
Acrylonitrile	133	13	123	29	103	38
1-Butanol	119	7	89	12	86	8
t-Butyl alcohol	70	31	N/A	--	N/A	--
1,4-Dioxane	103	20	103	16	102	7
Ethanol	122	13	N/A	--	N/A	--
Ethyl Acetate	164	12	119	29	107	41
Ethylene oxide	111	12	N/A	--	N/A	--
Isobutyl alcohol	115	4	86	13	82	13
Isopropyl alcohol	114	8	N/A	--	N/A	--
Methanol	107	10	102	6	N/A	--
Methyl ethyl ketone	87	13	N/A	--	N/A	--
Methyl isobutyl ketone	78	13	N/A	--	N/A	--
2-Pentanone	101	8	N/A	--	N/A	--
1-Propanol	N/A	--	98	10	89	7
Propionitrile	100	16	100	11	90	17
Pyridine	46	59	N/A	--	N/A	--

- <sup>a</sup> 25 µg/L spikes, using internal calibration.  
<sup>b</sup> 100 µg/L spikes, using internal calibration.  
<sup>c</sup> 750 µg/L spikes, using internal calibration.  
<sup>d</sup> Average of 7 replicates  
<sup>e</sup> Problematic due to transient laboratory contamination.

N/A Data not available

TABLE 5

METHOD PERFORMANCE DATA FOR NON-PURGEABLE VOLATILE COMPOUNDS  
IN SOLID MATRICES BY AZEOTROPIC MICRODISTILLATION (METHOD 5031)

	Incinerator Ash				Kaolin			
	Low Conc. <sup>a</sup>		High Conc. <sup>b</sup>		Low Conc. <sup>a</sup>		High Conc. <sup>b</sup>	
	Average <sup>c</sup>		Average <sup>c</sup>		Average <sup>c</sup>		Average <sup>c</sup>	
	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD
Acrylonitrile	50	53	10	31	102	6	12	52
1-Butanol	105	14	61	12	108	5	58	25
t-Butyl alcohol	101	21	60	13	97	9	59	23
1,4-Dioxane	106	19	48	18	105	10	48	25
Ethanol	117	25	52	20	108	11	48	24
Ethyl acetate	62	19	39	12	90	5	41	25
Isopropyl alcohol	119	21	61	15	108	11	58	24
Methanol	55	53	33	28	117	17	37	22
Methyl ethyl ketone	81	21	40	12	91	8	42	20
Methyl isobutyl ketone	68	11	57	14	71	5	55	23
2-Pentanone	79	13	54	10	91	5	54	19
Pyridine	52	24	44	20	50	10	49	31

<sup>a</sup> 0.5 mg/kg spikes, using internal calibration.

<sup>b</sup> 25 mg/kg spikes, using internal calibration.

<sup>c</sup> Average of 7 replicates

TABLE 6

RESULTS FROM ANALYSIS<sup>a</sup> OF LOW AROMATIC DIESEL<sup>b</sup> BY GC/FID  
(5 replicates/test)

Spike Concentration	Analysis Results
12.5 ppm	ND
75 ppm	54 ± 7 ppm
105 ppm	90 ± 15 ppm
150 ppm	125 ± 12 ppm
1000 ppm	960 ± 105 ppm

<sup>a</sup> Samples were prepared using 2 g aliquots of sandy loam soil spiked with known amounts of low aromatic diesel. Extractions were accomplished using methylene chloride as a solvent (Method 3550, high concentration option).

<sup>b</sup> Low aromatic diesel is sold in California (Section 2256, CCR). For this study it was purchased at a gas station in San Diego, California.

TABLE 7

RESULTS FROM ANALYSIS<sup>a</sup> OF LOW AROMATIC DIESEL<sup>b</sup> BY GC/FID  
(5 replicates/test)

Spike Concentration	Analysis Results
25 ppm	51.2 ± 6.4 ppm
75 ppm	75.9 ± 7.8 ppm
125 ppm	98.9 ± 5.2 ppm
150 ppm	162 ± 10.4 ppm

<sup>a</sup> Samples were prepared using 10 g aliquots of sandy loam soil spiked with known amounts of regular #2 diesel purchased at a gas station in Northern Virginia. Extractions were accomplished using methylene chloride as a solvent (Method 3550).



FIGURE 1

CHROMATOGRAM OF A 300 PPM GASOLINE STANDARD

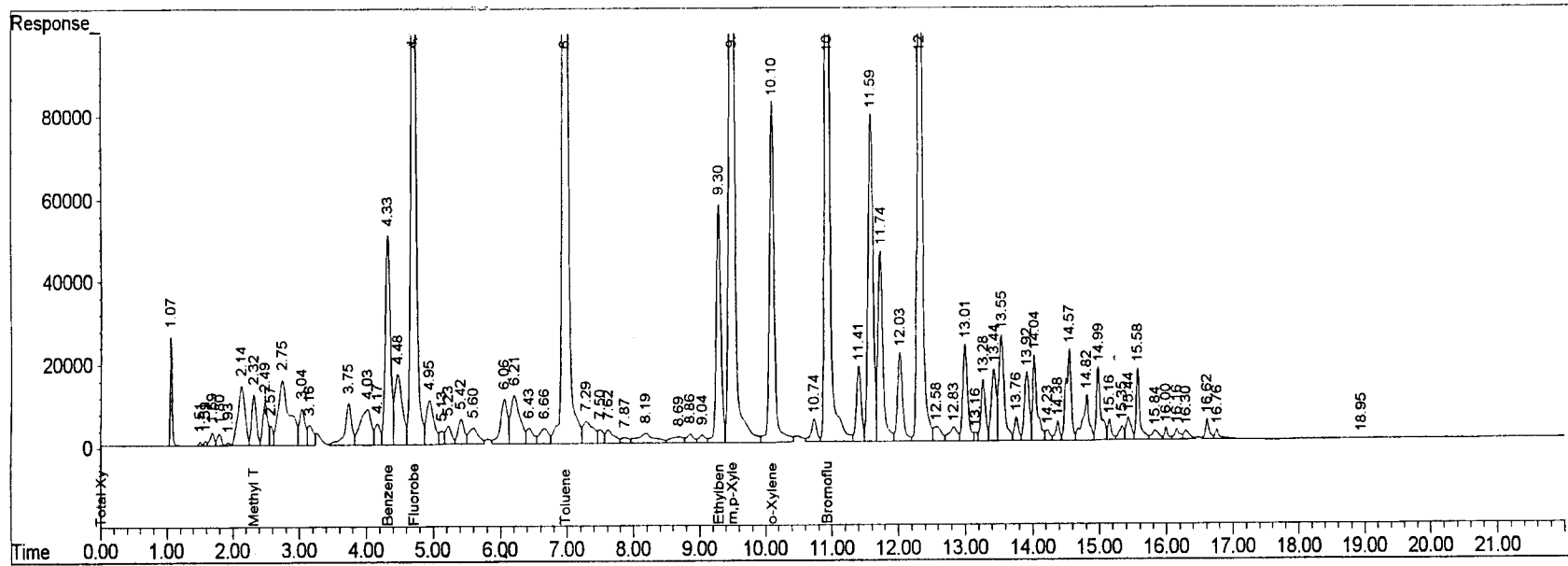


FIGURE 2  
CHROMATOGRAM OF A 30 PPM DIESEL STANDARD

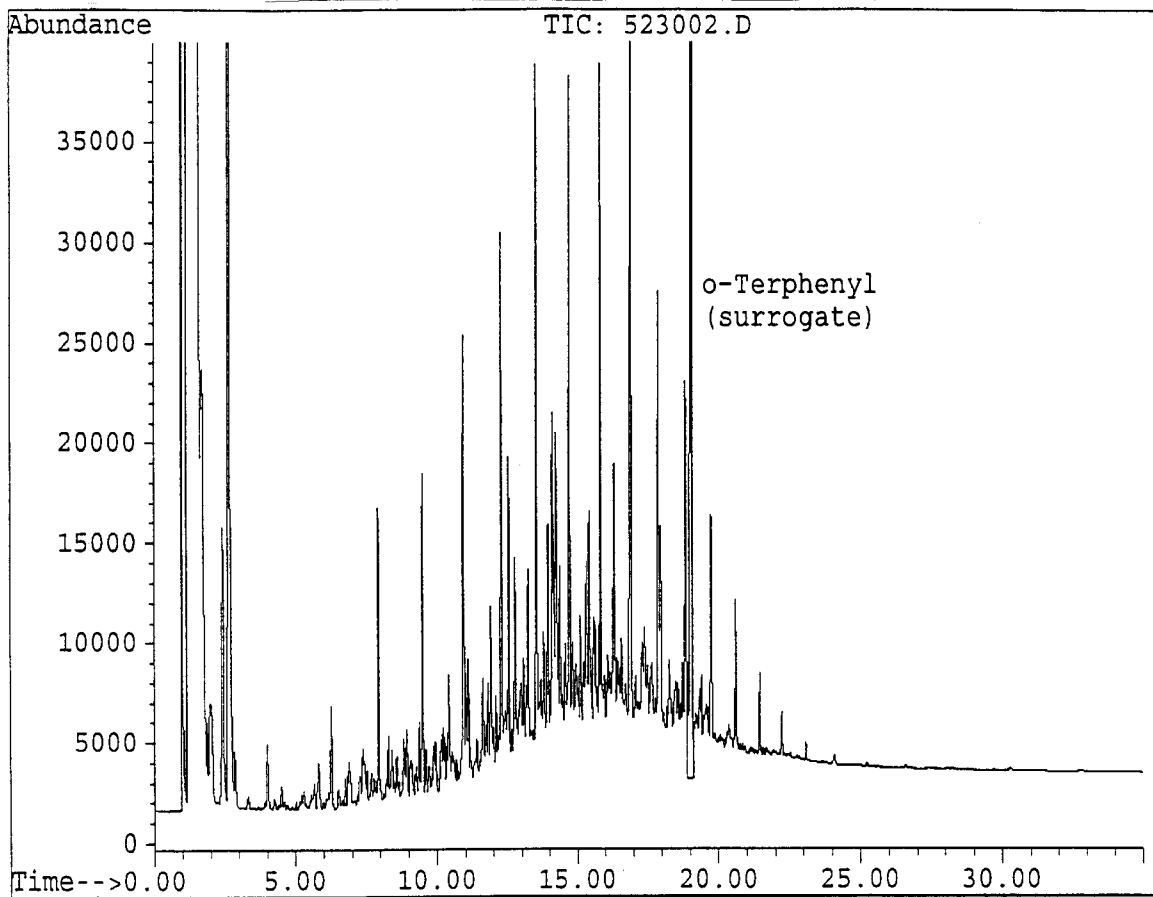


FIGURE 3

CHROMATOGRAM OF A 30 PPM DIESEL STANDARD WITH THE  
BASELINE PROJECTED BETWEEN C<sub>10</sub> AND C<sub>18</sub>

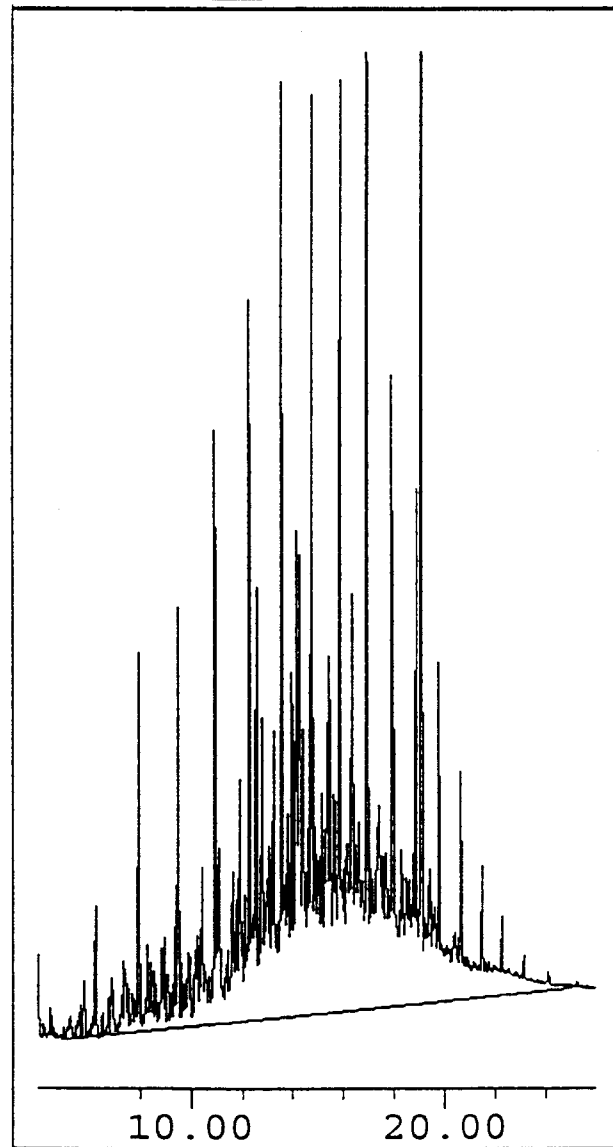
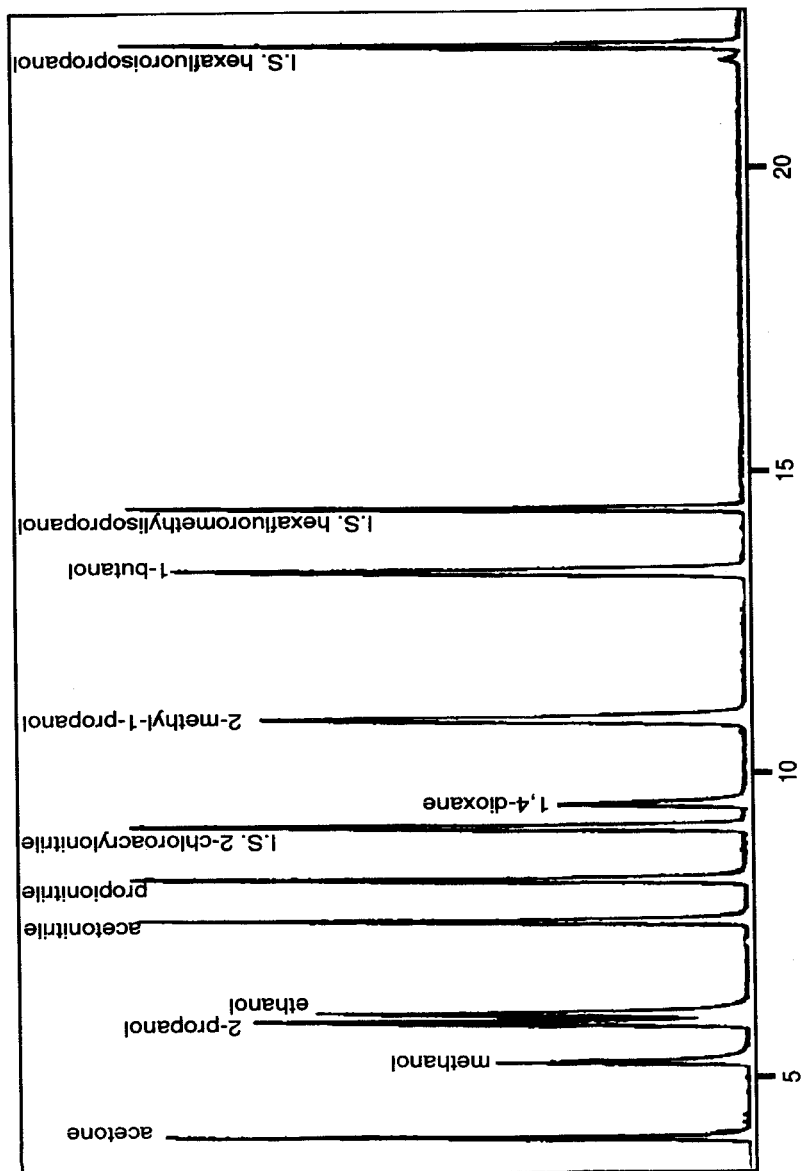


FIGURE 4

CHROMATOGRAM OF SEVERAL NONPURGEABLE VOLATILE COMPOUNDS IN SPIKED REAGENT WATER USING AZEOTROPIC MICRODISTILLATION (METHOD 5031)

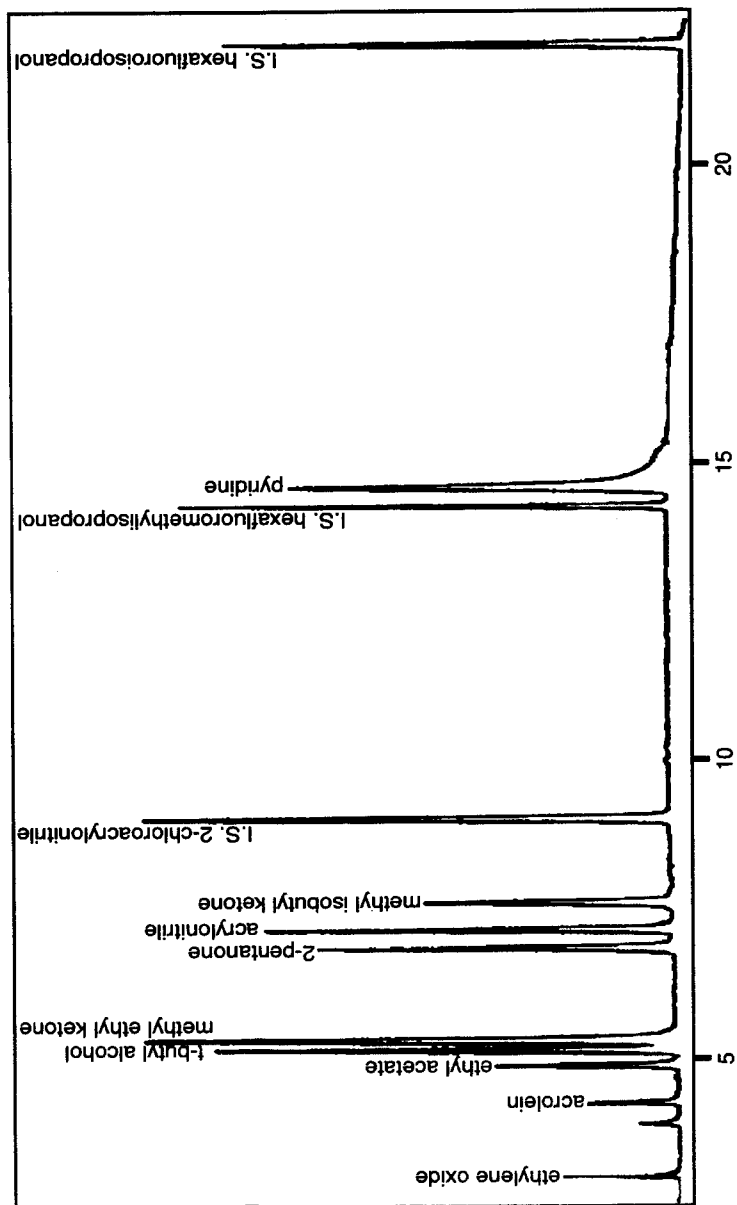


Mix 1: Analytes distilled at 0.25mg/L, Internal Stds. at 2.5 mg/L

Conditions: J&W DB-Wax column with 0.53 ID  
Temperature program: 30°C for 2 min.  
3°C/min. to 100°C and held for 0 min.  
25°C/min. to 200°C and held for 4 min.

FIGURE 5

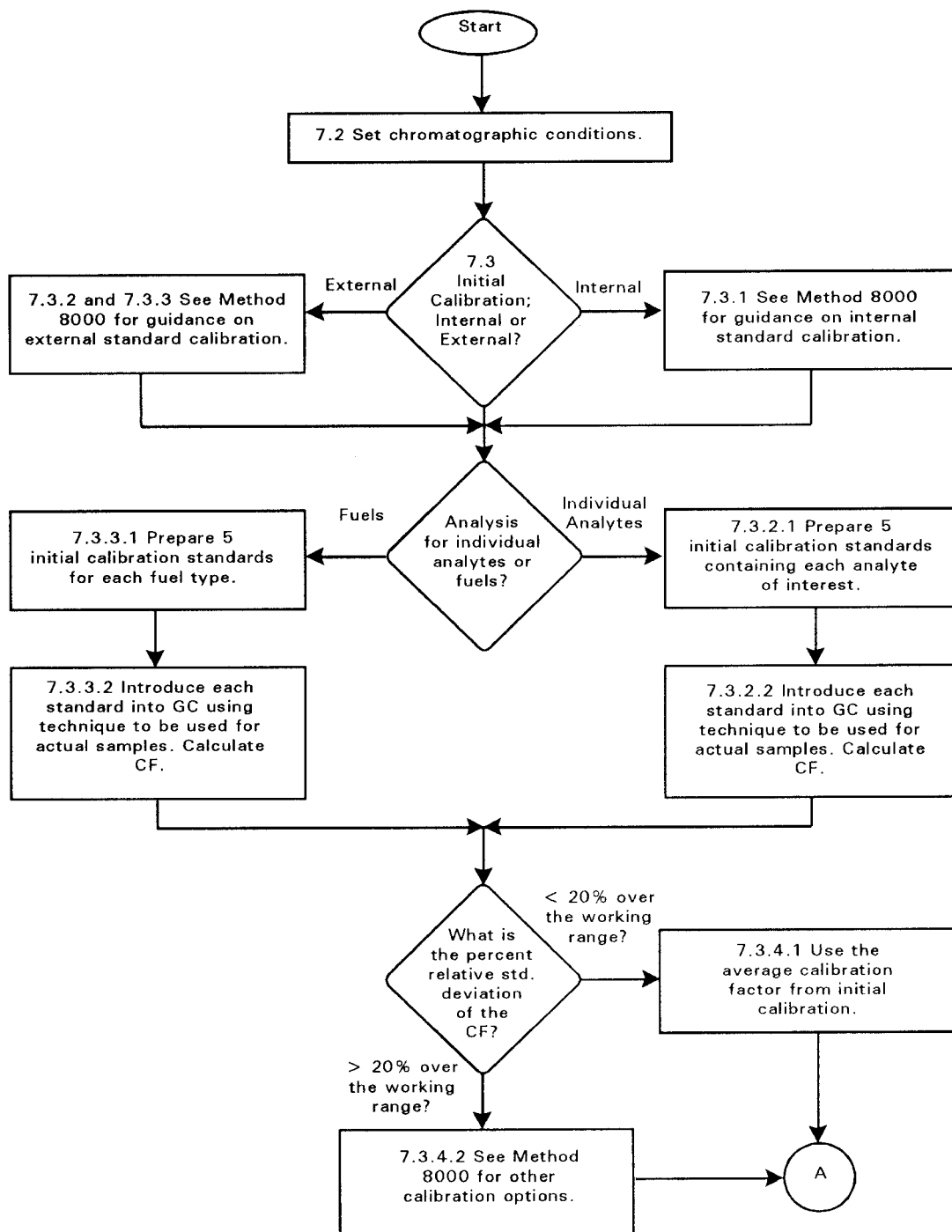
CHROMATOGRAM OF SEVERAL NONPURGEABLE VOLATILE COMPOUNDS IN SPIKED REAGENT WATER USING AZEOTROPIC MICRODISTILLATION (METHOD 5031)



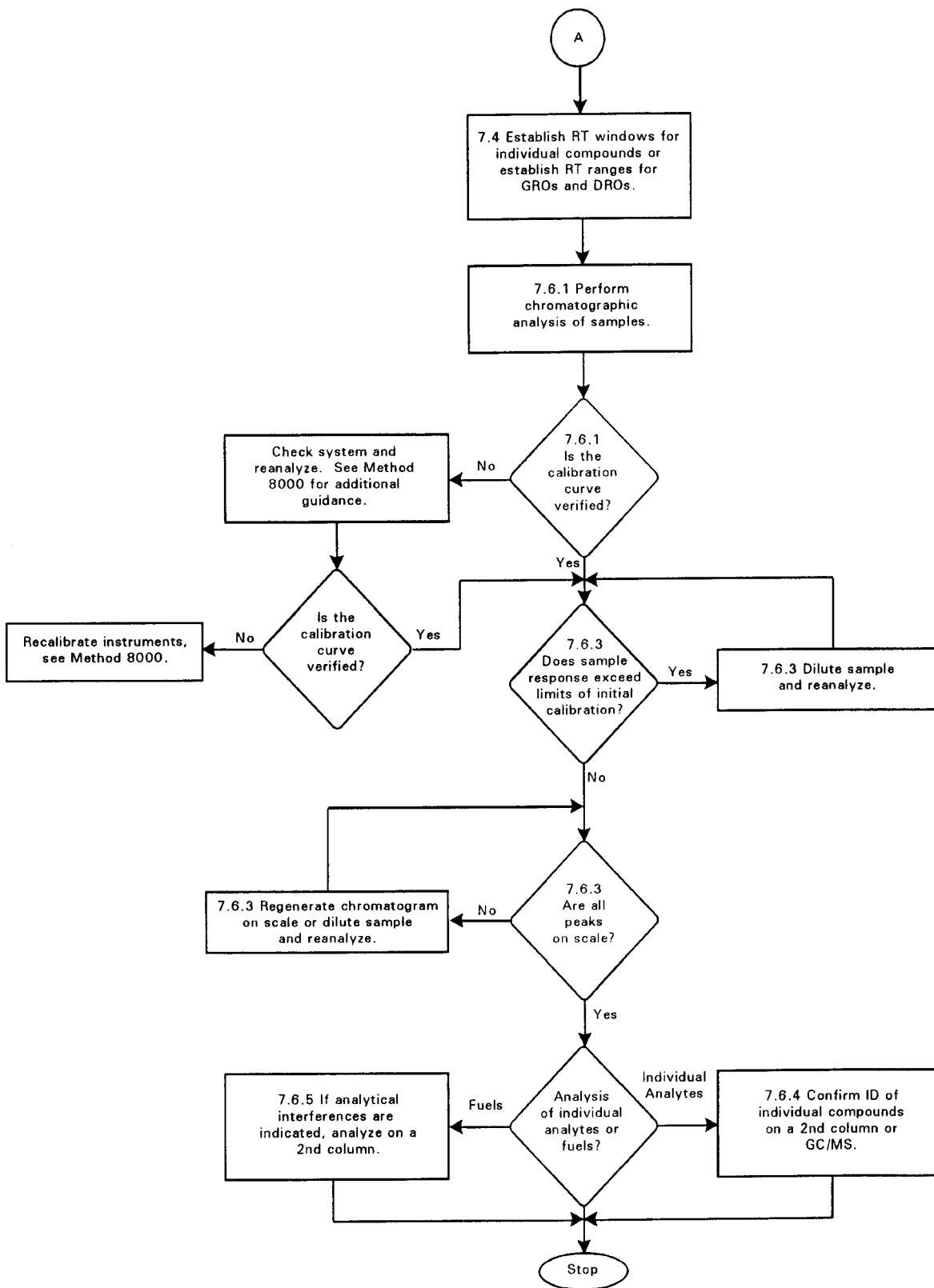
Mix 2: Analytes distilled at 0.25mg/L, Internal Stds. at 2.5 mg/L

Conditions: J&W DB-Wax column with 0.53 ID  
Temperature program: 30°C for 2 min.  
3°C/min. to 100°C and held for 0 min.  
25°C/min. to 200°C and held for 4 min.

METHOD 8015B  
NONHALOGENATED ORGANICS USING GC/FID



METHOD 8015B  
(continued)



## METHOD 5030B

### PURGE-AND-TRAP FOR AQUEOUS SAMPLES

#### 1.0 SCOPE AND APPLICATION

1.1 This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples and water miscible liquid samples. It also describes the analysis of high concentration soil and waste sample extracts prepared in Method 5035. The gas chromatographic determinative steps are found in Methods 8015 and 8021. The method is also applicable to GC/MS Method 8260.

1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax or a coated capillary column. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3 Method 5030, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 5030 and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Methods 8021 GC/PID) in series with Method 8015.

1.4 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system or by dilution of the sample prior to the purge-and-trap process.

1.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

#### 2.0 SUMMARY OF METHOD

2.1 Aqueous Samples: An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 High Concentration Extracts from Method 5035: An aliquot of the extract prepared in Method 5035 is combined with organic free reagent water in the purging chamber. It is then analyzed by purge-and-trap GC or GC/MS following the normal aqueous method.



### 3.0 INTERFERENCES

3.1 Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the entire system may be required.

3.4 The laboratory where volatiles analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. The analytical and sample storage areas should be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

### 4.0 APPARATUS AND MATERIALS

4.1 Microsyringes - 10- $\mu$ L, 25- $\mu$ L, 100- $\mu$ L, 250- $\mu$ L, 500- $\mu$ L, and 1,000- $\mu$ L. These syringes should be equipped with a 20-gauge (0.006 in ID) needle having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device. The needle length will depend upon the dimensions of the purging device employed.

4.2 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.3 Two 5-mL glass hypodermic syringes with Luer-Lok tip (other sizes are acceptable depending on sample volume used).

4.4 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

4.5 Vials - 2-mL, for GC autosampler.

## 4.6 Purge-and-trap device

The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

4.6.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be used, provided equivalent or improved performance is demonstrated.

4.6.2 The trap used to develop this method was 25 cm long with an inside diameter of 0.105 in. Starting from the inlet, the trap contains the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (see Figures 2 and 3). If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

4.6.3 The desorber must be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. The desorber design illustrated in Figures 2 and 3 meet these criteria.

4.6.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as shown in Figures 4 and 5.

### 4.6.5 Trap Packing Materials

4.6.5.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.6.5.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.6.5.3 Silica gel - 35/60 mesh, Davison, grade 15 or equivalent.

4.6.5.4 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

#### 4.6.5.5 Alternate Trap Materials

A number of hydrophobic carbon molecular sieve and graphitized carbon black materials have been developed. Various combinations of these materials have been shown to provide retention properties similar to the Tenax\Silica gel\Carbon trap. Alternate trap construction with such materials is allowed, provided that the adsorption and desorption characteristics obtained achieve equivalent or better method sensitivity and precision in comparison to the performance documented in the Determinative Method.

4.6.5.5.1 The following alternatives have been shown to be viable for most analytes of concern:

7.6-cm Carboxin™ B/1.3-cm Carboxin™ S-III  
VOCARB 3000 - 10.0-cm Carboxin™ B/6.0-cm Carboxin™ 1000/1.0-cm Carboxin™ 1001  
VOCARB 4000 - 8.5-cm Carboxin™ C/10.0-cm Carboxin™ B/6.0-cm Carboxin™ 1000/1.0-cm Carboxin™ 1001

These combinations require rapid heating to desorption temperatures of 245°C to 270°C (follow manufacturer's instructions). At these increased temperatures, catalytic and thermal decomposition of analytes has been reported. The VOCARB 4000 combination has also been demonstrated to catalytically break down 2-chloroethyl vinyl ether, and to partially decompose 2,2-dichloropropane. Bromoform and bromomethane have shown some thermal decomposition.

4.6.5.5.2 The amount of thermal decomposition products formed must be routinely tracked by daily monitoring of the formation of chloromethane and bromomethane. A daily check standard containing surrogates, internal standards, and 20 µg/L bromoform must be analyzed prior to the analysis of the daily check standard. If levels of chloromethane or bromomethane exceed 0.5 µg/L, then the trap may be too contaminated with salts or tightly bound contamination for analysis to continue. The trap must be replaced and the system recalibrated.

NOTE: Even newly constructed traps may have become contaminated prior to their first use from airborne vapors. These highly adsorptive materials must be kept tightly sealed in an area of minimum organic vapor contamination.

4.7 Heater or heated oil bath - capable of maintaining the purging chamber to within 1°C, over a temperature range from ambient to 100°C.

4.8 Capillary GC Columns - Any GC column that meets the performance specifications of the determinative method may be used. See the specific determinative method for recommended columns, conditions and retention times.

4.8.1 The wide-bore columns have the capacity to accept the standard gas flows from the trap during thermal desorption, and chromatography can begin with the onset of thermal desorption. Depending on the pumping capacity of the MS, an additional interface between the end of the column and the MS may be required. An open split interface, an all-glass jet

separator, or a cryogenic (Sec. 4.8.2) device are acceptable interfaces. The type of interface and its adjustments can have a significant impact on the method detection limits. Other interfaces can be used if the performance specifications described in this method can be achieved.

4.8.2 A system using a narrow-bore column will require lower gas flows of approximately 2 - 4 mL/minute. Because of these low desorption flows, early eluting analytes need to be refocussed to elute in a narrow band. This refocussing may be carried out by using a cryogenic interface. This type of interface usually uses liquid nitrogen to condense the desorbed sample components in a narrow band on an uncoated fused silica precolumn. When all components have been desorbed from the trap, the interface is rapidly heated under a stream of carrier gas to transfer the analytes to the analytical column. The end of the analytical column should be placed within a few mm of the MS ion source. A potential problem with this interface is blockage of the interface by ice caused by desorbing water from the trap. This condition will result in a major loss in sensitivity and chromatographic resolution. Low surrogate compound recoveries can be a sign that this is occurring.

## 5.0 REAGENTS

5.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.2 See the determinative method and Method 5000 for guidance on internal and surrogate standards.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to the introductory material to this chapter, Organic Analytes, Sec. 4.1. Samples should be stored in capped bottles, with minimum headspace, at 4°C or less in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling the sample should not exceed 5 - 6 mm. When a bubble is present, also observe the cap and septum to ensure that a proper seal was made at time of sampling. Is there any evidence of leakage? If the sample was improperly sealed, the sample should be discarded.

6.2 All samples should be analyzed within 14 days of collection. Samples not analyzed within this period must be noted and data are considered minimum values.

## 7.0 PROCEDURE

7.1 The purge-and-trap technique for aqueous samples is found in Sec. 7.2 and guidance for analysis of solvent extracts from the High Concentration Method in Method 5035 is found in Sec. 7.3. The gas chromatographic determinative steps are found in Methods 8015 and 8021. The method is also applicable to GC/MS Method 8260. For the analysis of gasoline, use Method 8021 with GC/PID for BTEX in series with Method 8015 with the GC/FID detector for hydrocarbons.

7.2 This section provides guidance on the analysis of aqueous samples and samples that are water miscible, by purge-and-trap analysis.

## 7.2.1 Initial calibration

Prior to using this introduction technique for any GC method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the specific determinative methods and Method 5000 give details on preparation of standards. The GC/MS methods require instrument tuning prior to proceeding with calibration.

7.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 4.6. Condition the Tenax trap overnight at 180°C (condition other traps at the manufacturers recommended temperature) in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily for 10 min while backflushing at 180°C with the column at 220°C.

7.2.1.2 Connect the purge-and-trap device to a gas chromatograph or gas chromatograph/mass spectrometer system.

7.2.1.3 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. Add 5.0 mL of organic-free reagent water to the purging device. The organic-free reagent water is added to the purging device using a 5-mL glass syringe (a 10-mL or 25-mL syringe may be used if preferred) fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of the 20-gauge needle. Next, using a 10- $\mu$ L or 25- $\mu$ L micro-syringe equipped with a long needle (Sec. 4.1), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards. Add the aliquot of calibration solution directly to the organic-free reagent water in the purging device by inserting the needle through the sample inlet. When discharging the contents of the micro-syringe, be sure that the end of the syringe needle is well beneath the surface of the organic-free reagent water. Similarly, add 10.0  $\mu$ L of the internal standard solution. Close the 2-way syringe valve at the sample inlet. (The calibration standard, internal standard and surrogate standard may be added directly to the organic free reagent water in the syringe prior to transferring the water to the purging device, see Sec. 7.2.4.7).

7.2.1.4 Follow the purge-and-trap analysis as outlined in Sec. 7.2.4.

7.2.1.5 Calculate response factors (RF) or calibration factors (CF) for each analyte of interest using the procedure described in Method 8000.

7.2.1.6 The average CF (external standards) or RF (internal standards) must be calculated for each compound. For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

7.2.1.6.1 Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.

7.2.1.6.2 Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

7.2.1.6.3 1,1,2,2-Tetrachloroethane and 1,1-dichloroethane: These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.1.7 The analytes in Method 8021 normally are not as strongly affected by small changes in purge flow or system contamination. When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross contamination and memory effects from a high concentration sample or even the standard are a common problem. Extra rinsing of the purge chamber after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bakeout of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

7.2.2 Calibration verification: Refer to Method 8000 for details on calibration verification.

7.2.2.1 To prepare a calibration standard, inject an appropriate volume of a primary dilution standard to an aliquot of organic free reagent water in a volumetric flask, a gas tight syringe, or to a purge device, and inject an appropriate amount of internal standard to the organic free reagent water. Be sure the same amount of internal standard is added to each standard and sample. The volume of organic free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL). The surrogate and internal standard solutions must be added with a syringe needle long enough to ensure addition below the surface of the water. Assemble the purge-and-trap device as outlined in 4.6. Follow the guidance for the purge-and-trap procedure in Sec. 7.2.4. Ongoing GC or GC/MS calibration criteria must be met as specified in Method 8000 before analyzing samples.

### 7.2.3 Sample screening

7.2.3.1 Screening of the sample prior to purge-and-trap analysis may provide guidance on whether sample dilution is necessary and may prevent contamination of the purge-and-trap system.

7.2.3.2 SW-846 contains two screening techniques that may be utilized: the automated headspace sampler (Method 5021) connected to a gas chromatograph equipped with a photoionization detector in series with an electrolytic conductivity detector; and extraction of the samples with hexadecane (Method 3820) and analysis of the extract on a gas chromatograph equipped with a flame ionization detector and/or electron capture detector. In addition, other appropriate screening techniques may be employed at the discretion of the analyst.

### 7.2.4 Sample introduction and purging

7.2.4.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.2.4.2 Assemble the purge-and-trap device. The operating conditions for the GC and GC/MS are given in Sec. 7.0 of the specific determinative method to be employed. Whole oven cooling may be needed for certain GC columns and/or certain GC/MS systems to achieve adequate resolution of the gases. Normally a 30 meter wide-bore column will require cooling the GC oven to 25°C or below for resolution of the gases.

7.2.4.3 GC or GC/MS calibration verification criteria must be met (Method 8000) before analyzing samples.

7.2.4.4 Adjust the purge gas flow rate (nitrogen or helium) to 25-40 mL/min (also see Table 1 for guidance on specific analyte groups), on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

7.2.4.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. Alternatively, carefully transfer the remaining sample into a 20-mL VOA vial. Seal the vial with zero headspace. The second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 10- or 25-mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hrs. Care must be taken to prevent air from leaking into the syringe.

7.2.4.6 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.2.4.6.1 Dilutions may be made in volumetric flasks (10-mL to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.2.4.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

7.2.4.6.3 Inject the proper aliquot of samples from the syringe prepared in Sec. 7.2.4.5 into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions.

7.2.4.6.4 Fill a 5-mL syringe with the diluted sample as in Sec. 7.2.4.5.

7.2.4.7 Add 10.0  $\mu\text{L}$  of surrogate spiking solution (found in each determinative method, Sec. 5.0) and, if applicable, 10.0  $\mu\text{L}$  of internal standard spiking solution through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. Matrix spiking solutions, if indicated, should be added (10.0  $\mu\text{L}$ ) to the sample at this time.

7.2.4.8 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

7.2.4.9 Close both valves and purge the sample for the time and at the temperature specified in Table 1. For GC/MS analysis using Method 8260, purge time is 11 minutes at ambient temperature.

## 7.2.5 Sample desorption

The procedures employed for sample desorption depend on the type of GC interface used. Procedures for non-cryogenic and cryogenic interfaces are described below. Analysts should also consult the instructions from the manufacturer of the purge-and-trap system and the supplier of the trap packing material.

7.2.5.1 Non-cryogenic interface - After the recommended 11-minute purge (see Table 1 for guidance on purge times for specific analyte groups), place the purge-and-trap system in the desorb mode and preheat the trap to 180°C (or other temperature recommended for the specific trap packing material) without a flow of carrier gas passing through the trap.

**NOTE:** Some purge-and-trap systems are capable of performing a moisture removal step (e.g., dry purge) which can eliminate excess moisture from the trap and gas lines by purging the trap just prior to the desorption step. However, the utility of a moisture removal step depends on the nature of the trap packing material. In general, when using a carbon-based, hydrophobic trap packing, this step may prevent moisture from entering the GC system and affecting chromatography, but may require that the trap be cooled to keep the temperature at or below 25°C. However, for packings that are less hydrophobic or hydrophilic (such as silica gel), a moisture removal step may actually create more significant problems, including loss of sensitivity, poor chromatography, and premature failure of the trap packing material, through the release of increasing amounts of water into the GC system during the course of an analytical shift. The problem may be evident as erratic responses for the early-eluting internal standards and surrogates over the course of the day. Optimum results may be achieved through the proper choices of: the moisture control device, the trap packing material, trap temperature during moisture removal, and carrier gas flow. The use of trap back pressure control may also be necessary. Consult instructions from both the manufacturer of the purge-and-trap system and the supplier of the trap packing material before employing a moisture removal step.

Start the flow of the carrier gas, begin the GC temperature program, and start GC data acquisition. The carrier gas flow rate will depend on the trap employed. A flow rate of 15 mL/min is used for the standard silica gel trap (Sec. 4.6.2), while 10 mL/min may



be adequate for other traps. Continue the carrier gas flow for about 4 min, or as recommended by the manufacturer. Desorption times as low as 1.5 min may be adequate for analytes in Method 8015.

7.2.5.2 Cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode, make sure the cryogenic interface is  $-150^{\circ}\text{C}$  or lower, and rapidly heat the trap to  $180^{\circ}\text{C}$  (temperature may vary depending on the trap material) while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Method 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to  $250^{\circ}\text{C}$ ; simultaneously begin the temperature program of the gas chromatograph and start the data acquisition.

## 7.2.6 Trap Reconditioning

7.2.6.1 After desorbing the sample, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at  $180^{\circ}\text{C}$  for Methods 8021 and 8260, and  $210^{\circ}\text{C}$  for Method 8015. Trap temperatures up to  $220^{\circ}\text{C}$  may be employed. However, the higher temperatures will shorten the useful life of the trap. (Trap temperatures may vary depending on the trap material). After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.2.6.2 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 mL flushes of organic free reagent water (or methanol followed by organic free reagent water) to avoid carryover of volatile organics into subsequent analyses.

## 7.2.7 Interpretation and calculation of data

7.2.7.1 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated response from a compound, this analysis must be followed by the analysis of organic free reagent water. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can meet the organic-free reagent water criteria specified in Chapter One.

7.2.7.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Method 8000 and the specific determinative method for details on calculating analyte response.

## 7.2.8 Analysis of water-miscible liquids

7.2.8.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with organic-free reagent water.

7.2.8.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample into a 100-mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a 5-mL gas-tight syringe.

7.2.8.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with organic-free reagent water by adding at least 20.0  $\mu\text{L}$ , but not more than 100.0  $\mu\text{L}$  of liquid sample. The sample is ready for addition of surrogate and, if applicable, internal and matrix spiking standards.

7.3 This section provides guidance on the analysis of solvent extracts from High Concentration Samples prepared by Method 5035.

7.3.1 The GC or GC/MS system should be set up as in Sec. 7.0 of the specific determinative method. This should be done prior to the addition of the solvent extract to organic-free reagent water.

7.3.2 Table 2 can be used to determine the volume of solvent extract to add to the 5 mL of organic-free reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 100.0  $\mu\text{L}$ . All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.3.3 Remove the plunger from a 5.0-mL Luer-lok type syringe equipped with a syringe valve and fill until overflowing with organic-free reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10.0  $\mu\text{L}$  of internal standard solution. Also add the volume of solvent extract determined in Sec. 7.3.2 and a volume of the same solvent used in Method 5035 to total 100.0  $\mu\text{L}$  (excluding methanol in standards).

7.3.4 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

7.3.5 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100.0  $\mu\text{L}$  of methanol to simulate the sample conditions.

#### 7.4 Sample analysis

The samples prepared by this method may be analyzed by Methods 8015, 8021, and 8260. Refer to these methods for appropriate analysis conditions. For the analysis of gasoline, use Method 8021 with GC/PID for BTEX in series with Method 8015 with the GC/FID detector for hydrocarbons.

### 8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 5000 for sample preparation QC procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be

processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling technique. Each analysis batch of 20 or less samples must contain: a reagent blank; either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis; and a laboratory control sample, unless the determinative method provides other guidance.

8.4 Surrogate standards should be added to all samples when specified in the appropriate determinative method

## 9.0 METHOD PERFORMANCE

Refer to the determinative methods for performance data.

## 10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
2. Bellar, T., "Measurement of Volatile Organic Compounds in Soils Using Modified Purge-and-Trap and Capillary Gas Chromatography/Mass Spectrometry", U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH, November, 1991.

TABLE 1  
PURGE-AND-TRAP OPERATING PARAMETERS

	Analysis Method	
	8015	8021/8260
Purge gas	N <sub>2</sub> or He	N <sub>2</sub> or He
Purge gas flow rate (mL/min)	20	40
Purge time (min)	15.0 ±0.1	11.0 ±0.1
Purge temperature (°C)	85 ±2	Ambient
Desorb temperature (°C)	180	180
Backflush inert gas flow (mL/min)	20-60	20-60 <sup>1</sup>
Desorb time (min)	1.5	4

<sup>1</sup> The desorption flow rate for Method 8021 with a wide bore capillary column will optimize at approximately 10 to 15 mL/minute.

TABLE 2

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF  
HIGH-CONCENTRATION SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract <sup>a</sup>
500-10,000 µg/kg	100 µL
1,000-20,000 µg/kg	50 µL
5,000-100,000 µg/kg	10 µL
25,000-500,000 µg/kg	100 µL of 1/50 dilution <sup>b</sup>

Calculate appropriate dilution factor for concentrations exceeding this table.

- <sup>a</sup> The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 µL added to the syringe.
- <sup>b</sup> Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

FIGURE 1  
EXAMPLE OF PURGING DEVICE

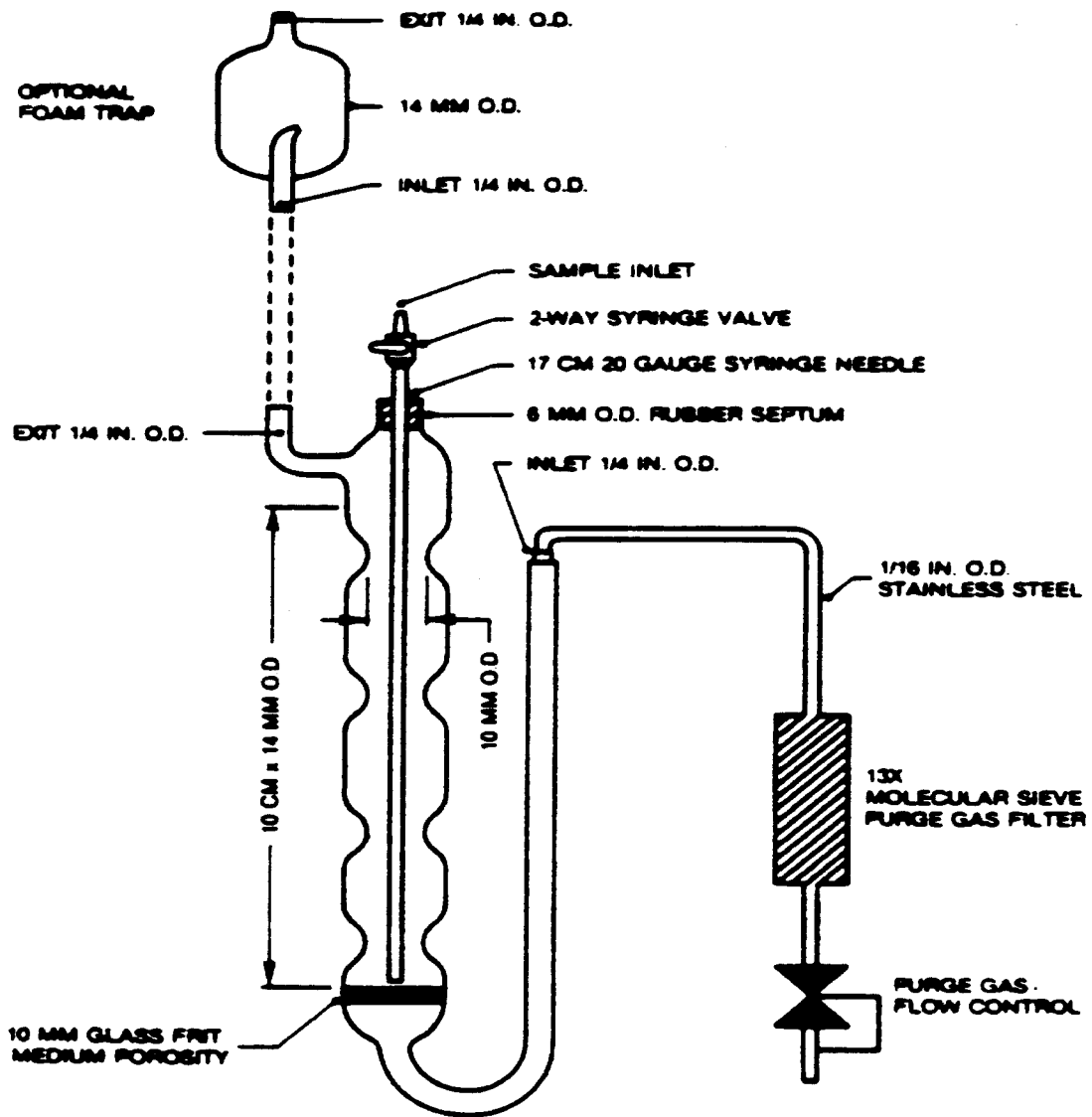


FIGURE 2  
 EXAMPLE OF TRAP PACKINGS AND CONSTRUCTION  
 TO INCLUDE DESORB CAPABILITY

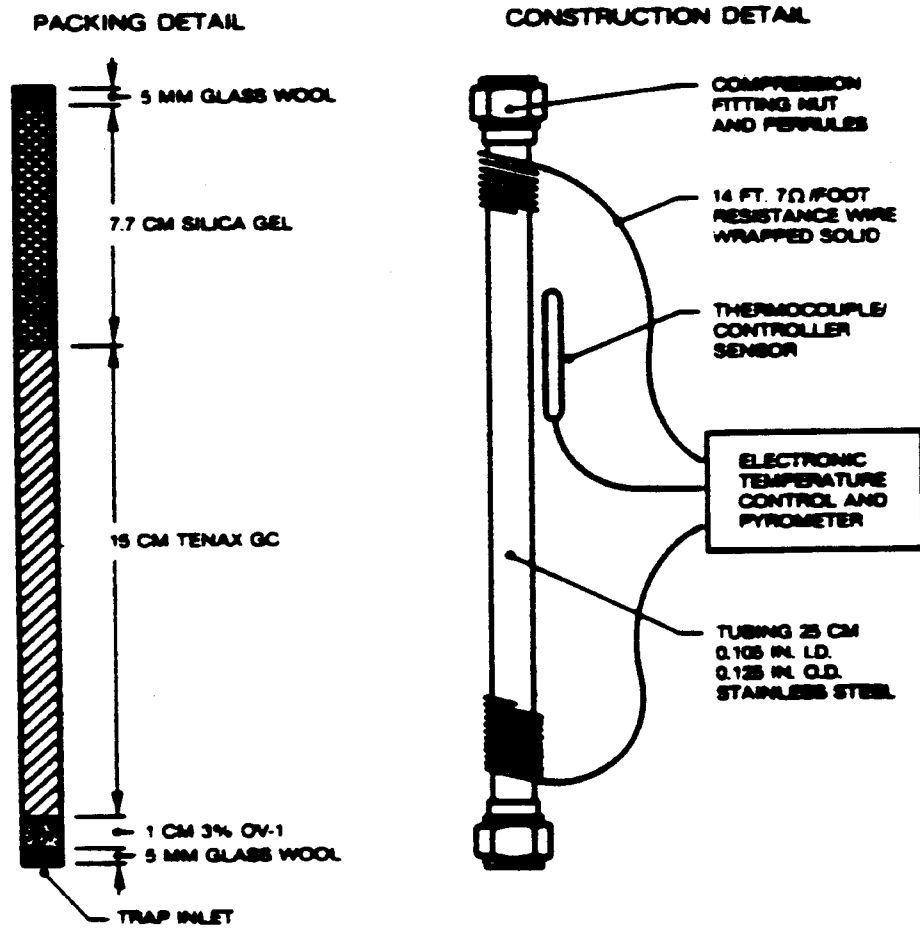


FIGURE 3  
SCHEMATIC OF TYPICAL PURGE AND TRAP DEVICE  
PURGE MODE

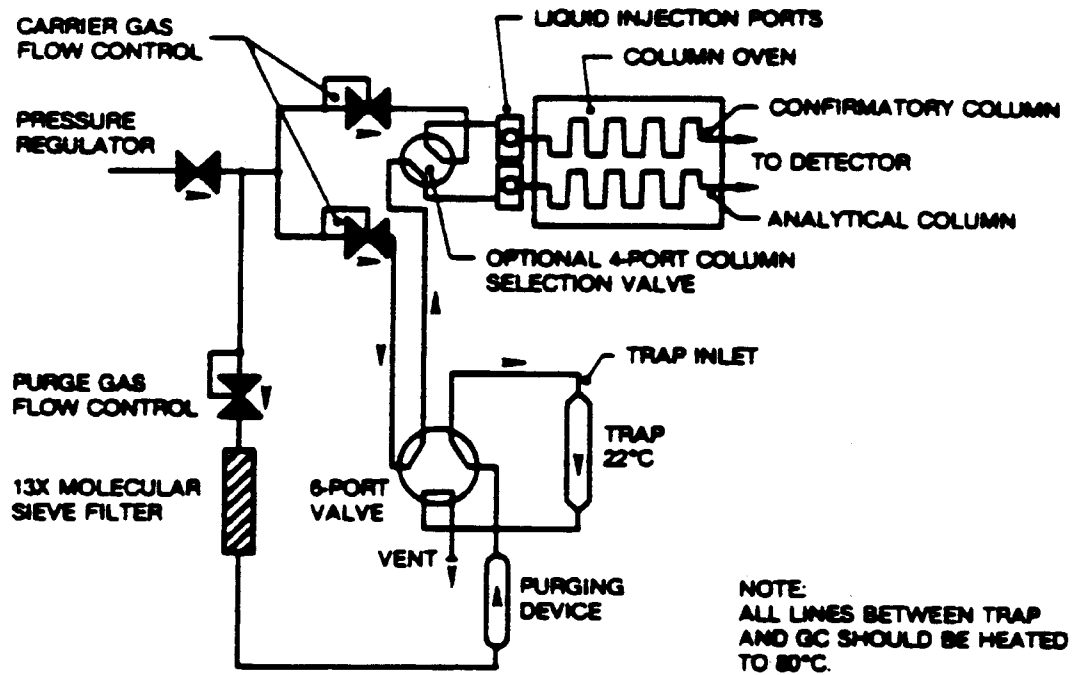
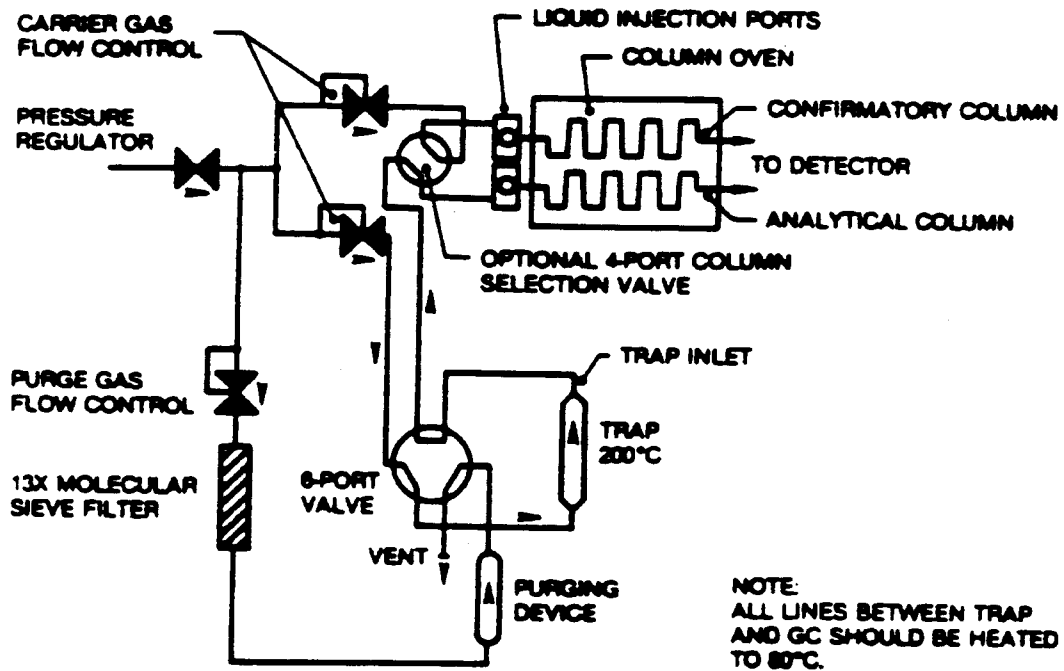
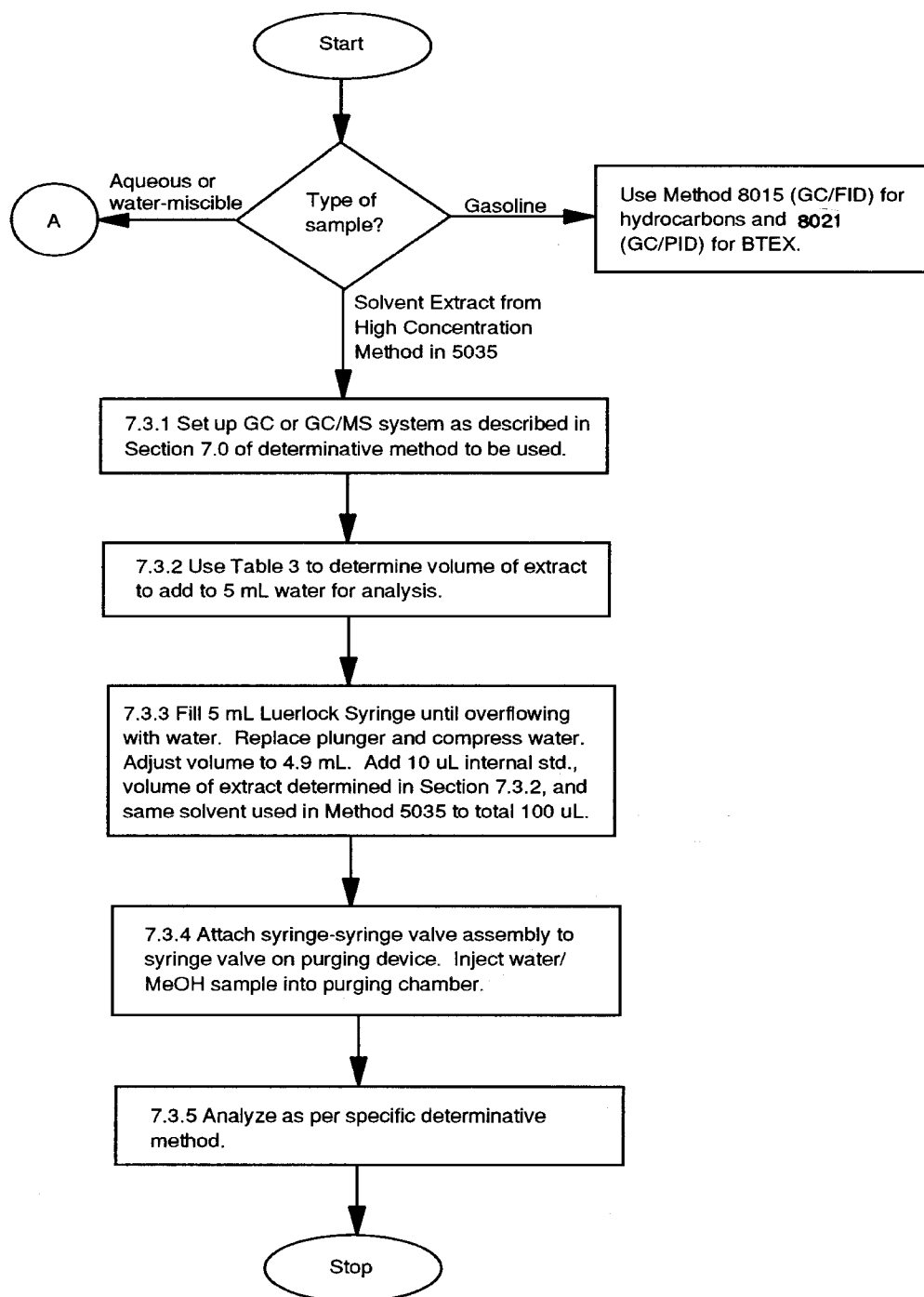




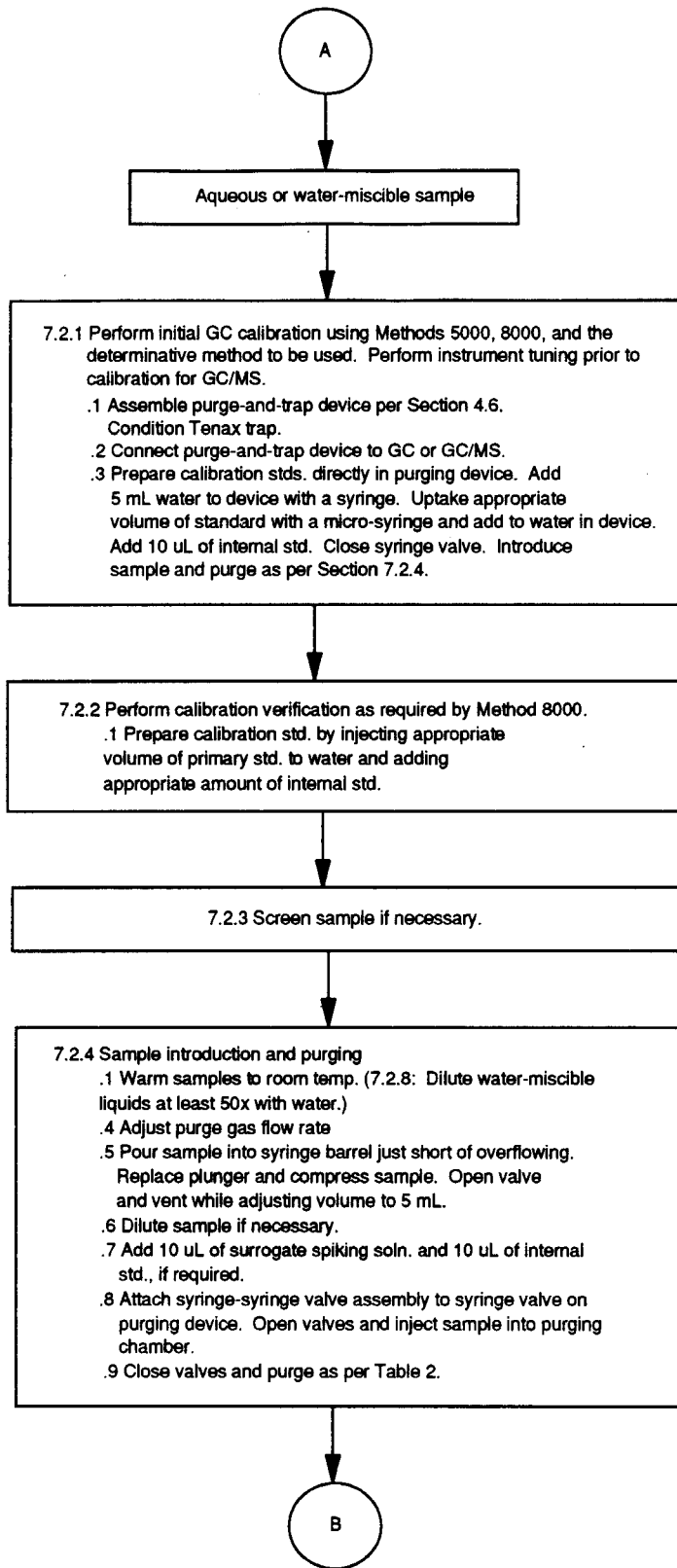
FIGURE 4  
SCHEMATIC OF TYPICAL PURGE AND TRAP DEVICE  
DESORB MODE



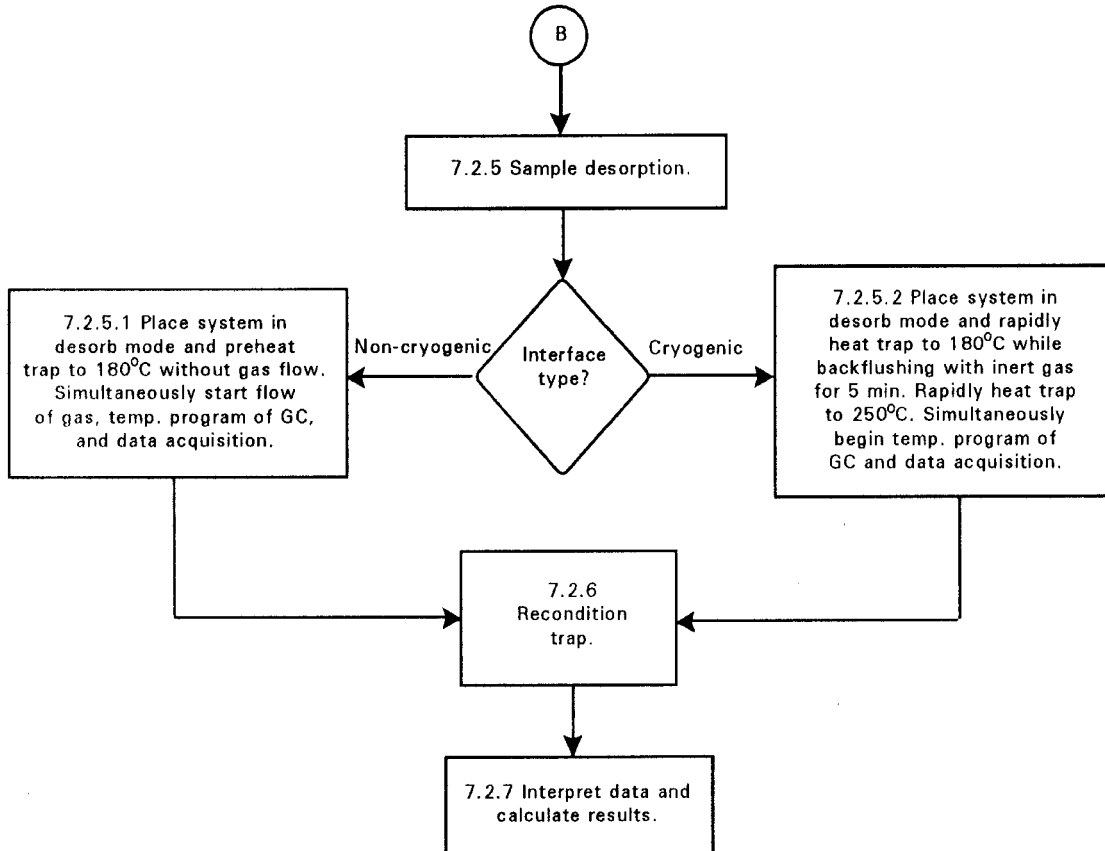
METHOD 5030B  
PURGE-AND-TRAP FOR AQUEOUS SAMPLES



METHOD 5030B  
continued



METHOD 5030B  
continued



## METHOD 8270C

SEMIVOLATILE ORGANIC COMPOUNDS  
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

## 1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

Compounds	CAS No <sup>a</sup>	<u>Appropriate Preparation Techniques<sup>b</sup></u>				
		3510	3520	3540/ 3541	3550	3580
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthene-d <sub>10</sub> (IS)		X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea	591-08-2	LR	ND	ND	ND	LR
Aldrin	309-00-2	X	X	X	X	X
2-Aminoanthraquinone	117-79-3	X	ND	ND	ND	X
Aminoazobenzene	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcarbazole	132-32-1	X	X	ND	ND	ND
Anilazine	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite	140-57-8	HS(43)	ND	ND	ND	X
Aroclor 1016	12674-11-2	X	X	X	X	X
Aroclor 1221	11104-28-2	X	X	X	X	X
Aroclor 1232	11141-16-5	X	X	X	X	X
Aroclor 1242	53469-21-9	X	X	X	X	X
Aroclor 1248	12672-29-6	X	X	X	X	X
Aroclor 1254	11097-69-1	X	X	X	X	X
Aroclor 1260	11096-82-5	X	X	X	X	X
Azinphos-methyl	86-50-0	HS(62)	ND	ND	ND	X
Barban	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP
Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X
Benzo(k)fluoranthene	207-08-9	X	X	X	X	X
Benzo(g,h,i)perylene	191-24-2	X	X	X	X	X
Benzo(a)pyrene	50-32-8	X	X	X	X	X

Appropriate Preparation Techniques<sup>b</sup>

Compounds	CAS No <sup>a</sup>	Appropriate Preparation Techniques <sup>b</sup>				
		3510	3520	3540/ 3541	3550	3580
p-Benzoquinone	106-51-4	OE	ND	ND	ND	X
Benzyl alcohol	100-51-6	X	X	ND	X	X
α-BHC	319-84-6	X	X	X	X	X
β-BHC	319-85-7	X	X	X	X	X
δ-BHC	319-86-8	X	X	X	X	X
γ-BHC (Lindane)	58-89-9	X	X	X	X	X
Bis(2-chloroethoxy)methane	111-91-1	X	X	X	X	X
Bis(2-chloroethyl) ether	111-44-4	X	X	X	X	X
Bis(2-chloroisopropyl) ether	108-60-1	X	X	X	X	X
Bis(2-ethylhexyl) phthalate	117-81-7	X	X	X	X	X
4-Bromophenyl phenyl ether	101-55-3	X	X	X	X	X
Bromoxynil	1689-84-5	X	ND	ND	ND	X
Butyl benzyl phthalate	85-68-7	X	X	X	X	X
Captafol	2425-06-1	HS(55)	ND	ND	ND	X
Captan	133-06-2	HS(40)	ND	ND	ND	X
Carbaryl	63-25-2	X	ND	ND	ND	X
Carbofuran	1563-66-2	X	ND	ND	ND	X
Carbophenothion	786-19-6	X	ND	ND	ND	X
Chlordane (NOS)	57-74-9	X	X	X	X	X
Chlorfenvinphos	470-90-6	X	ND	ND	ND	X
4-Chloroaniline	106-47-8	X	ND	ND	ND	X
Chlorobenzilate	510-15-6	X	ND	ND	ND	X
5-Chloro-2-methylaniline	95-79-4	X	ND	ND	ND	X
4-Chloro-3-methylphenol	59-50-7	X	X	X	X	X
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	X	ND	ND	ND	X
1-Chloronaphthalene	90-13-1	X	X	X	X	X
2-Chloronaphthalene	91-58-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X
4-Chloro-1,2-phenylenediamine	95-83-0	X	X	ND	ND	ND
4-Chloro-1,3-phenylenediamine	5131-60-2	X	X	ND	ND	ND
4-Chlorophenyl phenyl ether	7005-72-3	X	X	X	X	X
Chrysene	218-01-9	X	X	X	X	X
Chrysene-d <sub>12</sub> (IS)		X	X	X	X	X
Coumaphos	56-72-4	X	ND	ND	ND	X
p-Cresidine	120-71-8	X	ND	ND	ND	X
Crotoxypfos	7700-17-6	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitro-phenol	131-89-5	X	ND	ND	ND	LR
4,4'-DDD	72-54-8	X	X	X	X	X
4,4'-DDE	72-55-9	X	X	X	X	X
4,4'-DDT	50-29-3	X	X	X	X	X
Demeton-O	298-03-3	HS(68)	ND	ND	ND	X
Demeton-S	126-75-0	X	ND	ND	ND	X
Diallate (cis or trans)	2303-16-4	X	ND	ND	ND	X

Appropriate Preparation Techniques<sup>b</sup>

Compounds	CAS No <sup>a</sup>	Appropriate Preparation Techniques <sup>b</sup>				
		3510	3520	3540/ 3541	3550	3580
2,4-Diaminotoluene	95-80-7	DC,0E(42)	ND	ND	ND	X
Dibenz(a,i)acridine	224-42-0	X	ND	ND	ND	X
Dibenz(a,h)anthracene	53-70-3	X	X	X	X	X
Dibenzofuran	132-64-9	X	X	ND	X	X
Dibenzo(a,e)pyrene	192-65-4	ND	ND	ND	ND	X
1,2-Dibromo-3-chloropropane	96-12-8	X	X	ND	ND	ND
Di-n-butyl phthalate	84-74-2	X	X	X	X	X
Dichlone	117-80-6	OE	ND	ND	ND	X
1,2-Dichlorobenzene	95-50-1	X	X	X	X	X
1,3-Dichlorobenzene	541-73-1	X	X	X	X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X	X
1,4-Dichlorobenzene-d <sub>4</sub> (IS)		X	X	X	X	X
3,3'-Dichlorobenzidine	91-94-1	X	X	X	X	X
2,4-Dichlorophenol	120-83-2	X	X	X	X	X
2,6-Dichlorophenol	87-65-0	X	ND	ND	ND	X
Dichlorovos	62-73-7	X	ND	ND	ND	X
Dicrotophos	141-66-2	X	ND	ND	ND	X
Dieldrin	60-57-1	X	X	X	X	X
Diethyl phthalate	84-66-2	X	X	X	X	X
Diethylstilbestrol	56-53-1	AW,0S(67)	ND	ND	ND	X
Diethyl sulfate	64-67-5	LR	ND	ND	ND	LR
Dihydrosaffrole	56312-13-1	ND	ND	ND	ND	ND
Dimethoate	60-51-5	HE,HS(31)	ND	ND	ND	X
3,3'-Dimethoxybenzidine	119-90-4	X	ND	ND	ND	LR
Dimethylaminoazobenzene	60-11-7	X	ND	ND	ND	X
7,12-Dimethylbenz(a)-anthracene	57-97-6	CP(45)	ND	ND	ND	CP
3,3'-Dimethylbenzidine	119-93-7	X	ND	ND	ND	X
α,α-Dimethylphenethylamine	122-09-8	ND	ND	ND	ND	X
2,4-Dimethylphenol	105-67-9	X	X	X	X	X
Dimethyl phthalate	131-11-3	X	X	X	X	X
1,2-Dinitrobenzene	528-29-0	X	ND	ND	ND	X
1,3-Dinitrobenzene	99-65-0	X	ND	ND	ND	X
1,4-Dinitrobenzene	100-25-4	HE(14)	ND	ND	ND	X
4,6-Dinitro-2-methylphenol	534-52-1	X	X	X	X	X
2,4-Dinitrophenol	51-28-5	X	X	X	X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X	X	X	X	X
Dinocap	39300-45-3	CP,HS(28)	ND	ND	ND	CP
Dinoseb	88-85-7	X	ND	ND	ND	X
Dioxathion	78-34-2	ND	ND	ND	ND	ND
Diphenylamine	122-39-4	X	X	X	X	X
5,5-Diphenylhydantoin	57-41-0	X	ND	ND	ND	X
1,2-Diphenylhydrazine	122-66-7	X	X	X	X	X

Appropriate Preparation Techniques<sup>b</sup>

Compounds	CAS No <sup>a</sup>	Appropriate Preparation Techniques <sup>b</sup>				
		3510	3520	3540/ 3541	3550	3580
Di-n-octyl phthalate	117-84-0	X	X	X	X	X
Disulfoton	298-04-4	X	ND	ND	ND	X
Endosulfan I	959-98-8	X	X	X	X	X
Endosulfan II	33213-65-9	X	X	X	X	X
Endosulfan sulfate	1031-07-8	X	X	X	X	X
Endrin	72-20-8	X	X	X	X	X
Endrin aldehyde	7421-93-4	X	X	X	X	X
Endrin ketone	53494-70-5	X	X	ND	X	X
EPN	2104-64-5	X	ND	ND	ND	X
Ethion	563-12-2	X	ND	ND	ND	X
Ethyl carbamate	51-79-6	DC(28)	ND	ND	ND	X
Ethyl methanesulfonate	62-50-0	X	ND	ND	ND	X
Famphur	52-85-7	X	ND	ND	ND	X
Fensulfothion	115-90-2	X	ND	ND	ND	X
Fenthion	55-38-9	X	ND	ND	ND	X
Fluchloralin	33245-39-5	X	ND	ND	ND	X
Fluoranthene	206-44-0	X	X	X	X	X
Fluorene	86-73-7	X	X	X	X	X
2-Fluorobiphenyl (surr)	321-60-8	X	X	X	X	X
2-Fluorophenol (surr)	367-12-4	X	X	X	X	X
Heptachlor	76-44-8	X	X	X	X	X
Heptachlor epoxide	1024-57-3	X	X	X	X	X
Hexachlorobenzene	118-74-1	X	X	X	X	X
Hexachlorobutadiene	87-68-3	X	X	X	X	X
Hexachlorocyclopentadiene	77-47-4	X	X	X	X	X
Hexachloroethane	67-72-1	X	X	X	X	X
Hexachlorophene	70-30-4	AW,CP(62)	ND	ND	ND	CP
Hexachloropropene	1888-71-7	X	ND	ND	ND	X
Hexamethylphosphoramide	680-31-9	X	ND	ND	ND	X
Hydroquinone	123-31-9	ND	ND	ND	ND	X
Indeno(1,2,3-cd)pyrene	193-39-5	X	X	X	X	X
Isodrin	465-73-6	X	ND	ND	ND	X
Isophorone	78-59-1	X	X	X	X	X
Isosafrole	120-58-1	DC(46)	ND	ND	ND	X
Kepone	143-50-0	X	ND	ND	ND	X
Leptophos	21609-90-5	X	ND	ND	ND	X
Malathion	121-75-5	HS(5)	ND	ND	ND	X
Maleic anhydride	108-31-6	HE	ND	ND	ND	X
Mestranol	72-33-3	X	ND	ND	ND	X
Methapyrilene	91-80-5	X	ND	ND	ND	X
Methoxychlor	72-43-5	X	ND	ND	ND	X
3-Methylcholanthrene	56-49-5	X	ND	ND	ND	X
4,4'-Methylenebis (2-chloroaniline)	101-14-4	OE,OS(0)	ND	ND	ND	LR



Appropriate Preparation Techniques<sup>b</sup>

Compounds	CAS No <sup>a</sup>	Appropriate Preparation Techniques <sup>b</sup>				
		3510	3520	3540/ 3541	3550	3580
4,4'-Methylenebis (N,N-dimethylaniline)	101-61-1	X	X	ND	ND	ND
Methyl methanesulfonate	66-27-3	X	ND	ND	ND	X
2-Methylnaphthalene	91-57-6	X	X	ND	X	X
Methyl parathion	298-00-0	X	ND	ND	ND	X
2-Methylphenol	95-48-7	X	ND	ND	ND	X
3-Methylphenol	108-39-4	X	ND	ND	ND	X
4-Methylphenol	106-44-5	X	ND	ND	ND	X
Mevinphos	7786-34-7	X	ND	ND	ND	X
Mexacarbate	315-18-4	HE,HS(68)	ND	ND	ND	X
Mirex	2385-85-5	X	ND	ND	ND	X
Monocrotophos	6923-22-4	HE	ND	ND	ND	X
Naled	300-76-5	X	ND	ND	ND	X
Naphthalene	91-20-3	X	X	X	X	X
Naphthalene-d <sub>8</sub> (IS)		X	X	X	X	X
1,4-Naphthoquinone	130-15-4	X	ND	ND	ND	X
1-Naphthylamine	134-32-7	OS(44)	ND	ND	ND	X
2-Naphthylamine	91-59-8	X	ND	ND	ND	X
Nicotine	54-11-5	DE(67)	ND	ND	ND	X
5-Nitroacenaphthene	602-87-9	X	ND	ND	ND	X
2-Nitroaniline	88-74-4	X	X	ND	X	X
3-Nitroaniline	99-09-2	X	X	ND	X	X
4-Nitroaniline	100-01-6	X	X	ND	X	X
5-Nitro-o-anisidine	99-59-2	X	ND	ND	ND	X
Nitrobenzene	98-95-3	X	X	X	X	X
Nitrobenzene-d <sub>5</sub> (surr)		X	X	X	X	X
4-Nitrobiphenyl	92-93-3	X	ND	ND	ND	X
Nitrofen	1836-75-5	X	ND	ND	ND	X
2-Nitrophenol	88-75-5	X	X	X	X	X
4-Nitrophenol	100-02-7	X	X	X	X	X
5-Nitro-o-toluidine	99-55-8	X	X	ND	ND	X
Nitroquinoline-1-oxide	56-57-5	X	ND	ND	ND	X
N-Nitrosodi-n-butylamine	924-16-3	X	ND	ND	ND	X
N-Nitrosodiethylamine	55-18-5	X	ND	ND	ND	X
N-Nitrosodimethylamine	62-75-9	X	X	X	X	X
N-Nitrosomethylethylamine	10595-95-6	X	ND	ND	ND	X
N-Nitrosodiphenylamine	86-30-6	X	X	X	X	X
N-Nitrosodi-n-propylamine	621-64-7	X	X	X	X	X
N-Nitrosomorpholine	59-89-2	ND	ND	ND	ND	X
N-Nitrosopiperidine	100-75-4	X	ND	ND	ND	X
N-Nitrosopyrrolidine	930-55-2	X	ND	ND	ND	X
Octamethyl pyrophosphoramidate	152-16-9	LR	ND	ND	ND	LR
4,4'-Oxydianiline	101-80-4	X	ND	ND	ND	X

Appropriate Preparation Techniques<sup>b</sup>

Compounds	CAS No <sup>a</sup>	Appropriate Preparation Techniques <sup>b</sup>				
		3510	3520	3540/ 3541	3550	3580
Parathion	56-38-2	X	X	ND	ND	X
Pentachlorobenzene	608-93-5	X	ND	ND	ND	X
Pentachloronitrobenzene	82-68-8	X	ND	ND	ND	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Perylene-d <sub>12</sub> (IS)		X	X	X	X	X
Phenacetin	62-44-2	X	ND	ND	ND	X
Phenanthrene	85-01-8	X	X	X	X	X
Phenanthrene-d <sub>10</sub> (IS)		X	X	X	X	X
Phenobarbital	50-06-6	X	ND	ND	ND	X
Phenol	108-95-2	DC(28)	X	X	X	X
Phenol-d <sub>6</sub> (surr)		DC(28)	X	X	X	X
1,4-Phenylenediamine	106-50-3	X	ND	ND	ND	X
Phorate	298-02-2	X	ND	ND	ND	X
Phosalone	2310-17-0	HS(65)	ND	ND	ND	X
Phosmet	732-11-6	HS(15)	ND	ND	ND	X
Phosphamidon	13171-21-6	HE(63)	ND	ND	ND	X
Phthalic anhydride	85-44-9	CP,HE(1)	ND	ND	ND	CP
2-Picoline (2-Methylpyridine)	109-06-8	X	X	ND	ND	ND
Piperonyl sulfoxide	120-62-7	X	ND	ND	ND	X
Pronamide	23950-58-5	X	ND	ND	ND	X
Propylthiouracil	51-52-5	LR	ND	ND	ND	LR
Pyrene	129-00-0	X	X	X	X	X
Pyridine	110-86-1	ND	ND	ND	ND	ND
Resorcinol	108-46-3	DC,OE(10)	ND	ND	ND	X
Safrole	94-59-7	X	ND	ND	ND	X
Strychnine	57-24-9	AW,OS(55)	ND	ND	ND	X
Sulfallate	95-06-7	X	ND	ND	ND	X
Terbufos	13071-79-9	X	ND	ND	ND	X
Terphenyl-d <sub>14</sub> (surr)	1718-51-0	X	X	ND	X	X
1,2,4,5-Tetrachlorobenzene	95-94-3	X	ND	ND	ND	X
2,3,4,6-Tetrachlorophenol	58-90-2	X	ND	ND	ND	X
Tetrachlorvinphos	961-11-5	X	ND	ND	ND	X
Tetraethyl dithiopyrophosphate	3689-24-5	X	X	ND	ND	ND
Tetraethyl pyrophosphate	107-49-3	X	ND	ND	ND	X
Thionazine	297-97-2	X	ND	ND	ND	X
Thiophenol (Benzenethiol)	108-98-5	X	ND	ND	ND	X
Toluene diisocyanate	584-84-9	HE(6)	ND	ND	ND	X
o-Toluidine	95-53-4	X	ND	ND	ND	X
Toxaphene	8001-35-2	X	X	X	X	X
2,4,6-Tribromophenol (surr)	118-79-6	X	X	X	X	X
1,2,4-Trichlorobenzene	120-82-1	X	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	ND	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X
Trifluralin	1582-09-8	X	ND	ND	ND	X

Compounds	CAS No <sup>a</sup>	Appropriate Preparation Techniques <sup>b</sup>				
		3510	3520	3540/ 3541	3550	3580
2,4,5-Trimethylaniline	137-17-7	X	ND	ND	ND	X
Trimethyl phosphate	512-56-1	HE(60)	ND	ND	ND	X
1,3,5-Trinitrobenzene	99-35-4	X	ND	ND	ND	X
Tris(2,3-dibromopropyl) phosphate	126-72-7	X	ND	ND	ND	LR
Tri-p-tolyl phosphate	78-32-0	X	ND	ND	ND	X
O,O,O-Triethyl phosphorothioate	126-68-1	X	ND	ND	ND	X

<sup>a</sup> Chemical Abstract Service Registry Number

<sup>b</sup> See Sec. 1.2 for other acceptable preparation methods.

### KEY TO ANALYTE LIST

- IS = This compound may be used as an internal standard.  
surr = This compound may be used as a surrogate.  
AW = Adsorption to walls of glassware during extraction and storage.  
CP = Nonreproducible chromatographic performance.  
DC = Unfavorable distribution coefficient (number in parenthesis is percent recovery).  
HE = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).  
HS = Hydrolysis during storage (number in parenthesis is percent stability).  
LR = Low response.  
ND = Not determined.  
OE = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).  
OS = Oxidation during storage (number in parenthesis is percent stability).  
X = Greater than 70 percent recovery by this technique.

1.2 In addition to the sample preparation methods listed in the above analyte list, Method 3542 describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 11 contains surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid extraction of solids for PAHs (see Tables 13, 14, and 15 for performance data).

1.3 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated.

In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

1.4 The following compounds may require special treatment when being determined by this method:

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices,  $\alpha$ -BHC,  $\gamma$ -BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.4.6 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4.7 Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4.8 Toluene diisocyanate rapidly hydrolyses in water (half-life of less than 30 min.). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.9 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660  $\mu\text{g}/\text{kg}$  (wet weight) for soil/sediment samples, 1-200  $\text{mg}/\text{kg}$  for wastes (dependent on matrix and method of preparation), and 10  $\mu\text{g}/\text{L}$  for ground water samples (see Table 2). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

## 3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

## 4.0 APPARATUS AND MATERIALS

### 4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm ID) 1  $\mu$ m film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

### 4.1.3 Mass spectrometer

4.1.3.1 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 3 when 1  $\mu$ L of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.1.6 Guard column (optional) - (J&W Deactivated Fused Silica, 0.25 mm ID x 6 m, or equivalent) between the injection port and the analytical column joined with column joiners (Hewlett-Packard Catalog No. 5062-3556, or equivalent).

4.2 Syringe - 10- $\mu$ L.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Balance - Analytical, capable of weighing 0.0001 g.

4.5 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

## 5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Stock standard solutions (1000 mg/L) - Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.2 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at  $-10^{\circ}\text{C}$  or less or as recommended by the standard manufacturer. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

5.3.4 It is recommended that nitrosamine compounds be placed together in a separate calibration mix and not combined with other calibration mixes. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.3.5 Mixes with hydrochloride salts may contain hydrochloric acid, which can cause analytical difficulties. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.4 Internal standard solutions - The internal standards recommended are 1,4-dichlorobenzene- $\text{d}_4$ , naphthalene- $\text{d}_8$ , acenaphthene- $\text{d}_{10}$ , phenanthrene- $\text{d}_{10}$ , chrysene- $\text{d}_{12}$ , and perylene- $\text{d}_{12}$  (see Table 5). Other compounds may be used as internal standards as long as the requirements given in Sec. 7.3.2 are met.

5.4.1 Dissolve 0.200 g of each compound with a small volume of carbon disulfide. Transfer to a 50 mL volumetric flask and dilute to volume with methylene chloride so that the final solvent is approximately 20% carbon disulfide. Most of the compounds are also soluble in small volumes of methanol, acetone, or toluene, except for perylene- $\text{d}_{12}$ . The resulting solution will contain each standard at a concentration of 4,000 ng/ $\mu\text{L}$ . Each 1 mL sample extract undergoing analysis should be spiked with 10  $\mu\text{L}$  of the internal standard solution, resulting in a concentration of 40 ng/ $\mu\text{L}$  of each internal standard. Store at  $-10^{\circ}\text{C}$  or less when not in use. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.4.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute internal standard solution may be required. Area counts of the internal standard peaks should be between 50-200% of the area of the target analytes in the mid-point calibration analysis.

5.5 GC/MS tuning standard - A methylene chloride solution containing 50 ng/ $\mu\text{L}$  of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/ $\mu\text{L}$  each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at  $-10^{\circ}\text{C}$  or less when not in use. If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute tuning solution may be necessary. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five different concentrations. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method.

5.6.1 It is the intent of EPA that all target analytes for a particular analysis be included in the calibration standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.6.2 Each 1-mL aliquot of calibration standard should be spiked with 10  $\mu$ L of the internal standard solution prior to analysis. All standards should be stored at  $-10^{\circ}\text{C}$  or less, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared weekly and stored at  $4^{\circ}\text{C}$ . When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.7 Surrogate standards - The recommended surrogates are phenol- $\text{d}_6$ , 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene- $\text{d}_5$ , 2-fluorobiphenyl, and p-terphenyl- $\text{d}_{14}$ . See Method 3500 for instructions on preparing the surrogate solutions.

5.7.1 Surrogate Standard Check: Determine what the appropriate concentration should be for the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

NOTE: Method 3561 (SFE Extraction of PAHs) recommends the use of bromobenzene and p-quaterphenyl to better cover the range of PAHs listed in the method.

5.7.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute surrogate solution may be necessary.

5.8 Matrix spike and laboratory control standards - See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS).

5.8.1 Matrix Spike Check: Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery. It is recommended that this check be done whenever a new matrix spiking solution is prepared.

5.8.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute matrix and LCS spiking solution may be necessary.

5.8.3 Some projects may require the spiking of the specific compounds of interest, since the spiking compounds listed in Method 3500 would not be representative of the compounds of interest required for the project. When this occurs, the matrix and LCS spiking



standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - All solvents should be pesticide quality or equivalent.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Store the sample extracts at  $-10^{\circ}\text{C}$ , protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

## 7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis.

<u>Matrix</u>	<u>Methods</u>
Air	3542
Water	3510, 3520, 3535
Soil/sediment	3540, 3541, 3545, 3550, 3560, 3561
Waste	3540, 3541, 3545, 3550, 3560, 3561, 3580

7.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10- $\mu\text{L}$  syringe may be appropriate. The detection limit is very high (approximately 10,000  $\mu\text{g/L}$ ). Therefore, it is only permitted where concentrations in excess of 10,000  $\mu\text{g/L}$  are expected.

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

<u>Analytes of interest</u>	<u>Methods</u>
Aniline & aniline derivatives	3620
Phenols	3630, 3640, 8041 <sup>a</sup>
Phthalate esters	3610, 3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides & PCBs	3610, 3620, 3630, 3660, 3665
Nitroaromatics and cyclic ketones	3620, 3640
Polynuclear aromatic hydrocarbons	3611, 3630, 3640
Haloethers	3620, 3640
Chlorinated hydrocarbons	3620, 3640
Organophosphorus pesticides	3620

<u>Analytes of interest</u>	<u>Methods</u>
Petroleum waste	3611, 3650
All base, neutral, and acid priority pollutants	3640

<sup>a</sup> Method 8041 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered on GC/FID.

### 7.3 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

Mass range:	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	40°C, hold for 4 minutes
Temperature program:	40-270°C at 10°C/min
Final temperature:	270°C, hold until benzo[g,h,i]perylene elutes
Injector temperature:	250-300°C
Transfer line temperature:	250-300°C
Source temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Injection volume:	1-2 µL
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

7.3.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach has been shown to be useful: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

7.3.1.2 Use the DFTPP mass intensity criteria in Table 3 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions), provided that method performance is not adversely affected.

**NOTE:** All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

7.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD

should not exceed 20%. (See Sec. 8.0 of Method 8081 for the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

7.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column. The use of a guard column (Sec. 4.1.6) between the injection port and the analytical column may help prolong analytical column performance.

7.3.2 The internal standards selected in Sec. 5.4 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene- $d_4$ , use 152 m/z for quantitation).

7.3.3 Analyze 1-2  $\mu$ L of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 5.6 and Method 8000). The injection volume must be the same for all standards and sample extracts. Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

Calculate response factors (RFs) for each target analyte relative to one of the internal standards as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

- $A_s$  = Peak area (or height) of the analyte or surrogate.
- $A_{is}$  = Peak area (or height) of the internal standard.
- $C_s$  = Concentration of the analyte or surrogate, in  $\mu$ g/L.
- $C_{is}$  = Concentration of the internal standard, in  $\mu$ g/L.

#### 7.3.4 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

7.3.4.3 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

### 7.3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Section 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual CCC (see Table 4) must be less than or equal to 30%.

$$\text{mean RF} = \overline{\text{RF}} = \frac{\sum_{i=1}^n \text{RF}_i}{n}$$
$$\text{SD} = \sqrt{\frac{\sum_{i=1}^n (\text{RF}_i - \overline{\text{RF}})^2}{n-1}}$$

$$\text{RSD} = \frac{\text{SD}}{\overline{\text{RF}}} \times 100$$

7.3.5.3 If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Sec. 7.3.

7.3.5.4 If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000.

7.3.6 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

7.3.7 Linearity of target analytes - If the RSD of any target analytes is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation (Sec. 7.6.2).

7.3.7.1 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 in Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

**NOTE:** Method 8000 designates a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.7.2 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000B for method blank performance criteria.

#### 7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

#### 7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Table 4 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference for each CCC is less than or equal to 20%, then the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project,

and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

## 7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that sample extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10  $\mu\text{L}$  of the internal standard solution to the 1-mL concentrated sample extract obtained from sample preparation.

7.5.3 Inject a 1-2  $\mu\text{L}$  aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 7.3). The volume to be injected should contain 100 ng of base/neutral and 200 ng of acid surrogates (assuming 100% recovery), unless a more sensitive GC/MS system is being used and the surrogate solution is less concentrated than that listed in Sec. 5.7. The injection volume must be the same volume used for the calibration standards.

7.5.4 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 ng/ $\mu\text{L}$ , unless a more sensitive GC/MS system is being used).

**NOTE:** It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.5 The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM

may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

## 7.6 Qualitative analysis

7.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The RRT of the sample component is within  $\pm 0.06$  RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be identified, quantitated and reported as the sum of both compounds by the GC.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the

analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

- (1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within  $\pm 20\%$ . (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

## 7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 If the RSD of a compound's response factor is 15% or less, then the concentration in the extract may be determined using the average response factor ( $\overline{RF}$ ) from initial calibration data (Sec. 7.3.5). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas  $A_x$  and  $A_{is}$  should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.7.5 Quantitation of multicomponent compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/ $\mu$ L in the concentrated sample extract.



7.7.6 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are quantitated as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be summed and reported as the sum of both compounds.

## 8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Sec. 7.0 of Method 8000 and include calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8270:

8.2.1 The GC/MS system must be tuned to meet the DFTPP criteria listed in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 7.4, each 12 hours.

8.2.4 The RRT of the sample component must fall within the RRT window of the standard component provided in Sec. 7.6.1.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed, a septum is changed), see the guidance in Sec 8.2 of Method 8000 regarding whether recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 9.0 METHOD PERFORMANCE

9.1 Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations ranging from 5 to 1,300 µg/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7. These values are presented as guidance only and are not intended as absolute acceptance criteria. Laboratories should generate their own acceptance criteria for capillary column method performance. (See Method 8000.)

9.2 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of GC performance. These recoveries (using Method 3510 extraction) are presented in Table 8.

9.3 Method performance data (using Method 3541 Automated Soxhlet extraction) are presented in Table 9. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately 1 hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three determinations were performed and each extract was analyzed by gas chromatography/mass spectrometry following Method 8270. The low recovery of the more volatile compounds is probably due to volatilization losses during equilibration. These data are listed in Table 10 and were taken from Reference 7.

9.4 Surrogate precision and accuracy data are presented in Table 11 from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by Method 8270.

9.5 Single laboratory precision and bias data (using Method 3545 Accelerated Solvent Extraction) for semivolatile organic compounds are presented in Table 12. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (250, 2500, and 12,500 µg/kg) on three types of soil (clay, loam and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by Perstorp Environmental Soxtec™ (automated Soxhlet). The data presented in Table 12 represents seven replicate extractions and analyses for each individual sample and were taken from reference 9. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam 98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low-101.2%, mid-97.2% and high-99.2%.

9.6 Single laboratory precision and bias data (using Method 3561 SFE Extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (one, EC-1, a lake sediment from Environment Canada and the other, HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally-contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions range from 85 to 148% (overall average of 100%) based on the certified value (or a Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions range from 73 to 133% (overall average of 92%) based on the certified value for the marine sediment. The data are found in Tables 13 and 14 and were taken from Reference 10.

9.7 Single laboratory precision and accuracy data (using Method 3561 SFE Extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recoveries from four replicate extractions range from 60 to 122% (overall average of 89%) based on the certified value. Following are the instrument conditions that were utilized to extract a 3.4 g sample: Pressure - 300 atm; Time - 60 min.; Extraction fluid - CO<sub>2</sub>; Modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80°C; Restrictor temperature - 120°C; and, Trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 15 and were taken from Reference 11.

## 10.0 REFERENCES

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TABLE 1  
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75 <sup>a</sup>	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d <sub>4</sub> (IS)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	44,45,74
Thiophenol (Benzenethiol)	7.42	110	66,109,84
Methyl methanesulfonate	7.48	80	79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	79,95,109,140
Ethyl methanesulfonate	9.62	79	109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d <sub>8</sub> (IS)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	155,127,81,109
Diethyl sulfate	11.37	139	45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	108,77,79,90
Hexachloropropene	12.45	213	211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosopyrrolidine	12.65	100	41,42,68,69
Acetophenone	12.67	105	71,51,120
4-Methylphenol	12.82	107	108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	107,77,51,79
3-Methylphenol	12.93	107	108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,55,56,41
1,4-Phenylenediamine	13.62	108	80,53,54,52
1-Chloronaphthalene	13.65 <sup>a</sup>	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	76,50,148
o-Anisidine	15.00	108	80,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d <sub>10</sub> (IS)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	164,126,98,63
4-Chloroaniline	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00 <sup>a</sup>	143	115,116
1,4-Naphthoquinone	16.23	158	104,102,76,50,130
p-Cresidine	16.45	122	94,137,77,93
Dichlorovos	16.48	109	185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	135,134,91,77
N-Nitrosodi-n-butylamine	16.73	84	57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethyl phosphoramidate	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54 <sup>a</sup>	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro-o-toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d <sub>10</sub> (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosaffrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramidate	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
$\alpha,\alpha$ -Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d <sub>12</sub> (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyrilene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147



TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate <sup>b</sup>	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d <sub>12</sub> (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
$\alpha$ -BHC	--	183	181,109
$\beta$ -BHC	--	181	183,109
$\delta$ -BHC	--	183	181,109
$\gamma$ -BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d <sub>5</sub> (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d <sub>6</sub> (surr)	--	99	42,71
Terphenyl-d <sub>14</sub> (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard

surr = surrogate

<sup>a</sup>Estimated retention times

<sup>b</sup>Substitute for the non-specific mixture, tricresyl phosphate

TABLE 2

## ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

Compound	Estimated Quantitation Limits <sup>a</sup>	
	Ground water µg/L	Low Soil/Sediment <sup>b</sup> µg/kg
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-Bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits <sup>a</sup>	
	Ground water µg/L	Low Soil/Sediment <sup>b</sup> µg/kg
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butyl phthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phthalate	10	660
Diethylstilbestrol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
a,a-Dimethylphenethylamine	ND	ND
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits <sup>a</sup>	
	Ground water µg/L	Low Soil/Sediment <sup>b</sup> µg/kg
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl) phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethylphosphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydride	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
4,4'-Methylenebis(2-chloroaniline)	NA	ND
Methyl methanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits <sup>a</sup>	
	Ground water µg/L	Low Soil/Sediment <sup>b</sup> µg/kg
4-Methylphenol	10	660
Mevinphos	10	ND
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro-o-anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro-o-toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodi-n-butylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND
Octamethyl pyrophosphoramidate	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits <sup>a</sup>	
	Ground water µg/L	Low Soil/Sediment <sup>b</sup> µg/kg
Phthalic anhydride	100	ND
2-Picoline	ND	ND
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND
O,O,O-Triethyl phosphorothioate	NT	ND

<sup>a</sup> Sample EQLs are highly matrix-dependent. The EQLs listed here are provided for guidance and may not always be achievable.

<sup>b</sup> EQLs listed for soil/sediment are based on wet weight. Normally, data are reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined

NA = Not Applicable

NT = Not Tested

#### Other Matrices

#### Factor<sup>c</sup>

High-concentration soil and sludges by ultrasonic extractor

7.5

Non-water miscible waste

75

<sup>c</sup>EQL = (EQL for Low Soil/Sediment given above in Table 2) x (Factor)

TABLE 3  
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA<sup>a,b</sup>

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

<sup>a</sup> Data taken from Reference 3.

<sup>b</sup> Alternate tuning criteria may be used, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 4  
CALIBRATION CHECK COMPOUNDS (CCC)

<u>Base/Neutral Fraction</u>	<u>Acid Fraction</u>
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	



TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES  
ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d <sub>4</sub>	Naphthalene-d <sub>8</sub>	Acenaphthene-d <sub>10</sub>
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethylphenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d <sub>8</sub> (surr)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d <sub>6</sub> (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

TABLE 5  
(continued)

Phenanthrene-d <sub>10</sub>	Chrysene-d <sub>12</sub>	Perylene-d <sub>12</sub>
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethylaminoazobenzene	
Hexachlorobenzene	Pyrene	
N-Nitrosodiphenylamine	Terphenyl-d <sub>14</sub> (surr)	
Pentachlorophenol	7,12-Dimethylbenzo(a)anthracene	
Pentachloronitrobenzene	Di-n-octyl phthalate	
Phenacetin	Indeno(1,2,3-cd)pyrene	
Phenanthrene	3-Methylcholanthrene	
Pronamide		

(surr) = surrogate

TABLE 6  
MULTILABORATORY PERFORMANCE DATA<sup>a</sup>

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p <sub>s</sub> (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192

TABLE 6  
(continued)

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p <sub>s</sub> (%)
Heptachlor epoxide	100	54.7	70.9-109.4	26-155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L

$\bar{x}$  = Average recovery for four recovery measurements, in µg/L

p, p<sub>s</sub> = Measured percent recovery

D = Detected; result must be greater than zero

<sup>a</sup> Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION<sup>a</sup>

Compound	Accuracy, as recovery, $x'$ ( $\mu\text{g/L}$ )	Single analyst precision, $s_r'$ ( $\mu\text{g/L}$ )	Overall precision, $S'$ ( $\mu\text{g/L}$ )
Acenaphthene	0.96C+0.19	0.15 $\bar{x}$ -0.12	0.21 $\bar{x}$ -0.67
Acenaphthylene	0.89C+0.74	0.24 $\bar{x}$ -1.06	0.26 $\bar{x}$ -0.54
Aldrin	0.78C+1.66	0.27 $\bar{x}$ -1.28	0.43 $\bar{x}$ +1.13
Anthracene	0.80C+0.68	0.21 $\bar{x}$ -0.32	0.27 $\bar{x}$ -0.64
Benz(a)anthracene	0.88C-0.60	0.15 $\bar{x}$ +0.93	0.26 $\bar{x}$ -0.21
Benzo(b)fluoranthene	0.93C-1.80	0.22 $\bar{x}$ +0.43	0.29 $\bar{x}$ +0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19 $\bar{x}$ +1.03	0.35 $\bar{x}$ +0.40
Benzo(a)pyrene	0.90C-0.13	0.22 $\bar{x}$ +0.48	0.32 $\bar{x}$ +1.35
Benzo(g,h,i)perylene	0.98C-0.86	0.29 $\bar{x}$ +2.40	0.51 $\bar{x}$ -0.44
Benzyl butyl phthalate	0.66C-1.68	0.18 $\bar{x}$ +0.94	0.53 $\bar{x}$ +0.92
$\beta$ -BHC	0.87C-0.94	0.20 $\bar{x}$ -0.58	0.30 $\bar{x}$ +1.94
$\delta$ -BHC	0.29C-1.09	0.34 $\bar{x}$ +0.86	0.93 $\bar{x}$ -0.17
Bis(2-chloroethyl) ether	0.86C-1.54	0.35 $\bar{x}$ -0.99	0.35 $\bar{x}$ +0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16 $\bar{x}$ +1.34	0.26 $\bar{x}$ +2.01
Bis(2-chloroisopropyl) ether	1.03C-2.31	0.24 $\bar{x}$ +0.28	0.25 $\bar{x}$ +1.04
Bis(2-ethylhexyl) phthalate	0.84C-1.18	0.26 $\bar{x}$ +0.73	0.36 $\bar{x}$ +0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13 $\bar{x}$ +0.66	0.16 $\bar{x}$ +0.66
2-Chloronaphthalene	0.89C+0.01	0.07 $\bar{x}$ +0.52	0.13 $\bar{x}$ +0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20 $\bar{x}$ -0.94	0.30 $\bar{x}$ -0.46
Chrysene	0.93C-1.00	0.28 $\bar{x}$ +0.13	0.33 $\bar{x}$ -0.09
4,4'-DDD	0.56C-0.40	0.29 $\bar{x}$ -0.32	0.66 $\bar{x}$ -0.96
4,4'-DDE	0.70C-0.54	0.26 $\bar{x}$ -1.17	0.39 $\bar{x}$ -1.04
4,4'-DDT	0.79C-3.28	0.42 $\bar{x}$ +0.19	0.65 $\bar{x}$ -0.58
Dibenzo(a,h)anthracene	0.88C+4.72	0.30 $\bar{x}$ +8.51	0.59 $\bar{x}$ +0.25
Di-n-butyl phthalate	0.59C+0.71	0.13 $\bar{x}$ +1.16	0.39 $\bar{x}$ +0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20 $\bar{x}$ +0.47	0.24 $\bar{x}$ +0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25 $\bar{x}$ +0.68	0.41 $\bar{x}$ +0.11
1,4-Dichlorobenzene	0.73C-1.47	0.24 $\bar{x}$ +0.23	0.29 $\bar{x}$ +0.36
3,3'-Dichlorobenzidine	1.23C-12.65	0.28 $\bar{x}$ +7.33	0.47 $\bar{x}$ +3.45
Dieldrin	0.82C-0.16	0.20 $\bar{x}$ -0.16	0.26 $\bar{x}$ -0.07
Diethyl phthalate	0.43C+1.00	0.28 $\bar{x}$ +1.44	0.52 $\bar{x}$ +0.22
Dimethyl phthalate	0.20C+1.03	0.54 $\bar{x}$ +0.19	1.05 $\bar{x}$ -0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12 $\bar{x}$ +1.06	0.21 $\bar{x}$ +1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14 $\bar{x}$ +1.26	0.19 $\bar{x}$ +0.35
Di-n-octyl phthalate	0.76C-0.79	0.21 $\bar{x}$ +1.19	0.37 $\bar{x}$ +1.19
Endosulfan sulfate	0.39C+0.41	0.12 $\bar{x}$ +2.47	0.63 $\bar{x}$ -1.03
Endrin aldehyde	0.76C-3.86	0.18 $\bar{x}$ +3.91	0.73 $\bar{x}$ -0.62
Fluoranthene	0.81C+1.10	0.22 $\bar{x}$ -0.73	0.28 $\bar{x}$ -0.60
Fluorene	0.90C-0.00	0.12 $\bar{x}$ +0.26	0.13 $\bar{x}$ +0.61
Heptachlor	0.87C-2.97	0.24 $\bar{x}$ -0.56	0.50 $\bar{x}$ -0.23
Heptachlor epoxide	0.92C-1.87	0.33 $\bar{x}$ -0.46	0.28 $\bar{x}$ +0.64

TABLE 7  
(continued)

Compound	Accuracy, as recovery, $x'$ ( $\mu\text{g/L}$ )	Single analyst precision, $s_r'$ ( $\mu\text{g/L}$ )	Overall precision, $S'$ ( $\mu\text{g/L}$ )
Hexachlorobenzene	0.74C+0.66	0.18 $\bar{x}$ -0.10	0.43 $\bar{x}$ -0.52
Hexachlorobutadiene	0.71C-1.01	0.19 $\bar{x}$ +0.92	0.26 $\bar{x}$ +0.49
Hexachloroethane	0.73C-0.83	0.17 $\bar{x}$ +0.67	0.17 $\bar{x}$ +0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29 $\bar{x}$ +1.46	0.50 $\bar{x}$ -0.44
Isophorone	1.12C+1.41	0.27 $\bar{x}$ +0.77	0.33 $\bar{x}$ +0.26
Naphthalene	0.76C+1.58	0.21 $\bar{x}$ -0.41	0.30 $\bar{x}$ -0.68
Nitrobenzene	1.09C-3.05	0.19 $\bar{x}$ +0.92	0.27 $\bar{x}$ +0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27 $\bar{x}$ +0.68	0.44 $\bar{x}$ +0.47
Aroclor 1260	0.81C-10.86	0.35 $\bar{x}$ +3.61	0.43 $\bar{x}$ +1.82
Phenanthrene	0.87C+0.06	0.12 $\bar{x}$ +0.57	0.15 $\bar{x}$ +0.25
Pyrene	0.84C-0.16	0.16 $\bar{x}$ +0.06	0.15 $\bar{x}$ +0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15 $\bar{x}$ +0.85	0.21 $\bar{x}$ +0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23 $\bar{x}$ +0.75	0.29 $\bar{x}$ +1.31
2-Chlorophenol	0.78C+0.29	0.18 $\bar{x}$ +1.46	0.28 $\bar{x}$ +0.97
2,4-Dichlorophenol	0.87C-0.13	0.15 $\bar{x}$ +1.25	0.21 $\bar{x}$ +1.28
2,4-Dimethylphenol	0.71C+4.41	0.16 $\bar{x}$ +1.21	0.22 $\bar{x}$ +1.31
2,4-Dinitrophenol	0.81C-18.04	0.38 $\bar{x}$ +2.36	0.42 $\bar{x}$ +26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10 $\bar{x}$ +42.29	0.26 $\bar{x}$ +23.10
2-Nitrophenol	0.07C-1.15	0.16 $\bar{x}$ +1.94	0.27 $\bar{x}$ +2.60
4-Nitrophenol	0.61C-1.22	0.38 $\bar{x}$ +2.57	0.44 $\bar{x}$ +3.24
Pentachlorophenol	0.93C+1.99	0.24 $\bar{x}$ +3.03	0.30 $\bar{x}$ +4.33
Phenol	0.43C+1.26	0.26 $\bar{x}$ +0.73	0.35 $\bar{x}$ +0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16 $\bar{x}$ +2.22	0.22 $\bar{x}$ +1.81

$x'$  = Expected recovery for one or more measurements of a sample containing a concentration of C, in  $\mu\text{g/L}$ .

$s_r'$  = Expected single analyst standard deviation of measurements at an average concentration of  $\bar{x}$ , in  $\mu\text{g/L}$ .

$S'$  = Expected interlaboratory standard deviation of measurements at an average concentration found of  $\bar{x}$ , in  $\mu\text{g/L}$ .

C = True value for the concentration, in  $\mu\text{g/L}$ .

$\bar{x}$  = Average recovery found for measurements of samples containing a concentration of C, in  $\mu\text{g/L}$ .

<sup>a</sup> Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 8

## EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

Compound	Percent Recovery on Day 0		Percent Recovery on Day 7	
	Mean	RSD	Mean	RSD
3-Amino-9-ethylcarbazole	80	8	73	3
4-Chloro-1,2-phenylenediamine	91	1	108	4
4-Chloro-1,3-phenylenediamine	84	3	70	3
1,2-Dibromo-3-chloropropane	97	2	98	5
Dinoseb	99	3	97	6
Parathion	100	2	103	4
4,4'-Methylenebis(N,N-dimethylaniline)	108	4	90	4
5-Nitro-o-toluidine	99	10	93	4
2-Picoline	80	4	83	4
Tetraethyl dithiopyrophosphate	92	7	70	1

Data taken from Reference 6.

TABLE 9

MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE ORGANICS  
FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET EXTRACTION  
(METHOD 3541) WITH HEXANE-ACETONE (1:1)<sup>a</sup>

Compound	Clay Soil		Topsoil	
	Mean Recovery	RSD	Mean Recovery	RSD
1,3-Dichlorobenzene	0	--	0	--
1,2-Dichlorobenzene	0	--	0	--
Nitrobenzene	0	--	0	--
Benzal chloride	0	--	0	--
Benzotrichloride	0	--	0	--
4-Chloro-2-nitrotoluene	0	--	0	--
Hexachlorocyclopentadiene	4.1	15	7.8	23
2,4-Dichloronitrobenzene	35.2	7.6	21.2	15
3,4-Dichloronitrobenzene	34.9	15	20.4	11
Pentachlorobenzene	13.7	7.3	14.8	13
2,3,4,5-Tetrachloronitrobenzene	55.9	6.7	50.4	6.0
Benefin	62.6	4.8	62.7	2.9
alpha-BHC	58.2	7.3	54.8	4.8
Hexachlorobenzene	26.9	13	25.1	5.7
delta-BHC	95.8	4.6	99.2	1.3
Heptachlor	46.9	9.2	49.1	6.3
Aldrin	97.7	12	102	7.4
Isopropalin	102	4.3	105	2.3
Heptachlor epoxide	90.4	4.4	93.6	2.4
trans-Chlordane	90.1	4.5	95.0	2.3
Endosulfan I	96.3	4.4	101	2.2
Dieldrin	129	4.7	104	1.9
2,5-Dichlorophenyl-4-nitrophenyl ether	110	4.1	112	2.1
Endrin	102	4.5	106	3.7
Endosulfan II	104	4.1	105	0.4
p,p'-DDT	134	2.1	111	2.0
2,3,6-Trichlorophenyl-4'-nitrophenyl ether	110	4.8	110	2.8
2,3,4-Trichlorophenyl-4'-nitrophenyl ether	112	4.4	112	3.3
Mirex	104	5.3	108	2.2

<sup>a</sup> The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4-nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.



TABLE 10

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION  
OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY  
AUTOMATED SOXHLET (METHOD 3541)<sup>a</sup>

Compound	Mean Recovery	RSD
Phenol	47.8	5.6
Bis(2-chloroethyl)ether	25.4	13
2-Chlorophenol	42.7	4.3
Benzyl alcohol	55.9	7.2
2-Methylphenol	17.6	6.6
Bis(2-chloroisopropyl)ether	15.0	15
4-Methylphenol	23.4	6.7
N-Nitroso-di-n-propylamine	41.4	6.2
Nitrobenzene	28.2	7.7
Isophorone	56.1	4.2
2-Nitrophenol	36.0	6.5
2,4-Dimethylphenol	50.1	5.7
Benzoic acid	40.6	7.7
Bis(2-chloroethoxy)methane	44.1	3.0
2,4-Dichlorophenol	55.6	4.6
1,2,4-Trichlorobenzene	18.1	31
Naphthalene	26.2	15
4-Chloroaniline	55.7	12
4-Chloro-3-methylphenol	65.1	5.1
2-Methylnaphthalene	47.0	8.6
Hexachlorocyclopentadiene	19.3	19
2,4,6-Trichlorophenol	70.2	6.3
2,4,5-Trichlorophenol	26.8	2.9
2-Chloronaphthalene	61.2	6.0
2-Nitroaniline	73.8	6.0
Dimethyl phthalate	74.6	5.2
Acenaphthylene	71.6	5.7
3-Nitroaniline	77.6	5.3
Acenaphthene	79.2	4.0
2,4-Dinitrophenol	91.9	8.9
4-Nitrophenol	62.9	16
Dibenzofuran	82.1	5.9
2,4-Dinitrotoluene	84.2	5.4
2,6-Dinitrotoluene	68.3	5.8
Diethyl phthalate	74.9	5.4
4-Chlorophenyl-phenyl ether	67.2	3.2
Fluorene	82.1	3.4
4-Nitroaniline	79.0	7.9

TABLE 10  
(continued)

Compound	Mean Recovery	RSD
4,6-Dinitro-2-methylphenol	63.4	6.8
N-Nitrosodiphenylamine	77.0	3.4
4-Bromophenyl-phenyl ether	62.4	3.0
Hexachlorobenzene	72.6	3.7
Pentachlorophenol	62.7	6.1
Phenanthrene	83.9	5.4
Anthracene	96.3	3.9
Di-n-butyl phthalate	78.3	40
Fluoranthene	87.7	6.9
Pyrene	102	0.8
Butyl benzyl phthalate	66.3	5.2
3,3'-Dichlorobenzidine	25.2	11
Benzo(a)anthracene	73.4	3.8
Bis(2-ethylhexyl) phthalate	77.2	4.8
Chrysene	76.2	4.4
Di-n-octyl phthalate	83.1	4.8
Benzo(b)fluoranthene	82.7	5.0
Benzo(k)fluoranthene	71.7	4.1
Benzo(a)pyrene	71.7	4.1
Indeno(1,2,3-cd)pyrene	72.2	4.3
Dibenzo(a,h)anthracene	66.7	6.3
Benzo(g,h,i)perylene	63.9	8.0
1,2-Dichlorobenzene	0	--
1,3-Dichlorobenzene	0	--
1,4-Dichlorobenzene	0	--
Hexachloroethane	0	--
Hexachlorobutadiene	0	--

<sup>a</sup> Number of determinations was three. The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil; the spike concentration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spiking.

Data taken from Reference 7.

TABLE 11

PRECISION AND BIAS VALUES FOR METHOD 3542<sup>1</sup>

Compound	Mean Recovery	Standard Deviation	Relative Standard Deviation Percent
2-Fluorophenol	74.6	28.6	38.3
Phenol-d <sub>5</sub>	77.8	27.7	35.6
Nitrobenzene-d <sub>5</sub>	65.6	32.5	49.6
2-Fluorobiphenyl	75.9	30.3	39.9
2,4,6-Tribromophenol	67.0	34.0	50.7
Terphenyl-d <sub>14</sub>	78.6	32.4	41.3

<sup>1</sup> The surrogate values shown in Table 11 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 12

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES  
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Phenol	93.3	78.7	135.9	73.9	82.8	124.6	108.8	130.6	89.7	<b>102.0</b>
Bis(2-chloroethyl) ether	102.1	85.1	109.1	96.0	88.0	103.6	122.3	119.9	90.8	<b>101.9</b>
2-Chlorophenol	100.8	82.6	115.0	93.8	88.9	111.1	115.0	115.3	91.9	<b>101.6</b>
1,3-Dichlorobenzene	127.7	129.7	110.0	*364.2	129.9	119.0	*241.3	*163.7	107.1	<b>120.6</b>
1,4-Dichlorobenzene	127.9	127.0	110.5	*365.9	127.8	116.4	*309.6	*164.1	105.8	<b>119.2</b>
1,2-Dichlorobenzene	116.8	115.8	101.3	*159.2	113.4	105.5	*189.3	134.0	100.4	<b>112.5</b>
2-Methylphenol	98.9	82.1	119.7	87.6	89.4	111.0	133.2	128.0	92.1	<b>104.7</b>
Bis(2-chloroisopropyl)ether	109.4	71.5	108.0	81.8	81.0	88.6	118.1	148.3	94.8	<b>100.2</b>
o-Toluidine	100.0	89.7	117.2	100.0	*152.5	120.3	100.0	*199.5	102.7	<b>110.3</b>
N-Nitroso-di-n-propylamine	103.0	79.1	107.7	83.9	88.1	96.2	109.9	123.3	91.4	<b>98.1</b>
Hexachloroethane	97.1	125.1	111.0	*245.4	117.1	128.1	*566.7	147.9	103.7	<b>118.6</b>
Nitrobenzene	104.8	82.4	106.6	86.8	84.6	101.7	119.7	122.1	93.3	<b>100.2</b>
Isophorone	100.0	86.4	98.2	87.1	87.5	109.7	135.5	118.4	92.7	<b>101.7</b>
2,4-Dimethylphenol	100.0	104.5	140.0	100.0	114.4	123.1	100.0	*180.6	96.3	<b>109.8</b>
2-Nitrophenol	80.7	80.5	107.9	91.4	86.7	103.2	122.1	107.1	87.0	<b>96.3</b>
Bis(chloroethoxy)methane	94.4	80.6	94.7	86.5	84.4	99.6	130.6	110.7	93.2	<b>97.2</b>
2,4-Dichlorophenol	88.9	87.8	111.4	85.9	87.6	103.5	123.3	107.0	92.1	<b>98.6</b>
1,2,4-Trichlorobenzene	98.0	97.8	98.8	123.0	93.7	94.5	137.0	99.4	95.3	<b>104.2</b>
Naphthalene	101.7	97.2	123.6	113.2	102.9	129.5	*174.5	114.0	89.8	<b>106.1</b>
4-Chloroaniline	100.0	*150.2	*162.4	100.0	125.5	*263.6	100.0	*250.8	114.9	<b>108.1</b>
Hexachlorobutadiene	101.1	98.7	102.2	124.1	90.3	98.0	134.9	96.1	96.8	<b>104.7</b>
4-Chloro-3-methylphenol	90.4	80.2	114.7	79.0	85.2	109.8	131.6	116.2	90.1	<b>99.7</b>
2-Methylnaphthalene	93.2	89.9	94.6	104.1	92.2	105.9	146.2	99.1	93.3	<b>102.1</b>
Hexachlorocyclopentadiene	100.0	100.0	0.0	100.0	100.0	6.8	100.0	100.0	*238.3	<b>75.8</b>
2,4,6-Trichlorophenol	94.6	90.0	112.0	84.2	91.2	103.6	101.6	95.9	89.8	<b>95.9</b>
2,4,5-Trichlorophenol	84.4	91.9	109.6	96.1	80.7	103.6	108.9	83.9	87.9	<b>94.1</b>
2-Chloronaphthalene	100.0	91.3	93.6	97.6	93.4	98.3	106.8	93.0	92.0	<b>96.2</b>
2-Nitroaniline	90.0	83.4	97.4	71.3	88.4	89.9	112.1	113.3	87.7	<b>92.6</b>
2,6-Dinitrotoluene	83.1	90.6	91.6	86.4	90.6	90.3	104.3	84.7	90.9	<b>90.3</b>
Acenaphthylene	104.9	95.9	100.5	99.0	97.9	108.8	118.5	97.8	92.0	<b>101.7</b>
3-Nitroaniline	*224.0	115.6	97.6	100.0	111.8	107.8	0.0	111.7	99.0	<b>92.9</b>
Acenaphthene	102.1	92.6	97.6	97.2	96.9	104.4	114.2	92.0	89.0	<b>98.4</b>
4-Nitrophenol	0.0	93.2	121.5	18.1	87.1	116.6	69.1	90.5	84.5	<b>75.6</b>
2,4-Dinitrotoluene	73.9	91.9	100.2	84.7	93.8	98.9	100.9	84.3	87.3	<b>90.7</b>

TABLE 12 (cont.)

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES  
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Dibenzofuran	89.5	91.7	109.3	98.5	92.2	111.4	113.8	92.7	90.4	<b>98.8</b>
4-Chlorophenyl phenyl ether	83.0	94.5	98.7	95.7	94.3	94.2	111.4	87.7	90.3	<b>94.4</b>
Fluorene	85.2	94.9	89.2	102.0	95.5	93.8	121.3	85.7	90.9	<b>95.4</b>
4-Nitroaniline	77.8	114.8	94.5	129.6	103.6	95.4	*154.1	89.3	87.5	<b>99.1</b>
N-Nitrosodiphenylamine	82.6	96.7	93.8	92.9	93.4	116.4	97.5	110.9	86.7	<b>96.8</b>
4-Bromophenyl phenyl ether	85.6	92.9	92.8	91.1	107.6	89.4	118.0	97.5	87.1	<b>95.8</b>
Hexachlorobenzene	95.4	91.7	92.3	95.4	93.6	83.7	106.8	94.3	90.0	<b>93.7</b>
Pentachlorophenol	68.2	85.9	107.7	53.2	89.8	88.1	96.6	59.8	81.3	<b>81.2</b>
Phenanthrene	92.1	93.7	93.3	100.0	97.8	113.3	124.4	101.0	89.9	<b>100.6</b>
Anthracene	101.6	95.0	93.5	92.5	101.8	118.4	123.0	94.5	90.6	<b>101.2</b>
Carbazole	94.4	99.3	96.6	105.5	96.7	111.4	115.7	83.2	88.9	<b>99.1</b>
Fluoranthene	109.9	101.4	94.3	111.6	96.6	109.6	123.2	85.4	92.7	<b>102.7</b>
Pyrene	106.5	105.8	107.6	116.7	90.7	127.5	103.4	95.5	93.2	<b>105.2</b>
3,3'-Dichlorobenzidine	100.0	*492.3	131.4	100.0	*217.6	*167.6	100.0	*748.8	100.0	<b>116.5</b>
Benzo(a)anthracene	98.1	107.0	98.4	119.3	98.6	104.0	105.0	93.4	89.3	<b>101.5</b>
Chrysene	100.0	108.5	100.2	116.8	93.0	117.0	106.7	93.6	90.2	<b>102.9</b>
Benzo(b)fluoranthene	106.6	109.9	75.6	121.7	100.7	93.9	106.9	81.9	93.6	<b>99.0</b>
Benzo(k)fluoranthene	102.4	105.2	88.4	125.5	99.4	95.1	144.7	89.2	78.1	<b>103.1</b>
Benzo(a)pyrene	107.9	105.5	80.8	122.3	97.7	104.6	101.7	86.2	92.0	<b>99.9</b>
Indeno(1,2,3-cd)pyrene	95.1	105.7	93.8	126.0	105.2	90.4	133.6	82.6	91.9	<b>102.7</b>
Dibenz(a,h)anthracene	85.0	102.6	82.0	118.8	100.7	91.9	142.3	71.0	93.1	<b>98.6</b>
Benzo(g,h,i)perylene	98.0	0.0	81.2	0.0	33.6	78.6	128.7	83.0	94.2	<b>66.4</b>
<b>Average</b>	<b>95.1</b>	<b>94.3</b>	<b>101.0</b>	<b>95.5</b>	<b>96.5</b>	<b>104.1</b>	<b>113.0</b>	<b>100.9</b>	<b>92.5</b>	

\* Values greater than 150% were not used to determine the averages, but the 0% values were used.

TABLE 13

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value <sup>a</sup> (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	(27.9) <sup>b</sup>	41.3 ± 3.6	(148)	8.7
Acenaphthylene	(0.8)	0.9 ± 0.1	(112)	11.1
Acenaphthene	(0.2)	0.2 ± 0.01	(100)	0.05
Fluorene	(15.3)	15.6 ± 1.8	(102)	11.5
Phenanthrene	15.8 ± 1.2	16.1 ± 1.8	102	11.2
Anthracene	(1.3)	1.1 ± 0.2	(88)	18.2
Fluoranthene	23.2 ± 2.0	24.1 ± 2.1	104	8.7
Pyrene	16.7 ± 2.0	17.2 ± 1.9	103	11.0
Benz(a)anthracene	8.7 ± 0.8	8.8 ± 1.0	101	11.4
Chrysene	(9.2)	7.9 ± 0.9	(86)	11.4
Benzo(b)fluoranthene	7.9 ± 0.9	8.5 ± 1.1	108	12.9
Benzo(k)fluoranthene	4.4 ± 0.5	4.1 ± 0.5	91	12.2
Benzo(a)pyrene	5.3 ± 0.7	5.1 ± 0.6	96	11.8
Indeno(1,2,3-cd)pyrene	5.7 ± 0.6	5.2 ± 0.6	91	11.5
Benzo(g,h,i)perylene	4.9 ± 0.7	4.3 ± 0.5	88	11.6
Dibenz(a,h)anthracene	(1.3)	1.1 ± 0.2	(85)	18.2

<sup>a</sup> Relative standard deviations for the SFE values are based on six replicate extractions.

<sup>b</sup> Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 14

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value <sup>a</sup> (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	9.0 ± 0.7	7.4 ± 0.6	82	8.1
Acenaphthylene	0.3 ± 0.1	0.4 ± 0.1	133	25.0
Acenaphthene	4.5 ± 1.5	3.3 ± 0.3	73	9.0
Fluorene	13.6 ± 3.1	10.4 ± 1.3	77	12.5
Phenanthrene	85.0 ± 20.0	86.2 ± 9.5	101	11.0
Anthracene	13.4 ± 0.5	12.1 ± 1.5	90	12.4
Fluoranthene	60.0 ± 9.0	54.0 ± 6.1	90	11.3
Pyrene	39.0 ± 9.0	32.7 ± 3.7	84	11.3
Benz(a)anthracene	14.6 ± 2.0	12.1 ± 1.3	83	10.7
Chrysene	14.1 ± 2.0	12.0 ± 1.3	85	10.8
Benzo(b)fluoranthene	7.7 ± 1.2	8.4 ± 0.9	109	10.7
Benzo(k)fluoranthene	2.8 ± 2.0	3.2 ± 0.5	114	15.6
Benzo(a)pyrene	7.4 ± 3.6	6.6 ± 0.8	89	12.1
Indeno(1,2,3-cd)pyrene	5.0 ± 2.0	4.5 ± 0.6	90	13.3
Benzo(g,h,i)perylene	5.4 ± 1.3	4.4 ± 0.6	82	13.6
Dibenz(a,h)anthracene	1.3 ± 0.5	1.1 ± 0.3	85	27.3

<sup>a</sup> Relative standard deviations for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 15

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs  
FROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561  
(SFE - LIQUID TRAP)

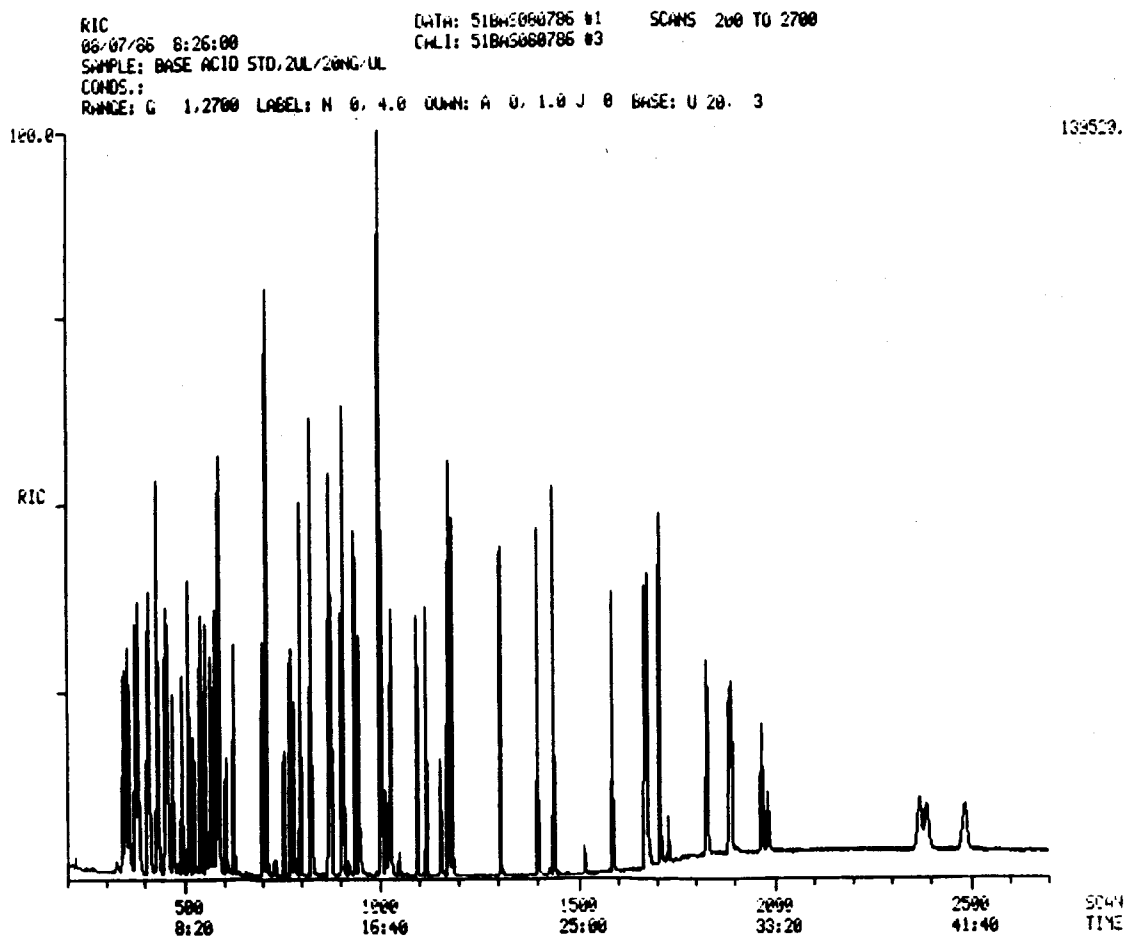
Compound	Certified Value (mg/kg)		SFE Value <sup>a</sup> (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	32.4	± 8.2	29.55	91	10.5
2-Methylnaphthalene	62.1	± 11.5	76.13	122	2.0
Acenaphthene	632	± 105	577.28	91	2.9
Dibenzofuran	307	± 49	302.25	98	4.1
Fluorene	492	± 78	427.15	87	3.0
Phenanthrene	1618	± 340	1278.03	79	3.4
Anthracene	422	± 49	400.80	95	2.6
Fluoranthene	1280	± 220	1019.13	80	4.5
Pyrene	1033	± 285	911.82	88	3.1
Benz(a)anthracene	252	± 38	225.50	89	4.8
Chrysene	297	± 26	283.00	95	3.8
Benzo(b)fluoranthene + Benzo(k)fluoranthene	153	± 22	130.88	86	10.7
Benzo(a)pyrene	97.2	± 17.1	58.28	60	6.5

<sup>a</sup> Relative standard deviations for the SFE values are based on four replicate extractions.

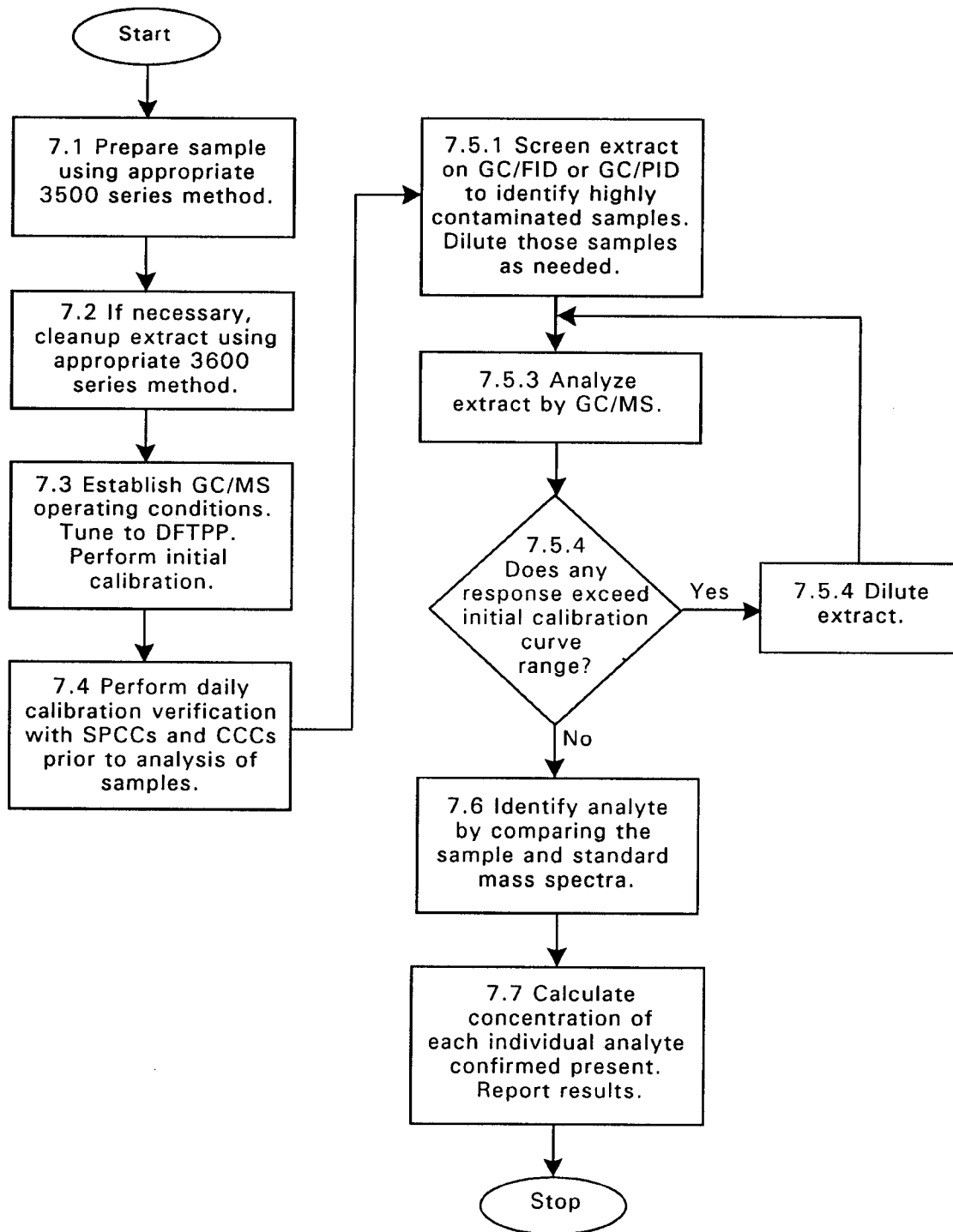
Data are taken from Reference 11.



FIGURE 1  
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD



METHOD 8270C  
SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS  
SPECTROMETRY (GC/MS)



# **APPENDIX E**

EPA Method  
Standard Operating Procedures

SOP Number: GCMS\_VOA.SOP  
Revision: 0

Effective Date: 09/18/06  
Supersedes: 8260B.SOP, Rev. 7 (3/13/01)  
624.SOP, Rev. 5 (01/02/01)

**TESTAMERICA-IRVINE  
STANDARD OPERATING PROCEDURE  
VOLATILE ORGANIC COMPOUNDS BY GAS  
CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS)  
EPA METHODS 8260B & 624**

Approved By: *Valerie Seychuk* Date: 9/18/06  
Department Manager

Approved By: *J.P.D.* Date: 9/18/06  
Quality Assurance Manager

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**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS**  
**SPECTROMETRY (GC/MS)**

**1. SCOPE AND APPLICATION**

- 1.1. The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The analytes are introduced to a capillary column, which is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer.
- 1.2. Method 8260 is used to determine volatile organic compounds in a variety of matrices, including but not exclusively: ground and surface water, aqueous sludge, waste solvents, oily wastes, various air sampling trapping media, tars, fibrous wastes, filter cakes, spent carbons, soils, and sediments. Method 624 is used for aqueous samples only.
- 1.3. Water samples (Method 5030B) and soils samples (Method 5030B or 5035) with low levels of target analytes are loaded onto a purge and trap autosampler. Samples containing high levels of target analytes are diluted prior to being loaded onto an autosampler. High level soil samples are extracted (Method 5035) and diluted with Methanol (MeOH) and water samples are diluted with ultrapure water. The samples are purged with Helium, which extracts the target compounds into a vapor phase. The vapor concentrates onto the trap which is then heated to release the target compounds into the gas chromatograph column.
- 1.4. The capillary gas chromatography column is interfaced to a mass spectrometer. Analytes are identified by comparing their measured mass spectra and retention times to the calibration standards' reference spectra and retention times. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using at least a five-point calibration curve.
- 1.5. See attached Analysis codes for applicable analyte list and reporting limits.

**2. DEFINITIONS**

- 2.1. There are no additional specific definitions associated with this test. See the QAPM and EPA Method 8000B, EPA Method 8260B, and EPA Method 624 for general definitions.

**3. APPARATUS AND MATERIALS**

- 3.1. Equipment and Supplies
  - 3.1.1. 1-ml vials with polytetrafluoroethylene (PTFE)-lined screw caps
  - 3.1.2. Teflon tape
  - 3.1.3. Fume hood
  - 3.1.4. 10 µl, 100 µl, and 250 µl micro syringes
  - 3.1.5. 1-ml and 10-ml syringes
  - 3.1.6. 1-ml vials with mininert screw caps
  - 3.1.7. Beakers
  - 3.1.8. 40ml glass vov screw top vials

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- 3.1.9. pH Test Strips
- 3.1.10. Encore™ sample extrusion tool
- 3.2. Reagents and Standards
  - 3.2.1. The vendors indicated below currently supply the following standards and reagents. In the instance where a different vendor might be used, an equivalent product will be purchased. As long as the item is documented clearly in a logbook or on the raw data, this SOP will remain valid though the vendor may have changed.
  - 3.2.2. Methanol (MeOH) – purge and trap grade (Burdick and Jackson or equiv.).
  - 3.2.3. Absolute Standard 502/524 VOC Mix #1 (2000 µg/ml)
  - 3.2.4. Absolute Standard 502/524 Volatile Compound Mix (2000µg/ml)
  - 3.2.5. O<sub>2</sub>Si 8260 VOC solution (60 compounds) (2000 ug/ml) for LCS
  - 3.2.6. Absolute Internal Standard (2000 µg/ml)
  - 3.2.7. Absolute Surrogate Standard (2000 µg/ml)
  - 3.2.8. Absolute MTBE (1000 µg/ml)
  - 3.2.9. Absolute Standard Ketone Mix (2000 µg/ml) add O<sub>2</sub>Si @ 2000 µg/ml (LCS)
  - 3.2.10. Absolute 4-Bromofluorobenzene (2500 µg/ml)
  - 3.2.11. Absolute 2-CEVE (1000 µg/ml and 2000 µg/ml) add O<sub>2</sub>Si @ 2000ppm (LCS)
  - 3.2.12. Restek Vinyl Acetate (2000 µg/ml) (two separate lot number)
  - 3.2.13. Absolute Carbon Disulfide (1000 µg/ml) add O<sub>2</sub>Si @ 2000 µg/ml for LCS
  - 3.2.14. Absolute Oxygenates Initial Calibration Standard, custom mix (2500 µg/ml)
  - 3.2.15. Absolute gasoline additive mix (2000 µg/ml)
  - 3.2.16. Absolute Tert-butyl alcohol (1000 µg/ml and 20,000 µg/ml)
  - 3.2.17. O<sub>2</sub>Si TBA-d9 Standard 40000 µg/ml
  - 3.2.18. O<sub>2</sub>Si Oxygenates Mix for the LCS 2000 µg/ml
  - 3.2.19. O<sub>2</sub>Si Tert-butyl alcohol for the LCS 2000 µg/ml
  - 3.2.20. Restek Ethanol 20000ug/ml
  - 3.2.21. Absolute 2-Chloro-1,3-Butadiene (1000 ug/ml)
  - 3.2.22. Absolute Thiophene (1000 ug/ml)

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- 3.2.23. Absolute 1,2-Dichlorotrifluoroethane (1000 ug/ml)
- 3.2.24. Absolute Iodomethane (1000 ug/ml)
- 3.2.25. Absolute 1,2-Dibromotetrafluoroethane (1000 ug/ml)
- 3.2.26. Absolute Freon 113 (1000 ug/ml)
- 3.2.27. O<sub>2</sub>Si t-Butyl Formate (1000 ug/ml)
- 3.2.28. O<sub>2</sub>Si Acrolein (200 ug/ml)
- 3.2.29. O<sub>2</sub>Si Freon Mix 3-1 (200 ug/ml)
- 3.2.30. Restek Custom Revised VOC Mix #1 (20,000 ug/ml)
- 3.2.31. Restek Custom 1,2-Dibromotetrafluoroethane (2000 ug/ml)
- 3.2.32. Restek Custom Halogenated Alkanes Plus Standard (2000 ug/ml)
- 3.2.33. Restek Custom Isopropanol (20,000 ug/ml)
- 3.2.34. Restek 1,1,2-Trichlorotrifluoroethane (2000 ug/ml)
- 3.2.35. Absolute Appendix II Volatile Standard/11 Components (2000 ug/ml, 20,000 ug/ml)
- 3.2.36. AccuStandard Acrylonitrile (1000 ug/ml)
- 3.2.37. O<sub>2</sub>Si Acrolein (different lot# for 2<sup>nd</sup> source) (200 ug/ml)
- 3.2.38. O<sub>2</sub>Si Freon Mix 3-1 (different lot# for 2<sup>nd</sup> source) (200 ug/ml)
- 3.2.39. Absolute Henderson VOC Mix (2000 ug/ml)
- 3.2.40. Absolute Henderson VOC Mix (different lot# for 2<sup>nd</sup> source) (2000 ug/ml)
- 3.2.41. Ultrapure water

**4. SAFETY**

- 4.1. Since all of the hazards of samples and chemicals used in this procedure are not entirely known, strict adherence to safety rules and use of prescribed personal protection equipment is mandatory. The health hazards of the standards, reagents and samples are not entirely known so caution must be exercised in all cases.
- 4.2. Employees performing this procedure must be familiar with the Chemical Hygiene Plan (CHP), and the precautions stated on the appropriate Material Safety Data Sheets (MSDS).
- 4.3. Personal Protective Equipment Required: Safety Glasses, Labcoat, Gloves

**5. INTERFERENCES**

- 5.1. Contamination may occur when a low concentration sample is analyzed immediately after a high concentration sample.

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**SPECTROMETRY (GC/MS)**

- 5.1.1. If the autosampler is contaminated by a sample, analyze a blank. If the target compounds are not present in the blank then analysis may continue.
- 5.1.2. If a low concentration sample is analyzed immediately after a high concentration sample, reanalysis of the low concentration sample is necessary if the concentrations are less than 10x the reporting limit. Reanalysis is not necessary if the low concentration sample is N.D. for all target analytes at their reporting limit.
- 5.2. Contamination may also occur when a sample contains surfactants. Signs of surfactant are foaming and/or bubbling when the sample is purged. After a sample, which contains surfactants, is analyzed, rinse the system carefully. Look carefully for signs of carry over in the samples that are analyzed immediately after the surfactant sample.
- 5.3. The sample storage area must be free of organic solvent vapors. This is verified weekly with the use of refrigerator blanks analyzed by GCMS.

**6. SAMPLE HANDLING AND PRESERVATION**

- 6.1. Water Samples
  - 6.1.1. The holding time for water samples is fourteen (14) days from the date of collection. All samples must be preserved with HCl to a pH < 2.
    - 6.1.1.1. Using pH test strips, measure the pH of the sample after the sample aliquot is removed for analysis to ensure it was properly preserved.
    - 6.1.1.2. Document the pH on the run log. If the  $5 > \text{pH} > 8$ , document on the run log that the sample was analyzed though improperly preserved. Flag the results in ELEMENT with a 'P' qualifier as well as a 'pH' qualifier in which the actual pH is entered and notify the Project Manager.
    - 6.1.1.3. If the  $5 < \text{pH} < 8$ , document on the run log that the sample was analyzed though improperly preserved. Flag the results in ELEMENT with a 'P1' qualifier and notify the Project Manager.
  - 6.1.2. The unpreserved water must be analyzed within 7 days.
  - 6.1.3. All samples must be stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  prior to analysis.
  - 6.1.4. Water samples are submitted in 40 ml glass VOA vials with Teflon lined silicon septa screw caps in duplicate or triplicate. They should not have any air bubbles present in the vials when they are inverted. Flag the results with an 'HS' qualifier if any headspace is present and notify the project manager
- 6.2. Soil Samples
  - 6.2.1. For EPA Methods 5030B, the holding time for solid samples is fourteen (14) days from the date of collection.



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**SPECTROMETRY (GC/MS)**

- 6.2.2. EPA Method 5035, when using Encore sampler, states that soil samples must be either analyzed or extracted within 48 hrs. The holding time may be extended to 7 days by storing the samples in a freezer until the time of analysis. By extracting the sample in MeOH, the holding time may be further extended by 7 additional days from the from the sampling date.
- 6.2.3. Samples must be stored at  $4\pm 2^{\circ}\text{C}$  prior to analysis with the exception of EnCore-type samples which must be stored between  $-10$  to  $-20^{\circ}\text{C}$ .
- 6.2.4. Solid samples can be submitted in brass or stainless steel boring tubes, or zero-headspace sampling devices such as Encore<sup>TM</sup> sampling devices.
- 6.3. Air Samples
  - 6.3.1. The holding time for air samples is three days (72 hours) from the time of sample collection.
  - 6.3.2. The sample must be loaded on the autosampler within 72 hours of sampling. (The sample does not necessarily have to be analyzed within 72 hours of sample collection.)
  - 6.3.3. There is no preservation of air samples.
- 6.4. Notify the project manager immediately if the method holding time has been exceeded.

**7. PROCEDURE**

- 7.1. Standard Preparation
  - 7.1.1. Prepare the working standards, LCS standard, and the calibration standards before initial calibration. Initial calibrations are performed as needed. Prepare the internal standard/surrogate solution every two weeks, or as needed. All daughter solutions of the non-gases expire with the parent or at one month. Whichever comes first. Standards for the permanent gases are monitored frequently by comparison to the initial calibration curve. Fresh standards are prepared if this check exceeds 20% drift. Also refer to the SOP "Reagent and Standard Control and Documentation".
  - 7.1.2. For EVERY standard, enter the standard information into Element. Element will create a unique ID. Have the information peer-reviewed in Element prior to use. Place any standard Certificates of Analysis in a notebook and annotate with a reference to the Element standard ID. Store the solution in the freezer.
  - 7.1.3. Demonstrate that every new lot of MeOH (new lot number), received from the supplier is free of analytes by analyzing a blank of MeOH.
  - 7.1.4. Transfer the stock standards (2000  $\mu\text{g/ml}$ ) from their 1-ml ampule to 1-ml vials with PTFE-lined screw caps.

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SPECTROMETRY (GC/MS)**

- 7.1.4.1. Store the stock standards in the freezer for no longer than six months.
- 7.1.4.2. Replace the standards sooner if a change in response is observed.
- 7.1.4.3. Prepare the LCS standards in the same manner.
- 7.1.5. Work under a hood when handling stock standards containing high concentrations of toxic analytes. Note that the following compounds are classified as known or suspected carcinogens and should be treated as potential health hazards. Keep exposure to these chemicals to a minimum:
- 7.1.5.1. Benzene
- 7.1.5.2. Carbon tetrachloride
- 7.1.5.3. 1,4-Dichlorobenzene
- 7.1.5.4. 1,2-Dichloroethane
- 7.1.5.5. Hexachlorobutadiene
- 7.1.5.6. 1,1,2,2-Tetrachloroethane
- 7.1.5.7. 1,1,2-Trichloroethene
- 7.1.5.8. Chloroform
- 7.1.5.9. 1,2-Dibromoethane
- 7.1.5.10. Tetrachloroethene,
- 7.1.5.11. Trichloroethene
- 7.1.5.12. Vinyl chloride.
- 7.1.6. Treat all compounds, not just carcinogens, as potential health hazards.
- 7.1.7. Prepare the **internal standard/surrogate solution** at 25ml. (Makes 17 individual 1ml vials).

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
t-Butanol-d9	250ul	40,000ug/ml	400ug/ml	25ml
8260 Internal Standard	625ul	2,000ug/ml	50ug/ml	25ml
8260 Surrogate Standard	625ul	2,000ug/ml	50ug/ml	25ml

- 7.1.8. Prepare the **internal standard** (without surrogate).

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
t-Butanol-d9	10ul	40,000ug/ml	400ug/ml	1ml
8260 Internal Standard	25ul	2,000ug/ml	50ug/ml	1ml

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7.1.9. Prepare the **surrogate standard at 50 µg/ml**.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
8260 Surrogate Standard	25ul	2,000ug/ml	50ug/ml	1ml

7.1.10. Prepare the **surrogate standard at 200 ug/ml**.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
8260 Surrogate Standard	100ul	2,000ug/ml	200ug/ml	1ml

7.1.11. Prepare the **4-BFB Tuning Solution at 25 ug/ml**.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
BFB Tune	10ul	2,500ug/ml	25ug/ml	1ml

7.1.12. Prepare the **50 µg/ml 8260 working standard**. Prepare several vials. Set one aside to be used for initial calibrations and the others to be used for continuing calibration checks (CCVs).

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
524 Volatile Compound Mix	25ul	2,000ug/ml	50ug/ml	1ml
Ketones Mix	25ul	2,000ug/ml	50ug/ml	1ml
2-CEVE STD	25ul	2,000ug/ml	50ug/ml	1ml
8260 Oxygenates Mix	20ul	2,500ug/ml	50ug/ml	1ml
524 VOC Mix #1	25ul	2,000ug/ml	50ug/ml	1ml

7.1.13. Prepare the **TBA/Ethanol/VA/CS2 working standard (low level)**. Prepare several vials. Set one aside to be used for initial calibrations and the others to be used for continuing calibration checks (CCVs).

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Ethanol	25ul	20,000ug/ml	500ug/ml	1ml
Vinyl Acetate	25ul	2,000ug/ml	50ug/ml	1ml
Carbon Disulfide	50ul	1,000ug/ml	50ug/ml	1ml
Tert-Butyl Alcohol	12.5ul	20,000ug/ml	250ug/ml	1ml

7.1.14. Prepare the **TBA/Ethanol/VA/CS2 working standard (high level)**.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Ethanol	100ul	20,000ug/ml	2,000ug/ml	1ml
Vinyl Acetate	100ul	2,000ug/ml	200ug/ml	1ml

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Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Carbon Disulfide	200ul	1,000ug/ml	200ug/ml	1ml
Tert-Butyl Alcohol	50ul	20,000ug/ml	1,000ug/ml	1ml

7.1.15. Prepare the 5 µg/ml working standard

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
8260+K+Oxy ICV Working Standard	25ul	200ug/ml	5ug/ml	1ml
TBA/ETOH/CS2/VA Working Standard	25ul	1,000/2,000/200 ug/ml	25/50/5 ug/ml	1ml

7.1.16. Prepare the **Laboratory Control Sample (LCS) standard**. NOTE: This standard is also used as the spike for TPH MS/MSD. When entering this standard information into ELEMENT, include a TPH parent standard with an amount of 1ml entered. This will be used for ELEMENT calculation purposes only to calculate TPH for the MS/MSD.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Ketones Mix (2 <sup>nd</sup> source)	25ul	2,000ug/ml	50ug/ml	1ml
2-CEVE (2 <sup>nd</sup> source)	25ul	2,000ug/ml	50ug/ml	1ml
Oxygenates (2 <sup>nd</sup> source)	25ul	2,000ug/ml	50ug/ml	1ml
8260 VOC Mix for LCS	25ul	2,000ug/ml	50ug/ml	1ml

7.1.17. Prepare the **LCS standard-TBA/Ethanol/VA/CS2 only**.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Ethanol (2 <sup>nd</sup> source)	25ul	20,000ug/ml	500ug/ml	1ml
Vinyl Acetate (2 <sup>nd</sup> source)	25ul	2,000ug/ml	50ug/ml	1ml
Carbon Disulfide (2 <sup>nd</sup> source)	25ul	2,000ug/ml	50ug/ml	1ml
TBA (2 <sup>nd</sup> source)	125ul	2,000ug/ml	250ug/ml	1ml

7.1.18. Prepare the **Appendix ICAL standard at 50ug/ml**.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
1,2-Dibromotetrafluoroethane	25ul	2,000ug/ml	50ug/ml	1ml
Custom Halogenated Alkanes Plus Standard	25ul	2,000ug/ml	50ug/ml	1ml

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Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Custom Isopropanol	25ul	20,000ug/ml	500ug/ml	1 ml
Freon 113	25ul	2,000ug/ml	50ug/ml	1ml
Appendix II Volatiles Standard	25ul	2,000ug/ml	50ug/ml	1ml
		20,000ug/ml	500ug/ml	
Acrylonitrile	50ul	1,000ug/ml	50ug/ml	1ml

**7.1.19. Prepare the Appendix Acrolein ICAL standard at 50ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Acrolein	250ul	200ug/ml	50ug/ml	1 ml

**7.1.20. Prepare the Appendix 3 Freon ICAL standard at 50ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Appendix 3 Freon Standard	250ul	200ug/ml	50ug/ml	1ml

**7.1.21. Prepare the Appendix ICAL standard at 200ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
1,2-Dibromotetrafluoroethane	100ul	2,000ug/ml	200ug/ml	1ml
Custom Halogenated Alkanes Plus Standard	100ul	2,000ug/ml	200ug/ml	1ml
Custom Isopropanol	100ul	20,000ug/ml	200ug/ml	1ml
Freon 113	100ul	2,000ug/ml	200ug/ml	1ml
Appendix II Volatiles Standard	100ul	2,000ug/ml	200ug/ml	1ml
		20,000ug/ml	2,000ug/ml	
Acrylonitrile	200ul	1,000ug/ml	200ug/ml	1ml

**7.1.22. Prepare the Appendix Acrolein ICAL standard at 200ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Acrolein	1ml	200ug/ml	200ug/ml	1ml

**7.1.23. Prepare the Appendix 3 Freon ICAL standard at 200ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Appendix 3 Freon Standard	1ml	200ug/ml	200ug/ml	1ml

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7.1.24. Prepare the **Appendix ICAL standard at 5ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
8260 Appendix ICAL Standard	100ul	50ug/ml	5ug/ml	1ml

7.1.25. Prepare the **Appendix Acrolein ICAL standard at 5ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
8260 Appendix Acrolein ICAL Standard	100ul	50ug/ml	5ug/ml	1ml

7.1.26. Prepare the **Appendix 3 Freon ICAL standard at 5ug/ml.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
8260 Appendix 3 Freon ICAL Standard	100ul	50ug/ml	5ug/ml	1ml

7.1.27. Prepare the **8260 Appendix LCS.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Tert-Butyl Formate	50ul	1,000ug/ml	50ug/ml	1ml
Appendix II Standard	25ul	2,000ug/ml 20,000ug/ml	50ug/ml 500ug/ml	1ml
Iodomethane	50ul	1,000ug/ml	50ug/ml	1ml
Thiophene	50ul	1,000ug/ml	50ug/ml	1ml
1,2-Dichlorofluoroethane	50ul	1,000ug/ml	50ug/ml	1ml
1,2-Dibromotetrafluoroethane	50ul	1,000ug/ml	50ug/ml	1ml
2-chloro-1,3-butadiene	50ul	1,000ug/ml	50ug/ml	1ml
Freon 113	50ul	1,000ug/ml	50ug/ml	1ml

7.1.28. Prepare the **Appendix Acrolein LCS.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Acrolein	250ul	200ug/ml	50ug/ml	1ml

7.1.29. Prepare the **Appendix 3 Freon LCS.**

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Freon Mix 3-1	250ul	200ug/ml	50ug/ml	1ml

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7.1.30. Prepare the **LCS standard for methanol extractions** with each extraction batch. Vortex as you would a sample after all parent standards are added.

Parent Standard	Amount Added	Initial Concentration	Final Concentration	Final Volume MeOH
Oxygenates (2 <sup>nd</sup> source)	12.5ul	2,000ug/ml	2.5ug/ml	10ml
Ketones (2 <sup>nd</sup> source)	12.5ul	2,000ug/ml	2.5ug/ml	10ml
2-CEVE (2 <sup>nd</sup> source)	12.5ul	2,000ug/ml	2.5ug/ml	10ml
Ethanol (2 <sup>nd</sup> source)	12.5ul	20,000ug/ml	25ug/ml	10ml
Vinyl Acetate (2 <sup>nd</sup> source)	12.5ul	2,000ug/ml	2.5ug/ml	10ml
Carbon Disulfide (2 <sup>nd</sup> source)	12.5ul	2,000ug/ml	2.5ug/ml	10ml
8260 VOC Mix for LCS	12.5ul	2,000ug/ml	2.5ug/ml	10ml
Tert-Butyl Alcohol (2 <sup>nd</sup> source)	6.25ul	20,000ug/ml	12.5ug/ml	10ml
Surrogate Mix	12.5ul	2,000ug/ml	2.5ug/ml	10ml

NOTE 1: Information entered into ELEMENT is for calculation purposes only, as a true standard is not being created. Enter all of the parent standards listed in the above table with amounts for all being 0.05mls with the exception of TBA, which will be entered at 0.025mls. Also include a TPH parent standard with an amount of 2mls entered. This will be used for ELEMENT calculation purposes only to calculate TPH for the MS/MSD.

NOTE 2: The unique ID that ELEMENT creates for this “standard” can be reused for further extract batches as long as no new parent standards are opened. If new parent standards are opened, this “standard” must be edited to reflect this change and ELEMENT will create a new unique ID.

7.2. Calibration Standards

7.2.1. Prepare the calibration standards as specified in the following table. Check the working standard frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

7.2.2. 8260 Calibration with a 10-ml purge. (Calibration may be performed without the addition of the Appendix standard, Freon 3-1 standard and the Acrolein standard depending on the instrument.)

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Calib. Points	Internal Std./Surr Mix 50ug/ml		5ppm Standard 8260		5ppm Appen. Std.	5ppm Freon 3-1	5ppm Acro- lein
0.4ppb	5ul		0.8ul				
0.5ppb	5ul		1ul				
1ppb	5ul		2ul		2ul	2ul	2ul
2ppb	5ul		4ul		4ul	4ul	4ul

Calib. Points	Internal Std (50ug/ml) (400ug/ml TBA-d9)	Surrogate (50ug/ml)	50ppm 8260 Standard	250/500 ppm TBA/ETOH	50ppm Appen. Std.	50ppm Freon 3-1	50ppm Acro- lein
5ppb	5ul	1ul	1ul	1ul	1ul	1ul	1ul
10ppb	5ul	2ul	2ul	2ul	2ul	2ul	2ul
25ppb	5ul	5ul	5ul	5ul	5ul	5ul	5ul

Calib. Points	Internal Std (50ug/ml) (400ug/ml TBA-d9)	Surrogate (200ug/ml)	200ppm 8260 Standard	1000/2000 ppm TBA/ETOH	200ppm Appen. Std.	200ppm Freon 3-1	200ppm Acro- lein
50ppb	5ul	2.5ul	2.5ul	2.5ul	2.5ul	2.5ul	2.5ul
100ppb	5ul	5ul	5ul	5ul	5ul	5ul	5ul
200ppb	5ul	10ul	10ul	10ul	10ul	10ul	10ul

7.3. Initial Calibration

7.3.1. BFB Tune

7.3.1.1. Perform a BFB tune on the mass spectrometer prior to performing the initial calibration.

7.3.1.2. Tune the mass spectrometer by directly injecting 50ng (2ul of the 25ug/ml tuning solution) of BFB.

7.3.1.3. The mass spectrometer must produce a mass spectrum that meets all the relative ion abundance criteria for BFB. See Appendix 1. The mass spectrum may be acquired as follows (pre-set in the software):

7.3.1.3.1. Three scans are acquired and averaged (the peak apex scan and the scans immediately preceding and following the apex).

7.3.1.3.2. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB.



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- 7.3.1.3.3. Do not background subtract part of the BFB peak.
- 7.3.1.4. If the BFB mass spectrum does not meet all the criteria, then replace the septa, bake the column and try the BFB tune again.
  - 7.3.1.4.1. If BFB direct inject fails 6X then retune instrument with BFB tune.
- 7.3.2. Calibration Standards
  - 7.3.2.1. Perform initial calibrations on an as needed basis and after major instrument maintenance. Analyze a minimum of five concentrations to establish the initial calibration curve.
  - 7.3.2.2. The surrogates are calibrated at a minimum of 5 levels in conjunction with the calibration standards.
  - 7.3.2.3. Analyze the initial calibration curve in the same manner as samples.
  - 7.3.2.4. Attach a heater jacket to each standard to increase the purge efficiency for the ketones and other water soluble and late eluting compounds.
  - 7.3.2.5. Heat the standards to  $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$  during the purge.
- 7.3.3. SPCCs
  - 7.3.3.1. The System Performance Check Compounds (SPCCs) are analyzed with the initial calibration curve, and must meet acceptance criteria before the calibration curve is used.
  - 7.3.3.2. The SPCCs are checked for a minimum average relative response factor.
  - 7.3.3.3. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene.
  - 7.3.3.4. The minimum acceptable average response factor (RF) for these compounds should be 0.30 for Chlorobenzene and 1,1,2,2-Tetrachloroethane; 0.10 for Bromoform, Chloromethane and 1,1-Dichloroethane.
- 7.3.4. CCCs
  - 7.3.4.1. The Calibration Check Compounds (CCCs) in the initial calibration curve and must meet acceptance criteria before the calibration curve can be used.
  - 7.3.4.2. The %RSD for the average response factors (RFs) for each CCCs must be  $\leq 30\%$ .
  - 7.3.4.3. The CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride
- 7.3.5. Average Response Factor

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- 7.3.5.1. The primary calibration criteria involve the use of average Response factors (RFs). The RFs from the initial calibration curve should have an RSD  $\leq$  15% for all compounds, except for the Calibration Check Compounds (CCCs).
- 7.3.5.2. Due to poor purge efficiency, the % RSD for Alcohols and Ketones must be  $\leq$  30%.
  - 7.3.5.2.1. The Average % RSD must meet the method required +15% criteria.
  - 7.3.5.2.2. The analytical reports must have a disclaimer in the case narrative to outline the 30% criteria for the alcohols and ketones.
  - 7.3.5.2.3. If one or more of the compounds, other than the CCCs and ketones, has a %RSD  $>$ 15%, generate a calibration curve and follow the steps outlined below.
- 7.3.6. Linear and Quadratic Curves
  - 7.3.6.1. A first order (linear) or second order (quadratic) regression may be used for quantitation (not forced through the origin).
    - 7.3.6.1.1. Print out a linear curve (5pts minimum) and a quadratic curve (6 pts minimum).
    - 7.3.6.1.2. The Coefficient of Determination ( $r^2$ ) must be  $>$  0.99 (Correlation Coefficient ( $r$ )  $>$  0.995) for the curve to be acceptable. If  $r^2$  is  $<$  0.99 then the instrument must be recalibrated for that compound.
    - 7.3.6.1.3. If the software does not calculate  $r^2$  for quadratic curves, use the linear curve  $r^2$  for validation.
    - 7.3.6.1.4. If  $r^2$  for the linear curve is at least 0.99 (rounded to 2 sig. figs.) then use the curve that gives the y intercept closest to zero (where applicable).
  - 7.3.6.2. Print out any calibration curves to evaluate linearity and obtain the correlation coefficients.
  - 7.3.6.3. Since the curve is not forced through the origin, inaccuracies may be present near the low end of the curve or negative values may be obtained at the reporting limit.
    - 7.3.6.3.1. Negative or "below cal" values occur when the regression calculation of "best fit" does not pass through the lowest calibration standard and the y- intercept falls above the y coordinate (area count) of the low standard of the curve.

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7.3.6.3.2. The cause is sometimes related to the slight bending in the curve at higher analyte concentrations and a 1<sup>st</sup> order curve is used. If this is the case the a 2<sup>nd</sup> order fit will take care of the problem.

7.3.6.3.3. Because of the high potential for inaccuracies or negative numbers at the low end of the curve, positive results below the low calibration standard may not be quantitated and may not be reported for linear regression or second order curves. **(No “J” flags below the low calibration standard when a calibration curve is used.)**

7.3.6.3.4. If the RF > 15% but < 50% and the total average (average of all RFs) is < 15%, then use the RF and requantitate “J” flags.

7.3.6.4. When evaluating results near the RL review them carefully to ensure they make sense (i.e. no negative values or positive values with areas below the lowest standard). If a result is questionable, the sample should be re-analyzed on another instrument or the result reported as estimated.

7.3.7. As an alternative to generating a calibration curve, if the compounds > 15% RSD are known historically to be not detected (ND) or the calibration curve does not meet the acceptance criteria, calculate the average RSD for all compounds and if < 15% quantitate using the average RF. Report data with a form identifying which compounds did not meet the 15% RSD criteria.

7.3.7.1. If the % RSD is > 50% for any compound re-evaluate the standard concentrations and perform any necessary maintenance and recalibrate the instrument.

7.4. Calibration Verification

7.4.1. Calibrations are initially verified with the first daily LCS analyzed after the calibration. The LCS is prepared from a second source.

7.4.2. All compounds in the ICAL must be within LCS control limits.

7.4.3. If any compounds do not meet LCS limits, no results may be reported for these compounds until a passing LCS is re-analyzed or the instrument is re-calibrated and a new passing LCS is analyzed. Exception: If the LCS is out high, ND results for the high compound may be reported and must be qualified “L.”

7.5. Continuing Calibration

7.5.1. Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift before any samples are analyzed:

7.5.1.1. The BFB tuning criteria in Appendix 1 must be achieved.

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- 7.5.1.2. Analyze a midpoint calibration standard (CCV). SPCC and CCC criteria must be met.
- 7.5.1.3. Analyze a method blank to ensure that the total system is free of contaminants.
- 7.5.2. Prepare the CCV standard.
  - 7.5.2.1. Pour approximately 12 ml of ultrapure water into the 10ml syringe.
  - 7.5.2.2. Adjust the water in the syringe to 10-ml.
  - 7.5.2.3. Purge an empty VOA vial with nitrogen gas to minimize laboratory contamination.
  - 7.5.2.4. Inject the syringe contents into the nitrogen purged voa vial.
  - 7.5.2.5. Add 5 $\mu$ l of the 50 $\mu$ g/ml concentration internal standard/surrogate solution to the ultrapure water in the VOA vial.
  - 7.5.2.6. Add 5  $\mu$ l of the 50 $\mu$ g/ml CCV/LCS standard to the ultrapure water in the VOA vial.
- 7.5.3. Check that the performance criteria of the SPCCs and CCCs are met prior to sample analysis.
  - 7.5.3.1. The SPCCs must meet the same acceptance criteria as defined in the initial calibration.
  - 7.5.3.2. The CCCs must have a percent difference  $\leq$  20% from the initial calibration.
  - 7.5.3.3. Calculate acceptance limits for the non-CCC analytes by performing a statistical evaluation of the %Difference of 20 Midpoint (CCV) standards. Use + 3 SD or + 20%, whichever is greater, for the acceptance limits.
  - 7.5.3.4. The non-CCC analytes should have an average percent difference within the acceptance limits previously established. This would include the SPCCs.
    - 7.5.3.4.1. If the CCV non-CCC result is greater than the high acceptance limit of the expected value and all samples are ND for the compound then report the results with a "C" qualifier.
    - 7.5.3.4.2. If the LCS result is below the acceptance limit, flag the result and report with a CAR and a GCMS calibration Check Criteria form.

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7.5.3.4.3. If any analyte exceeds the criteria, an average percent recovery < 20% may be used to accept the analytical run. A GCMS calibration Check Criteria form must be utilized if this condition exists. (if samples are ND and CCV is biased high, the Check Criteria form is not required.

7.5.3.5. If the SPCCs and/or CCCs fail, reanalyze the standard. Need 2 consecutive CCV to pass after 2 fail. If they pass, continue with analysis. If they fail again, take corrective action and recalibrate the instrument.

7.5.4. Determine that the retention times for any internal standard have not changed by more than 30 seconds from the midpoint standard measured during the most recent initial calibration.

7.5.5. Determine that the absolute areas of the quantitation ions of the internal standards in the CCV check standard have not changed by a factor of two (-50% to +100%) from the IS area from the midpoint standard measured during the most recent initial calibration (5 pt). If the retention times or areas have changed by more than these amounts, perform necessary maintenance and recalibrate.

7.5.5.1. For TBA-d9 internal standard is not monitored as (-50%-100%). TBA-d9 is used for the quantitation of TBA and ethanol due to the stability in water. TBA-d9 is temperature dependent and also monitors the behavior of TBA and ethanol in the system.

7.6. Analysis

7.6.1. Perform a BFB tune and analyze a CCV check standard and a method blank before analyzing any samples.

7.6.2. All sample and standard solutions must be allowed to warm to ambient temperature before analysis.

7.6.3. Water Samples (Refer to Volatiles Sample Preparation SOP).

7.6.4. Low Level Soil Samples – From a Boring Tube (Refer to Volatiles Sample Preparation SOP).

7.6.5. Dilution of High Level Soil Samples (Refer to Volatiles Sample Preparation SOP).

7.6.6. Dilution of soil samples containing high levels of analytes – From an Encore™ sampling device (Refer to Volatiles Sample Preparation SOP).

7.6.7. Air samples

7.6.7.1. Analyze an air sample by pouring approximately 12 ml of nano-pure water into a 10 ml syringe.

7.6.7.2. Adjust the water to 10 ml.

7.6.7.3. Inject the syringe contents into a nitrogen purged VOA vial.

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7.6.7.4. Add 5 uL of the 50 ug/ml internal standard/surrogate solution to the water in the VOA vial.

7.6.7.5. Using a gas tight syringe measure 5cc of the air sample.

7.6.7.5.1. For dilutions, use a lower volume of the air sample.

7.6.7.6. Add the measured volume of air sample to the VOA vial through the luer lock valve.

7.6.7.7. Dilute and re-analyze any sample that exceeds the highest calibration standard.

7.7. Compound Identification

7.7.1. For most analytes, at least two Mass Ions must be present on the mass spectra for qualitative ID and quantitation purposes.

7.7.1.1. As per EPA 8260B guidelines, the instrument data system is used to meet method criteria for qualitative identification.

7.7.2. If the instrument can only detect one ion, qualitative and quantitative ID will be based on Retention time of the target peak and the presence of the highest mass (Primary) ion. Examples would include Tert-Butyl Alcohol (TBA). In these cases, flag the sample result with the qualifier "ID".

7.7.3. If the secondary ion is present but the primary is not, then the compound will not be identified.

7.8. Tentatively Identified Compounds (TICs)

7.8.1. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

7.8.2. Use the following guidelines for making tentative identifications:

- (1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within  $\pm 20\%$ . (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

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- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

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- 7.8.3. The concentration of any non-target analytes identified in the sample should be estimated. The same formulae as calibrated analytes should be used with the following modifications: The areas  $A_x$  and  $A_{is}$  should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.
- 7.8.4. The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.
- 7.8.5. For the GC/MS methods, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs).
- 7.8.6. In general Reporting limits cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.
- 7.8.7. TICs that meet the above identification criteria (1-5) at 10% area of the IS: The RL would be 10% of the concentration of the internal standard used for quantitation. (e.g. 2.5 ug/L for 8260B, 4.0 ug/L for 8270C). In general TIC RLs will be set at a level approximately 5x the level of the poorest performer in the analysis.
- 7.8.8. If a compound meets the TIC criteria, the reporting limit will reflect the ratio between the TIC and the IS or 5x the level of the poorest performer whichever is lower
- 7.9. Dilutions
- 7.9.1. The laboratory reports samples from a single dilution as a standard practice. The dilution used is:
- 7.9.1.1. The minimum dilution required to achieve all target compounds within the calibration range. Or
- 7.9.1.2. The minimum dilution to reduce background matrix effects to a level of no interference effects.
- 7.9.1.3. Clients may request that we report results from more than one dilution in order to minimize the increase of reporting limits. In other words, reporting trace levels of analytes in the presence of high concentrations of other target analytes (such as low BTEX levels in the presence of high MTBE contamination).
- 7.9.2. Prior to loading samples on the instrument, review the worklist for client specific information and search the ELEMENT database for historical information on the project site.



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- 7.9.3. Samples are initially analyzed with no dilution unless one or more of the following conditions exist:
  - 7.9.3.1. The samples are neat material (a.k.a. product samples)
  - 7.9.3.2. Multi-phasic samples
  - 7.9.3.3. The samples emit a strong odor (strong enough to smell without placing the sample up to the nose).
  - 7.9.3.4. Colored samples
  - 7.9.3.5. The sample obviously contains surfactants
  - 7.9.3.6. There is historical site data available to the lab that indicates high target analyte concentrations
  - 7.9.3.7. There is a high field PID reading shown on the COC that was recorded by the field sampler
  - 7.9.3.8. The client supplied information to the lab indicating matrix issues or high concentrations of analytes.
  
- 7.9.4. If a dilution is required, report all results from the minimum dilution required to obtain the highest concentration analyte results within the instrument calibration range unless multiple dilutions are requested by the client.
  
- 7.9.5. Multiple Dilutions
  - 7.9.5.1. All concentrations listed below are estimates only and may vary by analyte and instrument depending on sensitivities.
  - 7.9.5.2. The number and magnitude of dilutions should be kept to a minimum for the benefit of the client and for maximum productivity.
  - 7.9.5.3. All compounds (except those past the calibration range) may be reported from a 1x dilution if concentrations are <500 ppb for any individual compound.
    - 7.9.5.3.1. Reshots will be at the minimum dilution necessary to get the highest level compound(s) within the calibration range (the highest compound to approximately mid range).
  - 7.9.5.4. If individual compounds exceed 500 ppb in a sample:
    - 7.9.5.4.1. Samples immediately following must be reanalyzed if the same compounds are detected at a level <10x the lowest ICAL concentration.
    - 7.9.5.4.2. All compounds will be reported if possible based on acceptable internal standard and surrogate recovery.

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7.9.5.4.3.If the sample must be reshot far any reason it will be shot at a dilution(s) estimated from the information on the attached table.

7.9.5.4.4.Analysis of the sample in subsequent sampling events will be initially diluted based on updated historical information.

7.9.6. Examples

7.9.6.1. If MTBE is present in a sample analyzed by EPA 8260B at 500 ug/L, the sample dilution factor required to report MTBE will be 4x or 5x.

7.9.6.1.1.If the concentration of benzene is critical to the project objectives, a client may request multiple dilutions of the sample and we will also analyze the sample without dilution to report the other compounds.

7.9.6.1.2.If MTBE is present at 2500 ug/L, the lowest dilution the laboratory can achieve for the other compounds of interest is a dilution of 5. Analysis of the sample without dilution will result in significant MTBE contamination of the next few sample analyses and damage to the analytical instrumentation.

7.9.6.1.3.Refer to the accompanying chart for current examples of maximum concentrations of compounds that can be present before dilutions are required for all other compounds in the samples.

7.9.6.1.4.Future regulations that push the reporting limits lower will impact the laboratory's ability to analyze samples of the same concentrations without dilution.

**MINIMUM POSSIBLE DILUTION FACTORS AVAILABLE IN MULTIPLE  
DILUTIONS**

Analyte	Analyte Concentration				
	<500ppb	1000ppb	2500ppb	5000ppb	10,000ppb
<b>BTEX</b>					
Benzene	1	2	5	10	20
Toluene	1	2	5	10	20
Ethylbenzene	1	2	5	10	20
Xylenes	1	4	10	20	40
1,2-DCA	1	2	5	10	20
Styrene	1	4	10	20	40
Naphthalene	1	4	10	20	40
<b>Solvents</b>					

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Analyte	Analyte Concentration				
	<500ppb	1000ppb	2500ppb	5000ppb	10,000ppb
TCE	1	4	10	20	40
PCE	1	4	10	20	40
1,1-DCE	1	2	5	10	20
Carbon Tetrachloride	1	4	10	20	40
TCA	1	2	5	10	20
Methylene Chloride	1	2	5	10	20
<b>Oxygenates</b>					
DIPE	1	2	5	10	20
TAME	1	2	5	10	20
ETBE	1	2	5	10	20
TBA	1	1	1	2	5
MTBE	1	2	5	10	20
Ethanol	1	1	1	2	5
<b>Gases</b>					
Chloromethane	1	1	5	10	20
Vinyl Chloride	1	2	5	10	20
Bromomethane	1	1	5	10	20
Chloroethene	1	1	5	10	20
<b>Miscellaneous</b>					
IPA	1	1	1	2	5
Acetone	1	1	5	10	20
Freon 113	1	1	5	10	20

Note: Product samples and samples containing percent levels of single or multiple analytes will be heavily diluted and multiple dilutions in these cases may not yield desired RLs.

7.10. Instrument Conditions

7.10.1. The following are general instrument conditions. These may vary slightly between instruments, or because of necessary instrument maintenance (e.g. column trimming) or because of column age.

7.10.2. Column: DBVRX 60mm x 250µm x 1.4 µm

7.10.3. Carrier gas (he) flow rate: 1.2 ml/min

7.10.4. Purge and Trap Settings

Step	Time (minutes)	Temp (°C)
Pre-heat	1	40

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Step	Time (minutes)	Temp (°C)
Purge	11	40
Desorb	2	190
Bake	10	210
Valve	--	120
Transfer line	--	110
External Heater	--	110

- 7.10.5. Initial temperature: 40 °C, hold for 3 minutes
- 7.10.6. Temperature program: 12 °C/min to 70 °C, hold for 1 minute  
10 °C/min to 190 °C, hold for 0 minute  
25 °C/min to 230°C, hold for 3.5 minutes
- 7.10.7. Final temperature: 230 °C
- 7.10.8. Column Bake out: overnight at 210 °C
- 7.10.9. Injector temperature: 200 °C

7.11. Preventative Maintenance

- 7.11.1. Record all performed maintenance in the instrument maintenance logbook.
- 7.11.2. Replace OI #10 traps as needed.
- 7.11.3. Replace injector septa and deactivate liners when necessary.
- 7.11.4. Clean the ion source and replace filaments when necessary.
- 7.11.5. Back-up all data monthly.
- 7.11.6. Fill a 20 ml plastic syringe with methanol and flush the purge needle and purge valve whenever needed (after high level or surfactant containing samples).
- 7.11.7. Change the oil in the foreline pump twice per year and record on the maintenance schedule posted on the instrument.
- 7.11.8. Check the diffusion pump oil at least annually and replace as necessary and record on the maintenance schedule posted on the instrument.
- 7.11.9. Replace the carrier gas trap at least annually and record on the maintenance schedule posted on the instrument.

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7.11.10. If an instrument is unusable or has limitation to its use (bad port, bad heater jacket, not usable for ketones, not for low level samples, etc) , it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution and verification of proper operation into the instrument maintenance logbook.

**8. QUALITY CONTROL**

8.1. Method Blank

8.1.1. Analyze a method blank after each CCV check standard every 12 hours, and prior to sample analysis, with every batch of 20 samples or less. and after a highly concentrated sample as a cross-contamination check. Prepare a method blank with 10-ml of ultrapure water. Add 5 µl of the 50 µg/ml concentration internal standard/surrogate solution to the ultrapure water.

8.1.1.1. The method blank results must be below the reporting limit (RL). If detection above the RL is observed, and associated samples detect the same compound, flag results with "B". Samples containing the compound(s) of contamination must be reanalyzed if they are < 20x the level found in the blank.

8.1.1.2. If data are being reported down to the MDL and if there are values in the blank above the MDL, the blank and data shall be flagged with a "J".

8.1.1.3. A methanol blank is analyzed whenever methanol extracts are prepared and analyzed. The methanol must be free of contaminants below the reporting limit.

8.1.2. If there is a positive hit in a Trip Blank, verify if there are any positive hits for the compound(s) in the associated project samples. Notify the project manager when reporting the data if there are any affected samples.

8.2. Laboratory Control Sample (LCS)

8.2.1. Analyze a LCS with every batch of 20 samples or less. Prepare the LCS by injecting 5 µl of the internal standard/surrogate solution to 10-ml of ultrapure water. Add 5 µl of the 50 µg/ml concentration LCS standard.

8.2.2. The acceptance limits for the LCS are evaluated semi-annually by in-house statistical analysis. If any reported LCS compound exceeds the pre-established limits:

8.2.2.1. If the LCS is out above the acceptance limits and the sample results are ND, fill out a CAR and report the data with an 'L' qualifier.

8.2.2.2. If the LCS is out of the acceptance limits and the sample results are positive, reanalyze the sample with acceptable QC.

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8.2.2.3. If the LCS is out below the acceptance limits and the sample results are ND, reanalyze once. If still out, reprepare a fresh LCS solution and reanalyze. If the LCS fails twice, two consecutive LCS standards must pass.

8.2.2.4. A LCS is prepared in Methanol for MeOH soil extraction batches. Add 100  $\mu$ l to 10 ml of purged DI water, add internal standard and analyze as a sample. Follow the acceptance criteria and corrective actions listed above.

8.3. Surrogates

8.3.1. Surrogates are added to each sample, Calibration check method blank, LCS, MS and MSD. The surrogate recoveries must fall within the acceptance limits evaluated semi-annually by in-house statistical analysis.

8.3.2. If any surrogates are outside of the acceptance limits, determine the cause of the problem and take corrective action.

8.3.2.1. If the surrogate is out due to sample matrix, note this information on the results when reporting by flagging the results with a 'Z' qualifier.

8.3.2.2. If the cause is not due to obvious chromatographic matrix interference, the sample must be reanalyzed to confirm matrix effects.

8.4. Matrix Spike (MS) and MS Duplicate (MSD)

8.4.1. Analyze a matrix spike/matrix spike duplicate with every batch of twenty samples, or less.

8.4.2. Add 5  $\mu$ l of the 50  $\mu$ g/ml concentration LCS standard to two separate aliquots of a sample and process the same as the other samples.

8.4.3. The acceptance limits for the MS/MSD are evaluated semi-annually by in-house statistical analysis.

8.4.3.1. If the MS/MSD are outside of the acceptance limits due to matrix effects flag the MS/MSD with the appropriate 'M' qualifier.

8.4.3.2. If the MS/MSD are outside of the acceptance limits due to instrument problems or due to analyst error, re-analyze the MS/MSD if possible.

8.5. Internal Standards

8.5.1. For every sample, determine that the retention times for any internal standard have not changed by more than 30 seconds from the midpoint level standard of the most recent initial calibration.

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- 8.5.2. Determine that the absolute areas of the quantitation ions of the internal standards in each sample have not changed by a factor of two (-50% to +100%) from the IS area measured from the current daily midpoint check.
- 8.5.3. If the retention times or areas have changed by more than these amounts, reanalyze the sample.
- 8.5.4. The continuing calibration verification retention times and areas must be compared to the mid-point standard level of the most recent initial calibration.
- 8.6. Calculations
- 8.6.1. Response Factors (RFs).
- 8.6.1.1. Response Factor:  $RF = \frac{(R_A)(C_{IS})}{(C_A)(R_{IS})}$
- $R_A$ =analyte response  
 $C_A$ =analyte concentration  
 $R_{IS}$ =internal standard response  
 $C_{IS}$ =internal standard concentration  
RF=response factor

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8.6.1.2. Waters

$$C_f = C_i \times PF \times DF$$

$C_f$  = Final concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$

$C_i$  = Concentration in  $\mu\text{g/L}$  from instrument

PF = Preparation Factor

DF = Any additional Dilution Factor

8.6.1.3. Low Level Soils

$$C_f = C_i \times DF \times CF$$

$C_f$  = Final concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$

$C_i$  = Concentration in  $\mu\text{g/L}$  from instrument

DF = Dilution Factor (5g / wt of soil purged, in g)

CF = Calibration Factor (2x soils)

8.6.1.4. High Level Soils (MeOH extracts)

$$C_f = C_i \times PF \times DF$$

$C_f$  = Final concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$

$C_i$  = Concentration in  $\mu\text{g/L}$  from instrument

PF = Prep Factor (Vol of MeOH in ml / Weight of soil, in g)

DF = Dilution Factor (10 ml / actual volume injected, in ml)

8.6.1.5. Surrogate Spike Results in High Level Soils (MeOH extracts)

$$C_f = C_i \times SF \times DF$$

$C_f$  = Final concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$

$C_i$  = Concentration in  $\mu\text{g/L}$  from instrument

SF = Surrogate Factor (Final Prep Vol of MeOH in ml / 10 ml)

DF = Dilution Factor (100  $\mu\text{l}$  / actual volume injected, in  $\mu\text{l}$ )

8.6.1.6. Determine the % Difference of the CCV using the following equation:

$$\% \text{ Difference} = \frac{[\text{Apparent conc. } (\mu\text{g/l}) - \text{True conc. } (\mu\text{g/L})] \times 100}{\text{True conc. } (\mu\text{g/l})}$$

8.6.1.7. Determine the % recovery for the LCS, Surrogates, and MS/MSD as follows:



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$$\% \text{ Recovery} = \frac{(S_p - S)}{S_a} \times 100 \qquad \text{RPD} = \frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100$$

Where: Sp = Spike result  
S = Sample result (LCS and Surrogate = 0)  
Sa = Spike amount  
R1 = Conc. of MS  
R2 = Conc. of MSD

**9. PAPERWORK FLOW**

9.1. Daily

- 9.1.1. Query ELEMENT for the desired analysis codes.
- 9.1.2. Compare the worklist with the previous day's Sample Track Sheets to determine which sample numbers to cross off. Cross off the sample numbers of those analyses that have been completed or are currently being analyzed. Write the date analyzed and GCMS # beside the crossed out line.
- 9.1.3. Complete the Sample Track Sheet and instrument logbook as each sample is loaded onto the autosampler. Use the Comments section to note whether or not a sample is a confirmation, a re-analysis, or a rush. Also include a dilution amount or any other important information in the Comments section.
- 9.1.4. Record the appropriate analyst (load, run, spectra) and check the method name on the sequence files.
- 9.1.5. All raw data must undergo second level review. All raw data must be initialed and dated.
- 9.1.6. Reports and their corresponding chromatograms automatically print out as each sample is analyzed. Perform a manual integration when a chromatogram has a co-elution or interferences. All manual integrations must undergo second level review. Follow SOP titled "Manual Integrations".
- 9.1.7. Print out the mass spectra that correspond to positive results on the quantitation report. Compare these spectra with the calibration spectra. If there is no spectral match (NSM), draw a line through the result on the report and write NSM beside it. Initial and date the first page of the quantitation report for each sample. Also, the code WRT, which stands for wrong retention time can be used if applicable.
- 9.1.8. Staple behind each chromatogram, its corresponding quantitation report and mass spectra. Match the chromatograms, quantitation reports, and spectra to their corresponding Sample Track Sheets and file them in the cabinet by ascending date and ascending gas chromatograph/mass spectrometer number.

9.2. Monthly

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9.2.1. Print initial calibrations and keep them on file for each instrument.

9.3. As Needed

9.3.1. Document on the Daily Log Summary and sample results whenever the sample matrix interferes with the recovery of the surrogate, internal standard and/or MS/MSD.

9.3.2. Document any non-matrix related analytical problems or variances by initiating a Corrective Action Report. Keep a copy with the data package,

9.3.3. Complete an instrument maintenance log for each instrument and include the date that the instrument became inoperable. Also include the steps taken to fix the instrument, replacement parts, the initials of the analyst/technical specialist (or the name of the service company that performed the maintenance), and the date that the instrument became operable again.

9.3.4. Fill out a GCMS Initial Calibration Criteria form whenever any compound exceeds the ICAL criteria. Include a copy of the form with each data set affected.

**10. METHOD PERFORMANCE**

10.1. Perform a method detection limit (MDL) study initially and after extensive instrument maintenance or a significant change in the method. An MDL study is accomplished by analyzing seven replicates of the lowest calibration standard and multiplying the SD by 3.143.

10.2. Perform retention time window studies at the same time as the MDL study.

10.2.1. Use the CCV check standard from the three previous days to determine the retention time windows.

10.3. Perform an Initial Demonstration of Capability (IDOC) before performing analyses by analyzing 4 LCS samples with an average recovery, which meets the in-house acceptance limits. If the average does not meet the requirement identify the problem and repeat the process.

10.4. See attached analysis codes for information regarding control limits, reporting limits, and method detection limits.

**11. POLLUTION PREVENTION**

11.1. Waste Minimization: N/A as method is performed with recommended sample and reagent volumes.

**12. WASTE MANAGEMENT**

12.1. See Waste Management Section of the Laboratory Safety Plan (LSP).

12.2. Preserved samples neutralized prior to disposal.

12.3. Unused standards are drummed and shipped out for incineration.

**13. METHOD REFERENCES**

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- 13.1. EPA Method 8260B, EPA SW-846 Update III, December 1996
- 13.2. EPA Method 624 - Purgeables, 40 CFR Appendix A to Part 136, 1996
- 13.3. EPA Method 8000B, EPA SW-846 Update III, December 1996
- 13.4. EPA Method 5030A, EPA SW-846 Update I, July 1992
- 13.5. EPA Method 5030B, EPA SW-846 Update III, December 1996
- 13.6. EPA Method 5035, EPA SW-846 Update III, December 1996
- 13.7. EPA Region IX Memo "Regional Interim Policy for Determination of Volatile Organic Compound Concentrations in Soil and Solid Matrices," June 23, 1999.

**14. APPENDICES**

- 14.1. BFB Tune Abundance Criteria (8260B, Table 4)
- 14.2. Characteristic Ions for Purgeable Organic Compounds (8260B, Table 5)

**15. REFERENCE VARIANCES**

- 15.1. Due to poor response for the primary ions, the secondary ions are used for the quantitation of the following ketones. The primary ion must be present, however, for the compound to be positively identified. The following table lists the ketones with their primary and secondary ions used for quantitation.

Compound	Primary Ion	Secondary Ion
Acetone	58	43
2-Butanone	72	43
4-Methyl-2-pentanone	100	43

- 15.2. Relative intensities of characteristic ions are evaluated at  $\pm 20\%$  of the reference standard rather than 30% as listed in 8260B. This meets 624 method criteria.
- 15.3. In order to add confidence to the non-CCC analytes' quantitation, though not specified in the method, control limits are generated for the non-CCC compounds based on  $\pm 3$  SD of at least 20 CCVs. Acceptance limits are based on either 20% or the calculated limits whichever is greater.
- 15.4. In order to meet lower detection limits, a 10 mL purge volume is used instead of 5ml.
- 15.5. Because of the low purge efficiency of Alcohols and Ketones a % RPD requirement of 30% (same as CCCs) is used for these compounds. The method criteria of the average % RSD within  $\pm 15\%$  must still be met and the clients must be notified of the criteria in the final report.
- 15.6. Variances for EPA 624

**NOTE:** The laboratory has merged the method 8260B and 624. If 8260B criteria are stricter than those listed in 624, 8260B limits will generally be adhered to and are not addressed below as variances.

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- 15.6.1. In order to meet lower detection limits, a 10 mL purge volume is used instead of 5ml.
- 15.6.2. The use of a heated purge at  $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$  increases the purge efficiency for the ketones and other water soluble and late eluting compounds.
- 15.6.3. Since there are no specific IS acceptance limits, additional acceptance limits have been added based on EPA 8260B.
- 15.6.4. The concentrations of surrogate and internal standard are at 25 ug/L rather than 30 ug/L as listed in the method.
- 15.6.5. The concentration of the calibration verification and laboratory control sample are at 25ug/L rather than 20 ug/L as listed in the method.
- 15.6.6. Dibromofluoromethane is used as third surrogate. This compound is not listed in the table of suggested surrogates for EPA method 624.

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STANDARD OPERATING PROCEDURE  
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS  
SPECTROMETRY (GC/MS)**

**Table 14-1: BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA**

M/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS**  
**SPECTROMETRY (GC/MS)**

**Table 14-2: Characteristic Ions**

TABLE 5  
CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	78	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	78	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
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**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS**  
**SPECTROMETRY (GC/MS)**

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d <sub>4</sub>	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141

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**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS**  
**SPECTROMETRY (GC/MS)**

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
$\beta$ -Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d <sub>6</sub>	84	83
Bromobenzene-d <sub>5</sub>	82	162
Bromochloromethane-d <sub>2</sub>	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d <sub>5</sub>	117	
1,4-Dichlorobenzene-d <sub>4</sub>	152	115, 150
1,1,2-Trichloroethane-d <sub>3</sub>	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d <sub>1</sub>	84	
Dibromofluoromethane	113	

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**TESTAMERICA-IRVINE  
STANDARD OPERATING PROCEDURE  
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS  
SPECTROMETRY (GC/MS)**

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Dichloroethane-d <sub>2</sub>	102	
Toluene-d <sub>8</sub>	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

\* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

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## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>624+Std add-ons (WP) in Water (EPA 624)</b>								
Preservation:4 C, HCL								
Container:40 mL Voa Vial								
Amount Required:3 VOA								
Hold Time:14 days								
Acetone	4.5	10 ug/l			20 - 150	35	30 - 140	30
Benzene	0.28	2.0 ug/l			65 - 125	20	70 - 120	20
Bromodichloromethane	0.30	2.0 ug/l			70 - 135	20	70 - 135	20
Bromoform	0.40	2.0 ug/l			55 - 135	25	55 - 130	25
Bromomethane	0.42	5.0 ug/l			55 - 145	25	65 - 140	20
2-Butanone (MEK)	4.7	10 ug/l			30 - 145	40	40 - 140	35
Carbon Disulfide	0.48	5.0 ug/l			40 - 140	20	50 - 130	20
Carbon tetrachloride	0.28	5.0 ug/l			65 - 140	25	65 - 140	25
Chlorobenzene	0.36	2.0 ug/l			75 - 125	20	75 - 120	20
Dibromochloromethane	0.28	2.0 ug/l			65 - 140	25	70 - 140	20
Chloroethane	0.40	5.0 ug/l			55 - 140	25	60 - 140	20
Chloroform	0.33	2.0 ug/l			65 - 135	20	70 - 130	20
Chloromethane	0.40	5.0 ug/l			45 - 145	25	50 - 140	25
1,2-Dichlorobenzene	0.32	2.0 ug/l			75 - 125	20	75 - 120	20
1,3-Dichlorobenzene	0.35	2.0 ug/l			75 - 125	20	75 - 120	20
1,4-Dichlorobenzene	0.37	2.0 ug/l			75 - 125	20	75 - 120	20
1,1-Dichloroethane	0.27	2.0 ug/l			65 - 130	20	70 - 125	20
1,2-Dichloroethane	0.28	2.0 ug/l			60 - 140	20	60 - 140	20
1,1-Dichloroethene	0.42	5.0 ug/l			60 - 130	20	70 - 125	20
trans-1,2-Dichloroethene	0.27	2.0 ug/l			65 - 130	20	70 - 125	20
1,2-Dichloropropane	0.35	2.0 ug/l			65 - 130	20	70 - 125	20
cis-1,3-Dichloropropene	0.22	2.0 ug/l			70 - 130	20	75 - 125	65
trans-1,3-Dichloropropene	0.32	2.0 ug/l			65 - 135	25	70 - 125	20
Ethylbenzene	0.25	2.0 ug/l			65 - 130	20	75 - 125	20
2-Hexanone	2.6	10 ug/l			25 - 140	35	45 - 140	30
Methylene chloride	0.95	5.0 ug/l			50 - 135	20	55 - 130	20
4-Methyl-2-pentanone (MIBK)	3.5	5.0 ug/l			40 - 140	35	45 - 140	30
Styrene	0.16	2.0 ug/l			50 - 145	30	75 - 130	20
1,1,2,2-Tetrachloroethane	0.24	2.0 ug/l			55 - 135	30	55 - 130	25
Tetrachloroethene	0.32	2.0 ug/l			65 - 130	20	70 - 125	20
Toluene	0.36	2.0 ug/l			70 - 125	20	70 - 120	20
1,1,1-Trichloroethane	0.30	2.0 ug/l			65 - 140	20	65 - 135	20
1,1,2-Trichloroethane	0.30	2.0 ug/l			65 - 130	25	70 - 125	20
Trichloroethene	0.26	2.0 ug/l			65 - 125	20	70 - 125	20
Trichlorofluoromethane	0.34	5.0 ug/l			60 - 145	25	65 - 145	20
Vinyl acetate	1.0	5.0 ug/l			40 - 150	30	45 - 145	20
Vinyl chloride	0.30	5.0 ug/l			45 - 140	30	55 - 135	30
Xylenes, Total	0.90	2.0 ug/l				20		20
surr: Dibromofluoromethane								80 - 120
surr: Toluene-d8								80 - 120
surr: 4-Bromofluorobenzene								80 - 120

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>8260B-All Analytes (Std List) in Water (EPA 8260B)</b>								
Preservation:4 C, HCL								
Container:40 mL Voa Vial								
Amount Required:3 VOA								
Hold Time:14 days								
1,1,1,2-Tetrachloroethane	0.27	5.0 ug/l			65 - 140	20	70 - 130	20
1,1,1-Trichloroethane	0.30	2.0 ug/l			65 - 140	20	65 - 135	20
1,1,2,2-Tetrachloroethane	0.24	2.0 ug/l			55 - 135	30	55 - 130	25
1,1,2-Trichloroethane	0.30	2.0 ug/l			65 - 130	25	70 - 125	20
1,1-Dichloroethane	0.27	2.0 ug/l			65 - 130	20	70 - 125	20
1,1-Dichloroethene	0.42	5.0 ug/l			60 - 130	20	70 - 125	20
1,1-Dichloropropene	0.28	2.0 ug/l			70 - 135	20	75 - 130	20
1,2,3-Trichlorobenzene	0.30	5.0 ug/l			60 - 135	20	65 - 125	20
1,2,3-Trichloropropane	0.40	10 ug/l			55 - 135	30	60 - 130	20
1,2,4-Trichlorobenzene	0.48	5.0 ug/l			65 - 135	20	70 - 135	20
1,2,4-Trimethylbenzene	0.23	2.0 ug/l			55 - 135	25	75 - 125	20
1,2-Dibromo-3-chloropropane	0.97	5.0 ug/l			45 - 145	30	50 - 135	30
1,2-Dibromoethane (EDB)	0.40	2.0 ug/l			70 - 130	25	75 - 125	20
1,2-Dichlorobenzene	0.32	2.0 ug/l			75 - 125	20	75 - 120	20
1,2-Dichloroethane	0.28	2.0 ug/l			60 - 140	20	60 - 140	20
1,2-Dichloropropane	0.35	2.0 ug/l			65 - 130	20	70 - 125	20
1,3,5-Trimethylbenzene	0.26	2.0 ug/l			70 - 130	20	75 - 125	20
1,3-Dichlorobenzene	0.35	2.0 ug/l			75 - 125	20	75 - 120	20
1,3-Dichloropropane	0.32	2.0 ug/l			65 - 135	25	70 - 120	20
1,4-Dichlorobenzene	0.37	2.0 ug/l			75 - 125	20	75 - 120	20
2,2-Dichloropropane	0.34	2.0 ug/l			60 - 145	25	65 - 140	25
2-Butanone (MEK)	4.7	10 ug/l			30 - 145	40	40 - 140	35
2-Chloroethyl vinyl ether	1.8	5.0 ug/l			25 - 170	25	25 - 170	25
2-Chlorotoluene	0.28	5.0 ug/l			65 - 135	20	70 - 125	20
2-Hexanone	2.6	10 ug/l			25 - 140	35	45 - 140	30
4-Chlorotoluene	0.29	5.0 ug/l			70 - 135	20	75 - 125	20
4-Methyl-2-pentanone (MIBK)	3.5	10 ug/l			40 - 140	35	45 - 140	30
Acetone	4.5	10 ug/l			20 - 150	35	30 - 140	30
Benzene	0.28	2.0 ug/l			65 - 125	20	70 - 120	20
Bromobenzene	0.27	5.0 ug/l			70 - 125	20	75 - 120	20
Bromochloromethane	0.32	5.0 ug/l			65 - 135	25	70 - 130	20
Bromodichloromethane	0.30	2.0 ug/l			70 - 135	20	70 - 135	20
Bromoform	0.40	5.0 ug/l			55 - 135	25	55 - 130	25
Bromomethane	0.42	5.0 ug/l			55 - 145	25	65 - 140	20
Carbon Disulfide	0.48	5.0 ug/l			40 - 140	20	50 - 130	20
Carbon tetrachloride	0.28	5.0 ug/l			65 - 140	25	65 - 140	25
Chlorobenzene	0.36	2.0 ug/l			75 - 125	20	75 - 120	20
Chloroethane	0.40	5.0 ug/l			55 - 140	25	60 - 140	20
Chloroform	0.33	2.0 ug/l			65 - 135	20	70 - 130	20
Chloromethane	0.40	5.0 ug/l			45 - 145	25	50 - 140	25
cis-1,2-Dichloroethene	0.32	2.0 ug/l			65 - 130	20	70 - 125	20
cis-1,3-Dichloropropene	0.22	2.0 ug/l			70 - 130	20	75 - 125	65
Dibromochloromethane	0.28	2.0 ug/l			65 - 140	25	70 - 140	20
Dibromomethane	0.36	2.0 ug/l			65 - 135	25	70 - 125	20
Dichlorodifluoromethane	0.26	5.0 ug/l			25 - 155	30	35 - 155	30
Di-isopropyl Ether (DIPE)	0.25	5.0 ug/l			60 - 140	25	60 - 135	20

## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
Ethanol	100	150 ug/l			40 - 155	30	40 - 155	30
Ethyl tert-Butyl Ether (ETBE)	0.28	5.0 ug/l			60 - 135	25	65 - 135	20
Ethylbenzene	0.25	2.0 ug/l			65 - 130	20	75 - 125	20
Hexachlorobutadiene	0.38	5.0 ug/l			60 - 135	20	65 - 135	20
Isopropylbenzene	0.25	2.0 ug/l			70 - 135	20	75 - 130	20
m,p-Xylenes	0.60	2.0 ug/l			65 - 130	25	75 - 125	20
Methylene chloride	0.95	5.0 ug/l			50 - 135	20	55 - 130	20
Methyl-tert-butyl Ether (MTBE)	0.32	5.0 ug/l			55 - 145	25	60 - 135	25
Naphthalene	0.41	5.0 ug/l			50 - 140	30	55 - 135	25
n-Butylbenzene	0.37	5.0 ug/l			65 - 135	20	70 - 130	20
n-Propylbenzene	0.27	2.0 ug/l			70 - 135	20	75 - 130	20
o-Xylene	0.30	2.0 ug/l			65 - 125	20	75 - 125	20
p-Isopropyltoluene	0.28	2.0 ug/l			65 - 130	20	75 - 125	20
sec-Butylbenzene	0.25	5.0 ug/l			70 - 125	20	70 - 125	20
Styrene	0.16	2.0 ug/l			50 - 145	30	75 - 130	20
tert-Amyl Methyl Ether (TAME)	0.33	5.0 ug/l			60 - 140	30	60 - 135	25
tert-Butanol (TBA)	4.9	50 ug/l			65 - 140	25	70 - 135	20
tert-Butylbenzene	0.22	5.0 ug/l			65 - 130	20	70 - 125	20
Tetrachloroethene	0.32	2.0 ug/l			65 - 130	20	70 - 125	20
Toluene	0.36	2.0 ug/l			70 - 125	20	70 - 120	20
trans-1,2-Dichloroethene	0.27	2.0 ug/l			65 - 130	20	70 - 125	20
trans-1,3-Dichloropropene	0.32	2.0 ug/l			65 - 135	25	70 - 125	20
Trichloroethene	0.26	2.0 ug/l			65 - 125	20	70 - 125	20
Trichlorofluoromethane	0.34	5.0 ug/l			60 - 145	25	65 - 145	20
Vinyl acetate	1.0	5.0 ug/l			40 - 150	30	45 - 145	20
Vinyl chloride	0.30	5.0 ug/l			45 - 140	30	55 - 135	30
surr: Dibromofluoromethane			80 - 120					
surr: 4-Bromofluorobenzene			80 - 120					
surr: Toluene-d8			80 - 120					

## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
<b>8260B-All Analytes (Std List) in Soil (EPA 8260B)</b>								
Preservation:4 C, Cool								
Container:4 oz Jar/BrassSleeve								
Amount Required:100 grams								
Hold Time:14 days								
1,1,1,2-Tetrachloroethane	0.57	5.0 ug/kg			65 - 145	20	70 - 130	20
1,1,1-Trichloroethane	0.70	2.0 ug/kg			65 - 145	20	65 - 135	20
1,1,2,2-Tetrachloroethane	0.86	2.0 ug/kg			40 - 160	30	55 - 140	30
1,1,2-Trichloroethane	0.87	2.0 ug/kg			65 - 140	30	65 - 135	20
1,1-Dichloroethane	0.50	2.0 ug/kg			65 - 135	25	70 - 130	20
1,1-Dichloroethene	0.60	5.0 ug/kg			65 - 135	25	70 - 125	20
1,1-Dichloropropene	0.40	2.0 ug/kg			65 - 135	20	70 - 130	20
1,2,3-Trichlorobenzene	1.0	5.0 ug/kg			45 - 145	30	60 - 130	20
1,2,3-Trichloropropane	1.0	10 ug/kg			50 - 150	30	60 - 135	25
1,2,4-Trichlorobenzene	1.0	5.0 ug/kg			50 - 140	30	70 - 135	20
1,2,4-Trimethylbenzene	0.78	2.0 ug/kg			65 - 140	25	70 - 125	20
1,2-Dibromo-3-chloropropane	1.5	5.0 ug/kg			40 - 150	30	50 - 135	30
1,2-Dibromoethane (EDB)	0.80	2.0 ug/kg			65 - 140	25	70 - 130	20
1,2-Dichlorobenzene	0.95	2.0 ug/kg			70 - 130	25	75 - 120	20
1,2-Dichloroethane	0.80	2.0 ug/kg			60 - 150	25	60 - 140	20
1,2-Dichloropropane	0.35	2.0 ug/kg			65 - 130	20	70 - 130	20
1,3,5-Trimethylbenzene	0.63	2.0 ug/kg			65 - 135	25	70 - 125	20
1,3-Dichlorobenzene	0.84	2.0 ug/kg			70 - 130	25	75 - 125	20
1,3-Dichloropropane	0.63	2.0 ug/kg			65 - 140	25	70 - 125	20
1,4-Dichlorobenzene	0.94	2.0 ug/kg			70 - 130	25	75 - 120	20
2,2-Dichloropropane	0.45	2.0 ug/kg			65 - 150	25	60 - 145	20
2-Butanone (MEK)	6.0	10 ug/kg			25 - 170	40	40 - 145	35
2-Chloroethyl vinyl ether	3.8	5.0 ug/kg			25 - 170	30	25 - 170	30
2-Chlorotoluene	0.87	5.0 ug/kg			60 - 135	25	70 - 125	20
2-Hexanone	9.1	10 ug/kg			35 - 160	40	40 - 150	35
4-Chlorotoluene	0.74	5.0 ug/kg			65 - 135	25	75 - 125	20
4-Methyl-2-pentanone (MIBK)	3.2	5.0 ug/kg			40 - 155	40	40 - 145	35
Acetone	8.0	10 ug/kg			20 - 145	40	25 - 145	30
Benzene	0.50	2.0 ug/kg			65 - 130	20	65 - 120	20
Bromobenzene	0.84	5.0 ug/kg			65 - 140	25	75 - 120	20
Bromochloromethane	0.90	5.0 ug/kg			65 - 145	25	70 - 135	20
Bromodichloromethane	0.42	2.0 ug/kg			65 - 145	20	70 - 135	20
Bromoform	0.80	5.0 ug/kg			50 - 145	30	55 - 135	25
Bromomethane	0.92	5.0 ug/kg			60 - 155	25	60 - 145	20
Carbon Disulfide	0.97	5.0 ug/kg			40 - 140	20	50 - 130	20
Carbon tetrachloride	0.50	5.0 ug/kg			60 - 145	25	65 - 140	20
Chlorobenzene	0.52	2.0 ug/kg			70 - 130	25	75 - 120	20
Chloroethane	1.5	5.0 ug/kg			60 - 150	25	60 - 140	25
Chloroform	0.50	2.0 ug/kg			65 - 135	20	70 - 130	20
Chloromethane	1.0	5.0 ug/kg			40 - 145	25	45 - 145	25
cis-1,2-Dichloroethene	0.83	2.0 ug/kg			65 - 135	25	70 - 125	20
cis-1,3-Dichloropropene	0.44	2.0 ug/kg			70 - 135	25	75 - 125	20
Dibromochloromethane	0.56	2.0 ug/kg			60 - 145	25	65 - 140	20
Dibromomethane	0.90	2.0 ug/kg			65 - 140	25	70 - 130	20
Dichlorodifluoromethane	1.5	5.0 ug/kg			30 - 160	35	35 - 160	30
Di-isopropyl Ether (DIPE)	0.50	5.0 ug/kg			60 - 150	25	60 - 140	20

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
Ethanol	120	300 ug/kg			30 - 165	40	35 - 160	30
Ethyl tert-Butyl Ether (ETBE)	0.58	5.0 ug/kg			60 - 145	30	60 - 140	20
Ethylbenzene	0.50	2.0 ug/kg			70 - 135	25	70 - 125	20
Hexachlorobutadiene	0.73	5.0 ug/kg			50 - 145	35	60 - 135	20
Isopropylbenzene	0.54	2.0 ug/kg			70 - 145	25	75 - 130	20
m,p-Xylenes	0.80	2.0 ug/kg			70 - 130	25	70 - 125	20
Methylene chloride	6.5	20 ug/kg			55 - 145	25	55 - 135	20
Methyl-tert-butyl Ether (MTBE)	1.0	5.0 ug/kg			55 - 155	35	60 - 140	25
Naphthalene	1.1	5.0 ug/kg			40 - 150	40	55 - 135	25
n-Butylbenzene	0.72	5.0 ug/kg			55 - 145	30	70 - 130	20
n-Propylbenzene	0.61	2.0 ug/kg			65 - 140	25	70 - 130	20
o-Xylene	0.50	2.0 ug/kg			65 - 130	25	70 - 125	20
p-Isopropyltoluene	0.72	2.0 ug/kg			60 - 140	25	75 - 125	20
sec-Butylbenzene	0.67	5.0 ug/kg			60 - 135	25	70 - 125	20
Styrene	0.58	2.0 ug/kg			70 - 140	25	75 - 130	20
tert-Amyl Methyl Ether (TAME)	0.64	5.0 ug/kg			60 - 150	25	60 - 145	20
tert-Butanol (TBA)	7.0	100 ug/kg			65 - 145	30	70 - 135	20
tert-Butylbenzene	0.62	5.0 ug/kg			60 - 140	25	70 - 125	20
Tetrachloroethene	0.49	2.0 ug/kg			65 - 135	25	70 - 125	20
Toluene	0.50	2.0 ug/kg			70 - 130	20	70 - 125	20
trans-1,2-Dichloroethene	0.70	2.0 ug/kg			70 - 135	25	70 - 125	20
trans-1,3-Dichloropropene	0.61	2.0 ug/kg			60 - 145	25	70 - 135	20
Trichloroethene	0.50	2.0 ug/kg			65 - 140	25	70 - 125	20
Trichlorofluoromethane	0.54	5.0 ug/kg			55 - 155	25	60 - 145	25
Vinyl acetate	1.8	5.0 ug/kg			40 - 150	30	45 - 145	20
Vinyl chloride	0.91	5.0 ug/kg			55 - 140	30	55 - 135	25
surr: 4-Bromofluorobenzene			80 - 120					
surr: Dibromofluoromethane			80 - 125					
surr: Toluene-d8			80 - 120					

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>8260B-All Analytes (Std List) in Soil-extr (EPA 8260B)</b>								
Preservation:4 C, Cool								
Container:4 oz Jar/BrassSleeve								
Amount Required:100 grams								
Hold Time:14 days								
1,1,1,2-Tetrachloroethane	0.21	2.5 ug/kg			60 - 150	20	70 - 140	20
1,1,1-Trichloroethane	0.38	1.0 ug/kg			60 - 140	20	65 - 140	20
1,1,2,2-Tetrachloroethane	0.54	1.0 ug/kg			50 - 145	25	55 - 135	25
1,1,2-Trichloroethane	0.53	1.0 ug/kg			60 - 145	25	65 - 130	20
1,1-Dichloroethane	0.40	1.0 ug/kg			60 - 145	25	65 - 130	20
1,1-Dichloroethene	0.48	2.5 ug/kg			55 - 155	25	75 - 140	20
1,1-Dichloropropene	0.39	1.0 ug/kg			60 - 140	25	70 - 130	20
1,2,3-Trichlorobenzene	0.58	2.5 ug/kg			50 - 140	25	60 - 135	20
1,2,3-Trichloropropane	0.48	5.0 ug/kg			50 - 145	30	55 - 130	25
1,2,4-Trichlorobenzene	0.51	2.5 ug/kg			60 - 140	25	65 - 135	20
1,2,4-Trimethylbenzene	0.33	1.0 ug/kg			60 - 140	25	70 - 125	20
1,2-Dibromo-3-chloropropane	0.59	2.5 ug/kg			40 - 160	30	45 - 135	25
1,2-Dibromoethane (EDB)	0.40	1.0 ug/kg			65 - 145	25	70 - 130	20
1,2-Dichlorobenzene	0.32	1.0 ug/kg			60 - 135	25	70 - 120	20
1,2-Dichloroethane	0.41	1.0 ug/kg			60 - 145	25	60 - 145	20
1,2-Dichloropropane	0.35	1.0 ug/kg			60 - 140	25	75 - 125	20
1,3,5-Trimethylbenzene	0.30	1.0 ug/kg			65 - 140	25	70 - 125	20
1,3-Dichlorobenzene	0.31	1.0 ug/kg			60 - 145	25	70 - 125	20
1,3-Dichloropropane	0.36	1.0 ug/kg			65 - 135	25	65 - 130	20
1,4-Dichlorobenzene	0.34	1.0 ug/kg			60 - 140	25	70 - 125	20
2,2-Dichloropropane	0.27	1.0 ug/kg			50 - 150	25	60 - 145	25
2-Butanone (MEK)	7.2	10 ug/kg			20 - 175	40	40 - 145	30
2-Chloroethyl vinyl ether	1.8	2.5 ug/kg			25 - 170	30	25 - 170	30
2-Chlorotoluene	0.32	2.5 ug/kg			60 - 145	25	70 - 125	20
2-Hexanone	1.0	5.0 ug/kg			30 - 155	40	35 - 140	30
4-Chlorotoluene	0.32	2.5 ug/kg			65 - 140	25	70 - 125	20
4-Methyl-2-pentanone (MIBK)	3.9	5.0 ug/kg			35 - 155	40	40 - 145	30
Acetone	11	15 ug/kg			25 - 135	40	25 - 135	30
Benzene	0.34	1.0 ug/kg			55 - 140	25	65 - 120	20
Bromobenzene	0.34	2.5 ug/kg			60 - 140	25	70 - 120	20
Bromochloromethane	0.50	2.5 ug/kg			60 - 145	25	65 - 125	20
Bromodichloromethane	0.31	1.0 ug/kg			60 - 150	25	65 - 135	20
Bromoform	0.39	2.5 ug/kg			50 - 140	30	50 - 130	25
Bromomethane	0.45	2.5 ug/kg			30 - 140	30	30 - 140	30
Carbon Disulfide	0.57	2.5 ug/kg			50 - 130	25	50 - 130	20
Carbon tetrachloride	0.28	2.5 ug/kg			65 - 145	25	65 - 145	20
Chlorobenzene	0.30	1.0 ug/kg			65 - 145	25	70 - 125	20
Chloroethane	0.45	2.5 ug/kg			35 - 140	30	40 - 140	25
Chloroform	0.44	1.0 ug/kg			60 - 140	25	75 - 130	20
Chloromethane	0.50	2.5 ug/kg			25 - 140	30	30 - 140	25
cis-1,2-Dichloroethene	0.48	1.0 ug/kg			55 - 135	25	65 - 130	20
cis-1,3-Dichloropropene	0.36	1.0 ug/kg			65 - 140	25	70 - 130	20
Dibromochloromethane	0.27	1.0 ug/kg			55 - 150	25	65 - 140	20
Dibromomethane	0.42	1.0 ug/kg			65 - 135	25	65 - 130	20
Dichlorodifluoromethane	0.61	2.0 ug/kg			10 - 155	35	10 - 155	30
Di-isopropyl Ether (DIPE)	0.51	2.5 ug/kg			60 - 150	25	60 - 140	20

## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
Ethanol	100	150 ug/kg			30 - 160	40	35 - 160	30
Ethyl tert-Butyl Ether (ETBE)	0.66	2.5 ug/kg			60 - 150	25	60 - 140	20
Ethylbenzene	0.27	1.0 ug/kg			50 - 150	25	80 - 120	20
Hexachlorobutadiene	0.39	2.5 ug/kg			55 - 145	35	60 - 135	20
Isopropylbenzene	0.35	1.0 ug/kg			65 - 145	25	70 - 125	20
m,p-Xylenes	0.53	1.0 ug/kg			60 - 145	25	70 - 125	20
Methylene chloride	4.3	10 ug/kg			55 - 145	25	60 - 140	20
Methyl-tert-butyl Ether (MTBE)	0.62	2.5 ug/kg			55 - 155	30	55 - 145	25
Naphthalene	0.81	2.5 ug/kg			35 - 160	30	50 - 140	25
n-Butylbenzene	0.34	2.5 ug/kg			55 - 155	25	70 - 130	20
n-Propylbenzene	0.36	1.0 ug/kg			50 - 150	25	70 - 130	20
o-Xylene	0.28	1.0 ug/kg			55 - 145	25	70 - 125	20
p-Isopropyltoluene	0.48	1.0 ug/kg			60 - 140	25	70 - 125	20
sec-Butylbenzene	0.31	2.5 ug/kg			55 - 145	25	70 - 125	20
Styrene	0.30	1.0 ug/kg			60 - 150	25	70 - 135	20
tert-Amyl Methyl Ether (TAME)	0.68	2.5 ug/kg			60 - 150	25	60 - 145	25
tert-Butanol (TBA)	5.0	50 ug/kg			60 - 155	25	65 - 140	20
tert-Butylbenzene	0.27	2.5 ug/kg			65 - 150	25	70 - 125	20
Tetrachloroethene	0.44	1.0 ug/kg			60 - 150	25	65 - 125	20
Toluene	0.33	1.0 ug/kg			55 - 140	25	80 - 120	20
trans-1,2-Dichloroethene	0.50	1.0 ug/kg			55 - 145	25	65 - 130	20
trans-1,3-Dichloropropene	0.35	0.50 ug/kg			60 - 145	25	65 - 135	20
Trichloroethene	0.38	1.0 ug/kg			65 - 150	25	70 - 130	20
Trichlorofluoromethane	0.58	2.5 ug/kg			35 - 150	30	50 - 145	25
Vinyl acetate	0.60	2.5 ug/kg			20 - 145	30	25 - 145	25
Vinyl chloride	0.65	2.5 ug/kg			10 - 120	35	10 - 120	30
surr: 4-Bromofluorobenzene			65 - 140					
surr: Dibromofluoromethane			55 - 140					
surr: Toluene-d8			60 - 140					



## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>8260B-All Analytes (Extended) in Water (EPA 8260B)</b>								
Preservation:4 C, HCL								
Container:40 mL Voa Vial								
Amount Required:3 VOA								
Hold Time:14 days								
1,1-Dichloro-1-fluoroethane	1.0	2.5 ug/l			60 - 140	40	70 - 125	30
1,2-Dibromotetrafluoroethane	1.0	5.0 ug/l			60 - 140	40	70 - 125	30
1,2-Dichloro-1,1,2-trifluoroethane	1.1	2.0 ug/l			60 - 140	40	70 - 125	30
2-Chloro-1,3-butadiene	0.60	1.0 ug/l			60 - 140	40	70 - 125	30
Acetonitrile	9.0	20 ug/l			60 - 140	40	70 - 125	30
Acrolein	4.0	20 ug/l			60 - 140	40	70 - 125	30
Acrylonitrile	0.70	20 ug/l			60 - 140	40	70 - 125	30
Allyl Chloride	0.40	1.0 ug/l			60 - 140	40	70 - 125	30
Chlorodifluoromethane	1.0	2.5 ug/l			60 - 140	40	70 - 125	40
Dichlorofluoromethane	0.90	2.5 ug/l			60 - 140	40	70 - 125	40
Ethyl Methacrylate	0.90	2.0 ug/l			60 - 140	40	70 - 125	30
Hexachloroethane	0.90	5.0 ug/l			60 - 140	40	70 - 125	30
Iodomethane	1.0	2.0 ug/l			60 - 140	40	70 - 125	30
Isobutanol (2-Methyl-1-Propanol)	7.0	20 ug/l			60 - 140	40	70 - 125	30
Isopropyl alcohol	16	100 ug/l			60 - 140	40	70 - 125	30
Methyl Acrylonitrile	0.90	2.0 ug/l			60 - 140	40	70 - 125	30
Methyl Methacrylate	0.90	2.0 ug/l			60 - 140	40	70 - 125	30
Propionitrile	7.0	20 ug/l			60 - 140	40	70 - 125	30
tert-Butyl Formate (TBF)	1.0	10 ug/l			60 - 140	40	70 - 125	30
Tetrahydrofuran (THF)	3.5	10 ug/l			60 - 140	40	70 - 125	30
Thiophene	1.1	5.0 ug/l			60 - 140	40	70 - 125	30
trans-1,4-Dichloro-2-butene	2.5	5.0 ug/l			60 - 140	40	70 - 125	30
Trichlorotrifluoroethane (Freon 113)	1.5	5.0 ug/l						
surr: 4-Bromofluorobenzene			80 - 120					
surr: Dibromofluoromethane			80 - 120					
surr: Toluene-d8			80 - 120					

## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
<b>8260B-All Analytes (Extended) in Soil (EPA 8260B)</b>								
Preservation:4 C, Cool								
Container:4 oz Jar/BrassSleeve								
Amount Required:100 grams								
Hold Time:14 days								
1,1-Dichloro-1-fluoroethane	2.0	5.0 ug/kg			60 - 140	40	60 - 140	40
1,2-Dibromotetrafluoroethane	2.0	5.0 ug/kg			60 - 140	40	60 - 140	40
1,2-Dichloro-1,1,2-trifluoroethane	1.7	2.0 ug/kg			60 - 140	40	60 - 140	40
2-Chloro-1,3-butadiene	0.80	2.0 ug/kg			60 - 140	40	60 - 140	40
Acetonitrile	17	40 ug/kg			60 - 140	40	60 - 140	40
Acrolein	13	100 ug/kg			60 - 140	40	60 - 140	40
Acrylonitrile	12	100 ug/kg			60 - 140	40	60 - 140	40
Allyl Chloride	1.3	2.0 ug/kg			60 - 140	40	60 - 140	40
Chlorodifluoromethane	2.0	5.0 ug/kg			60 - 140	40	60 - 140	40
Dichlorodifluoromethane	1.5	5.0 ug/kg			30 - 160	35	35 - 160	30
Ethyl Methacrylate	2.0	4.0 ug/kg			60 - 140	40	60 - 140	40
Hexachloroethane	1.5	10 ug/kg			60 - 140	40	60 - 140	40
Iodomethane	2.0	5.0 ug/kg			60 - 140	40	60 - 140	40
Isobutanol (2-Methyl-1-Propanol)	25	40 ug/kg			60 - 140	40	60 - 140	40
Isopropyl alcohol	35	200 ug/kg			60 - 140	40	60 - 140	40
Methyl Acrylonitrile	1.5	4.0 ug/kg			60 - 140	40	60 - 140	40
Methyl Methacrylate	2.0	4.0 ug/kg			60 - 140	40	60 - 140	40
Propionitrile	15	40 ug/kg			60 - 140	40	60 - 140	40
tert-Butyl Formate (TBF)	4.0	10 ug/kg			60 - 140	40	60 - 140	40
Tetrahydrofuran (THF)	6.0	20 ug/kg			60 - 140	40	60 - 140	40
Thiophene	2.0	10 ug/kg			60 - 140	40	60 - 140	40
trans-1,4-Dichloro-2-butene	3.5	10 ug/kg			60 - 140	40	60 - 140	40
Trichlorotrifluoroethane (Freon 113)	4.0	10 ug/kg			60 - 140	40	60 - 140	40
surr: 4-Bromofluorobenzene			80 - 120					
surr: Dibromofluoromethane			80 - 125					
surr: Toluene-d8			80 - 120					

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and 8081A.SOP Rev 2 (03/19/01)

**TESTAMERICA-IRVINE  
STANDARD OPERATING PROCEDURE  
ORGANOCHLORINE PESTICIDES BY GC  
EPA METHODS 608 AND 8081 A**

Approved By:  Date: 8/17/06  
Department Manager

Approved By:  Date: 8/17/06  
Quality Assurance Manager

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**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**ORGANOCHLORINE PESTICIDES BY GC**  
**EPA METHODS 608 AND 8081A**

**1. SCOPE AND APPLICATION**

- 1.1. EPA Method 608 is used to determine the concentration of various organochlorine pesticides in groundwater, surface water, or wastewater samples. EPA Method 8081A is used to determine the concentration of various organochlorine pesticides in extracts from solid and liquid matrices.
- 1.2. This standard operating procedure applies to both EPA Methods 608 and 8081 A (for Pesticides analysis only).
- 1.3. Liquid samples are extracted per Method 3510C . A measured volume of sample, approximately 1 L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less.
- 1.4. Solid samples are extracted with hexane-acetone (1:1) using Method 3545 (pressured fluid extration). The extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. (Oil by Method 3580A).
- 1.5. The extract is separated by gas chromatography and the parameters are then measured with an electron capture detector (ECD).
- 1.6. Single analytes and multi-component pesticides are identified using retention times and pattern recognition.
- 1.7. The common pesticides to be analyzed include:
  - 1.7.1. Aldrin
  - 1.7.2. a-BHC
  - 1.7.3. b-BHC
  - 1.7.4. d-BHC
  - 1.7.5. g-BHC
  - 1.7.6. Chlordane
  - 1.7.7. a-chlordane
  - 1.7.8. g-chlordane
  - 1.7.9. 4,4'-DDD
  - 1.7.10. 4,4'-DDE
  - 1.7.11. 4,4'-DDT
  - 1.7.12. Dieldrin
  - 1.7.13. Endosulfan I
  - 1.7.14. Endosulfan II
  - 1.7.15. Endosulfan sulfate
  - 1.7.16. Endrin
  - 1.7.17. Endrin aldehyde

**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**ORGANOCHLORINE PESTICIDES BY GC**  
**EPA METHODS 608 AND 8081A**

- 1.7.18. Endrin ketone
- 1.7.19. Heptachlor
- 1.7.20. Heptachlor epoxide
- 1.7.21. Methoxychlor
- 1.7.22. Toxaphene
- 1.7.8. Additional compounds that may be analyzed by this SOP include but are not limited to:
  - 1.7.8.1. 2,4'-DDD
  - 1.7.8.2. 2,4'-DDE
  - 1.7.8.3. 2,4'-DDT
  - 1.7.8.4. Benefin
  - 1.7.8.5. Trifluralin
  - 1.7.8.6. Pentachloronitrobenzene (PCNB)
  - 1.7.8.7. Mirex

NOTE: Although overlapping each other in many aspects, Method 608 and Method 8081A do differ in certain analysis requirements, notably:

- Method 8081A requires more calibration points (5 pts vs 3 pts).
- Method 608 requires a more stringent calibration factor RSD (10% vs 20%).
- Method 608 does NOT allow averaging of calibration factor RSD and ICV/CCV recoveries.
- Method 8081A does NOT allow the calibration curve to be forced through the point of origin.
- Method 8081A requires a higher frequency of calibration verification (once every 12-hour shift vs once daily).
- Method 8081A requires result confirmation from a secondary column.

An actual analysis may satisfy either one or both methods at a particular time. To meet both method requirements simultaneously, the more stringent QC criteria of the two should be followed. Be careful to indicate that compliance on the calibration and daily check lists.

## **2. DEFINITIONS**

- 2.1. There are no additional specific definitions associated with this test. See the QAPM and EPA methods 608 and 8081A for general definitions.
- 2.2. Primary column: the column used from which results are reported.
- 2.3. Secondary column: the column used for confirmations.

## **3. APPARATUS AND MATERIALS**

- 3.1. Equipment and Supplies

**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**ORGANOCHLORINE PESTICIDES BY GC**  
**EPA METHODS 608 AND 8081A**

- 3.1.1. Gas Chromatograph - HP5890 or equivalent
- 3.1.2. Column A: RTX-CLP or DB-608 (30m x 0.53mm x 0.83µm)
- 3.1.3. Column B: RTX-CLP2 or DB-XLB (30m x 0.53mm x 0.83µm)
- 3.1.4. Electron capture detector (ECD)
- 3.1.5. Autosampler – HP7673A
- 3.1.6. Injector liners
- 3.1.7. Volumetric flasks with ground-glass stoppers, Class A
- 3.1.8. 5 µl, 10 µl, 50 µl, 100 µl, 500 µl and 1000 µl micro syringe
- 3.1.9. Disposable pipets
- 3.1.10. 2 ml minert vials
- 3.1.11. 2 ml, 10 ml, and 40 ml glass vials with Teflon-lined screw caps or crimp tops
- 3.2. Reagents and Standards
  - 3.2.1. 1000 µg/ml Pesticides Mix (Ultra Scientific & Restek or equivalent)
  - 3.2.2. 200 µg/ml Pesticides Surrogate Spike Solution (Restek or equivalent)
  - 3.2.3. 1000 µg/ml Toxaphene Solution (Ultrascientific or equivalent)
  - 3.2.4. 1000 µg/ml Chlordane Solution (Supelco & Restek or equivalent)
  - 3.2.5. 500 µg/ml DDT/Endrin standard (Supelco or equivalent)
  - 3.2.6. Acetone, CH<sub>3</sub>COCH<sub>3</sub>, pesticide grade or equivalent
  - 3.2.7. n-Hexane, C<sub>6</sub>H<sub>14</sub>, pesticide grade or equivalent.
  - 3.2.8. Methanol, MeOH, pesticide grade or equivalent

**4. SAFETY**

- 4.1. Since all of the hazards of samples and chemicals used in this procedure are not entirely known, strict adherence to safety rules and use of prescribed personal protection equipment is mandatory. The health hazards of the standards, reagents and samples are not entirely known so caution must be exercised in all cases.
- 4.2. Employees performing this procedure must be familiar with the Chemical Hygiene Plan (CHP), and the precautions stated on the appropriate Material Safety Data Sheets (MSDS).
- 4.3. Personal Protective Equipment Required: Safety Glasses, Labcoat, and Gloves.

**5. INTERFERENCES**

- 5.1. The following are three broad categories of sources of interferences in EPA Methods 608 and 8081 A:

**TESTAMERICA-IRVINE**  
**STANDARD OPERATING PROCEDURE**  
**ORGANOCHLORINE PESTICIDES BY GC**  
**EPA METHODS 608 AND 8081A**

- 5.1.1. Contaminated solvents, reagents, glassware, or other sample processing hardware. Cross-contamination of clean glassware can easily occur when plastics are handled during extraction, especially when solvent-wetted surfaces are handled. Flexible plastic used during sample preparation can introduce Phthalate esters.
- 5.1.2. Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
- 5.1.3. Compounds extracted from the sample matrix to which the detector will respond.
- 5.2. For a detailed discussion on cleanup procedures, refer to Section 11 of Method 608, or Sections 3.7 and 7.2 of Method 8081 A. Some recommended cleanups are as follows:
  - 5.2.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic material, and by checking all solvents and reagents for phthalate contamination.
  - 5.2.2. Phthalate esters can be removed prior to analysis by using Method 3640 (Gel Permeation Cleanup), Method 3630 (Silica Gel Cleanup), or Method 3610 (Alumina).
  - 5.2.3. The presence of elemental sulfur will result in broad peaks that interfere with the ECD detection of early eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. It can be removed by technique described in Method 3660 or in Section 11.3 of Method 608, though rarely performed.
  - 5.2.4. Waxes, lipids and other high molecular weight materials can be removed by Method 3640 (GPC cleanup-pesticide option). Since these are not common contaminants, GPC is not currently performed.
  - 5.2.5. Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. These may be removed by Method 3620B (Florisil). Other cleanup methods are available but generally not performed.

**6. SAMPLE HANDLING AND PRESERVATION**

- 6.1. The required sample containers are 1.0 L amber glass bottles for liquid samples and 4 oz. jars or brass boring rings for solid samples.
- 6.2. All samples must be stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  prior to extraction.
- 6.3. Water and liquid samples have a holding time of 7 days before extraction.  
608 Only: Water samples shall be extracted within 72 hours of collection or 7 days if pH-adjusted to 5.0 – 9.0 using sulfuric acid or sodium hydroxide.  
Record the pH of all samples on the bench sheet/extraction logsheet.
- 6.4. Soil and solid samples have a holding time of 14 days before extraction.
- 6.5. Extracts have a holding time of 40 days before analysis.

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6.6. Notify the project manager immediately if the method holding time has been exceeded.

**7. PROCEDURE**

7.1. Standard Preparation

7.1.1. Store standards at 4°C in PTFE sealed containers, in the dark. Check the solutions frequently for signs of degradation or evaporation. Stock standard solutions must be replaced after one year from the date opened or according to the manufacturer's expiration date or sooner if routine QC indicates a problem. All other standards must be replaced 6 months or sooner if routine QC indicates a problem.

7.1.2. Transfer each solution from the volumetric flasks into two or three 40 ml or 12 ml VOA vials with Teflon-lined screw caps. Store the solutions at 4°C and protect from light. Check the solutions frequently for signs of degradation or evaporation. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicate a problem.

7.1.3. Enter the standard information into the standard logbook and the data system (Elmnt). All standard information must be reviewed for accuracy by a peer or department manager before the standard can be used.

7.1.4. Calibration Standards:

7.1.4.1. Prepare new calibration standards every 6 months, or sooner, if comparison with check standards indicates a problem. Enter the standard information into the standard logbook.

7.1.4.2. Prepare the calibration standards by adding the volumes of spike solutions shown in the following tables.

7.1.4.1.2.1. Table 1: Pesticides, Regular list

7.1.4.1.2.2. Table 2: A25—Regular list + 2,4-series

7.1.4.1.2.3. Table 3: A29—Regular list + 2,4-series + Benefin + Trifluralin + Pentachloronitrobenzene (PCNB) + Mirex

7.1.4.1.2.4. Table 4: Chlordane

7.1.4.1.2.5. Table 5: Toxaphene

**TABLE 1: Regular Pesticides**

Pest. Mother Cal. STD.	1 <sup>st</sup> source				
Stock	Pest. Mix 1000ug/ml	V of surr. Stock 200ug/ml	Bring to V	Final Conc.	# of Vials
Pesticide Mix 1000 ug/mL	0.125 mL	0.625 mL	25 mL	5K/5 ppm	2



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**Pesticide calibration**

Calibration	V of Mother soln.	Bring to V
25/25 ppb	0.125 mL	25 mL
50/50 ppb	0.25 mL	25 mL
100/100 ppb	0.5 mL	25 mL
200/200 ppb	1 mL	25 mL
300/300 ppb	1.5 mL	25 mL
500/500 ppb	2.5 mL	25 mL

**Pest. Cal. ICV STD. 2<sup>nd</sup> source**

Stock	Pesticide Mix 200 ug/ml	V of surr. Stock 200 ug/ml	Bring to V	Final Conc.	# of Vials
Pesticide Mix 200 ug/ml	0.0125	0.0125	25 mL	100/100PPB	16

**Pest. Cal. CCV1 STD. 1<sup>st</sup> source**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
0.5 mL	100 mL	25/25PPB	8

**Pest. Cal. CCV2 STD. 1<sup>st</sup> source**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
2 mL	100 mL	100/100 ppm	8

**Pest. Cal. CCV3 STD. 1<sup>st</sup> source**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
6 mL	100 mL	300/300ppm	8

**Pest. Cal. CCV4 STD. 1<sup>st</sup> source**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
0.004 mL	100 mL	2/2 ppb	8

**Pest. LCS/SPIKE Soln. 1<sup>st</sup> source**

Pesticide Mix 1000 ug/ml	Bring to V	Final Conc.	# of Vials
0.250 mL	500 mL	500 ppm	12

**Pest. Cal. low level**

Calibration	V of Mother soln.	Bring to V
2/2 ppb	0.01 mL	25 mL
10/10 ppb	0.05 mL	25 mL
25/25 ppb	0.125 mL	25 mL
100/100 ppb	0.5 mL	25 mL
300/300 ppb	1.5 mL	25 mL
500/500 ppb	2.5 mL	25 mL

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**TABLE 2: A25 Pesticide List**

**A25 Mother Cal. STD.                      1<sup>st</sup> source**

Stock	Source of stock	V of each stock	Bring to V	Final Conc.	# of Vials
Pesticide Mix 1000 ug/mL	Ultra	0.125	50 mL	2.5k/2.5k	1
8080 surrogate stock 200ug/ml	Restek	0.625			
2,4-DDD 100 ppm	Accustandard	1.25			
2,4-DDE 100 ppm	Accustandard	1.25			
2,4-DDT 100 ppm	Accustandard	1.25			

**A25 calibration**

Calibration	V of Mother soln.	Bring to V	# of Vials
25/25 ppb	0.25	25	1
50/50 ppb	0.5	25	1
100/100 ppb	1	25	1
200/200 ppb	2	25	1
300/300 ppb	3	25	1
500/500 ppb	5	25	1

**A25. ICV STD.                                      2<sup>nd</sup> source**

Stock	Source of stock	V of each stock	Bring to V	Final Conc.	# of Vials
Pesticide Mix 200 ug/mL	Restek	0.0125 mL	25 mL	100/100PPB	4
8080 surrogate stock 200 ug/mL	Restek	0.0125 mL			
2,4-DDD 100 ppm	Ultra	0.025 mL			
2,4-DDE 100 ppm	Ultra	0.025 mL			
2,4-DDT 100 ppm	Ultra	0.025 mL			

**A25 CCV1 Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
1 mL	100 mL	25/25 ppb	1

**A25 CCV2 Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
4 mL	100 mL	100/100 ppb	1

**A25 CCV3 Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
12 mL	100 mL	300/300 ppb	1

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**A25 Spike Soln.**

1<sup>st</sup> source

Stock	Source of stock	V of each stock	Bring to V	Final Conc.	# of Vials
Pesticide Mix 200 ug/ mL	Restek	0.25 mL	100 mL	500 ppm	1
2,4-DDD 100 ppm	Ultra	0.5 mL			
2,4-DDE 100 ppm	Ultra	0.5 mL			
2,4-DDT 100 ppm	Ultra	0.5 mL			

**TABLE 3: A29 Pesticide List**

**A29 Mother Cal. STD.**

1<sup>st</sup> source

Stock	Source of stock	V of each stock	Bring to V	Final Conc.	# of Vials
Trifluralin 1000 ug/mL	Absolute Std.	0.025 mL	10 mL	2.5k/2.5k	1
Pesticide Mix 1000 ug/mL	Ultra	0.025 mL			
8080 surrogate stock 200 ug/mL	Restek	0.125 mL			
2,4-DDD 100 ppm	Accustandard	0.250 mL			
2,4-DDE 100 ppm	Accustandard	0.250 mL			
2,4-DDT 100 ppm	Accustandard	0.250 mL			
Mirex 100mg/L	o2si	0.250 mL			
Pentachloronitrobenzene 100 ppm	Restek	0.250 mL			
Benefin 100 ppm	Accustandard	0.250 mL			

**A29 calibration**

Calibration	V of Mother soln.	Bring to V	# of Vials
25/25 ppb	0.100 mL	10 mL	1
50/50 ppb	0.200 mL	10 mL	1
100/100 ppb	0.400 mL	10 mL	1
200/200 ppb	0.800 mL	10 mL	1
300/300 ppb	1.2 mL	10 mL	1
500/500 ppb	2 mL	10 mL	1

**A29. ICV STD.**

2<sup>nd</sup> source

Stock	Source of stock	V of each stock	Bring to V	Final Conc.	# of Vials
Trifluralin 1000 ug/mL	Absolute Std.	0.005 mL	50 mL	100/100PPB	4
Pesticide Mix 200 ug/mL	Restek	0.025 mL			
8080 surrogate stock 200 ug/mL	Restek	0.025 mL			
2,4-DDD 100 ppm	Ultra	0.05 mL			
2,4-DDE 100 ppm	Ultra	0.05 mL			
2,4-DDT 100 ppm	Ultra	0.05 mL			
Mirex 100 mg/L	Ultra	0.05 mL			
Pentachloronitrobenzene 1000 ppm	Absolute Std.	0.005 mL			
Benefin 1000 ppm	Absolute Std.	0.005 mL			

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**A29 CCV1 Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
0.250 mL	25 mL	25/25 ppb	2

**A29 CCV2 Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
1 mL	25 mL	100/100 ppb	2

**A29 CCV3 Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
3 mL	25 mL	300/300 ppb	2

**A29 Spike Soln.**

1<sup>st</sup> source

Stock	Source of stock	V of each stock	Bring to V	Final Conc.	# of Vials
Trifluralin 1000 ug/mL	Absolute Std.	0.025 mL	50 mL	500 ppm	1
Pesticide Mix 1000 ug/mL	Ultra	0.025 mL			
2,4-DDD 100 ppm	Accustandard	0.250 mL			
2,4-DDE 100 ppm	Accustandard	0.250 mL			
2,4-DDT 100 ppm	Accustandard	0.250 mL			
Mirex 100 mg/L	o2si	0.250 mL			
Pentachloronitrobenzene 100 ppm	Restek	0.250 mL			
Benefin 100 ppm	Accustandard	0.250 mL			

**TABLE 4: Chlordane**

**CD Mother Cal. STD.**

1<sup>st</sup> source

Stock	CD Cal. Stock 1000 ug/mL	V of surr. Stock 200 ug/mL	Bring to V	Final Conc.	# of Vials
CD Cal. Stock 1000 ug/mL	0.5 mL	0.250 mL	10 mL	50K/5k	2

**CD calibration**

Calibration	V of Mother soln.	Bring to V
250/25 ppb	0.050 mL	10 mL
500/50 ppb	0.100 mL	10 mL
1K/100 ppb	0.200 mL	10 mL
2K/200 ppb	0.400 mL	10 mL
3K/300 ppb	0.600 mL	10 mL
5K/500 ppb	1 mL	10 mL

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**CD ICV STD.**

2nd source  
SUPELCO

Stock	CD mix 1000ug/ml	V of surr. Stock 200ug/ml	Bring to V	Final Conc.	# of Vials
CD mix 1000 ug/mL	0.025 mL	0.0125 mL	25 mL	1K/100PPB	4

**CD Cal. CCV1 STD.**

1<sup>st</sup> source

V of Mother soln.	Bring to V	Final Conc.	# of Vials
1.5 mL	25 mL	3K/300 ppb	4

**CD RL Std.**

V of Mother soln.	Bring to V	Final Conc.	# of Vials
0.25 mL	50 mL	250/25 ppb	1

**CD MDL Spike**

Stock	CD mix 1000ug/ml	Bring to V	Final Conc.	# of Vials
CD mix 1000 ug/mL	0.025 mL	50 mL	500 ppb	2

**TABLE 5: Toxaphene**

**TX Mother Cal. STD.**

1<sup>st</sup> source

Stock	TX Stock Stock 1000ug/ml	V of surr. Stock 200ug/ml	Bring to V	Final Conc.	# of Vials
TX Stock 1000 ug/mL	1.25 mL	0.3125 mL	25 mL	50K/2.5 K	2

**TX calibration**

Calibration	V of Mother soln.	Bring to V
500/25 ppb	0.25	25 mL
1K/50 ppb	0.5 mL	25 mL
2K/100 ppb	1 mL	25 mL
3K/150 ppb	1.5 mL	25 mL
5K/250 ppb	2.5 mL	25 mL
7K/350 ppb	3.5 mL	25 mL
10K/500 ppb	5 mL	25 mL

**TX ICV STD.**

2<sup>nd</sup> source - Ultra

Stock	TX ICV Stock 1000ug/ml	V of surr. Stock 200ug/ml	Bring to V	Final Conc.	# of Vials
TX ICV Stock 1000 ug/mL	0.125 mL	0.03125 mL	25 mL	5K/250 PPB	8

**TX Cal. CCV1 STD.**

1<sup>st</sup> source

V of Mother soln.	Bring to V	Final Conc.	# of Vials
6 mL	100 mL	3K/150 PPB	4

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**TX Cal. CCV2 STD.                      1st source**

<b>V of Mother soln.</b>	<b>Bring to V</b>	<b>Final Conc.</b>	<b># of Vials</b>
14 mL	100 mL	7K/350 PPB	4

**TX MDL Spike**

<b>Stock</b>	<b>TX ICV Stock 1000 ug/mL</b>	<b>Bring to V</b>	<b>Final Conc.</b>	<b># of Vials</b>
TX ICV Stock 1000 ug/mL	0.01 mL	50 mL	2000 ppb	2

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- 7.2. Initial Degradation Check
- 7.2.1. Prime (or deactivate) the GC column after one or more days of non-operation. Prime (or deactivate) the column by injecting the highest pesticide calibration standard. Inject this standard prior to calibration to prevent column adsorption.
- 7.2.2. Analyze a degradation standard containing DDT and Endrin initially. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and of Endrin (Endrin Ketone and Endrin Aldehyde).
- NOTE: This SOP complies to this criteria specified by Method 8081A for all single component pesticide analyses.
- 7.2.3. Calculate the percent breakdown of DDT and Endrin on both the primary and secondary columns and verify that it is  $\leq 15\%$ .
- 7.2.4. Take corrective action, if degradation of either DDT or Endrin exceeds 15%. Corrective action may include:
- 7.2.4.1. Replace the glass injection port insert
- 7.2.4.2. Change the "Y" connector or replace as needed
- 7.2.4.3. Prepare a new standard
- 7.2.4.4. Trim the first few inches to one foot of the column
- 7.2.4.5. Change the precolumn
- 7.2.4.6. Backflush the column with solvent according to the manufacturer's instructions
- 7.2.4.7. Deactivate the metal injector body with Sylon-CT or equivalent
- 7.2.4.8. Replace the column
- 7.2.4.9. Record all performed maintenance in the instrument maintenance logbook.
- 7.3. External Calibration Procedure and Initial Verification
- 7.3.1. For Method 608, analyze a minimum of three standard points for a working calibration curve. For Method 8081 A, use a minimum of five standard points. Using five points will satisfy both method requirements. Calibrate the primary and secondary columns simultaneously.
- 7.3.2. Repeat for Toxaphene and Chlordane.
- 7.3.3. The surrogates are calibrated in various levels at the same time as the pesticides standards.

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- 7.3.4. For Method 608, the % RSD of the Calibration Factors (CFs) for each target analyte and surrogate must be  $\leq 10\%$  for both the primary and secondary columns in order to quantitate the compound using the average CF. For Method 8081A, the requirement for the % RSD of the CFs is  $\leq 20\%$ . Only Method 8081A allows averaging the % RSD of the CFs. For simultaneous analysis using both methods, the %RSD must be  $\leq 10\%$ .

NOTE: combining both methods in one analysis works best when the % RSD of the CFs from a 5-point calibration is  $\leq 10\%$  or when the calibration curve passes closely to the point of origin and all check standards pass. Otherwise, careful considerations are needed for reporting results at low levels.

- 7.3.5. If one or more of the compounds has a %RSD of the CFs above the acceptable limit, generate and print out a calibration curve following the steps outlined below:

7.3.5.1. A first order (linear) or quadratic (non-linear) regression may be used for quantitation.

- **Examine the linearity and**
- **the accuracy in quantitating at the low calibration standard and at the point of origin in both options before selecting the better curve (1st order regression may be less accurate than 2<sup>nd</sup> order near the point of origin).**

For Method 608 only, the calibration curve is allowed to be forced through the origin. However, do not force through the point of origin when combined methods are to be used.

7.3.5.2. The Coefficient of Determination ( $r^2$ ) must be  $\geq 0.99$  (Correlation Coefficient ( $r$ )  $\geq 0.995$ ) for the curve to be acceptable. If  $r^2$  is  $< 0.99$  then the instrument must be recalibrated for that compound.

7.3.5.3. When the curve is not forced through the origin, inaccuracies may be present near the low end of the curve. *If reporting down to the MDL is required or if the quantitation of a low response (< lowest calibration standard) from a calibration curve results in a **negative or abnormally positive result (>RL)**:*

7.3.5.3.1. For Method 8081A: re-quantitate the concentration using the corresponding CF of the calibration and flag the results with a "J", indicating an estimated valued. Calculate the mean % RSD of the CFs and ensure that it is  $\leq 20\%$ .

7.3.5.3.2. For Method 608: force the calibration curve through the point of origin and re-quantitate the concentration.



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- 7.3.5.4. When evaluating results near the RL review them carefully to ensure they make sense (i.e. no significantly negative values or false hits when the response area is below the lowest standard). If a result is questionable, the sample should be re-analyzed on another instrument or the result reported as estimated.
- 7.3.6. For 8081 A only: As an alternative to generating a calibration curve or when the generated calibration curve does not meet the acceptance criteria, if the compounds with the %RSD of the CFs higher than 20% (but less than 50%) are known historically to be not detected (ND), calculate the average of all the %RSD of the CFs. If that average is  $\leq$  20%, it is acceptable to quantitate those **non-detect compounds** using their respective CF. Report data with form identifying which compounds did not meet the 20% RSD criteria in the calibration.
- 7.3.6.1. If the % RSD is  $>$  50% for any compound, that compound must be recalibrated regardless of the average % RSD.
- 7.3.6.2. Do not use averaging for normal positive hits.
- 7.3.7. Immediately after the initial calibration, analyze a 2<sup>nd</sup> source verification standard (ICV) at the midpoint. Verify that its recovery is within  $\pm$ 15% and the retention times are within their respective accepted windows.
- 7.3.8. If Toxaphene or Chlordane ICVs do not meet the 15% criteria, the standard is used for pattern recognition only and any positive results must be reanalyzed under a new calibration with an acceptable ICV.
- 7.4. Daily Calibration Verification (CCV)
- 7.4.1. On a daily basis, verify calibration at the beginning of each 12-hour shift by injecting calibration verification standards (CCV) prior to conducting any sample analysis. A verification must also be checked at intervals of 10 to 20 samples – alternated between two different calibration levels (e.g. 100 and 200 ppb) – and at the end of the analysis sequence. CCV standards can be from either the primary or secondary source.
- NOTE: Even though EPA Method 608 does not require calibration verification more than once each working day, this SOP complies to the more stringent criteria of EPA Method 8081A regarding the CCV frequency.
- 7.4.2. On a daily basis, analyze a standard at reporting limit for Toxaphene and Chlordane for pattern recognition purpose at the beginning of each analysis sequence. CCVs for Toxaphene and Chlordane are analyzed only if they are present in the samples and run against its respective calibration.
- 7.4.3. Verify the retention time with the CCVs:
- 7.4.3.1. If retention times have slightly shifted, perform the necessary instrument maintenance and/or reset the RT (but not the RT window).
- 7.4.4. Verify that the recovery of CCV is within  $\pm$ 15% of the initial calibration.

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- 7.4.5. If the CCV fails the acceptance criteria because of a poor injection or because of degraded CCV standard, reprepare and reanalyze a fresh standard once. If significant instrument maintenance or corrective action (beyond daily routine maintenance such as replacement of liner, etc.) is indeed required, two successive passing calibration verifications must be proven before proceeding with samples. Document the corrective action with an internal corrective action report (CAR) and in the instrument log book.
- 7.4.6. For 8081A analysis only: If  $\pm 15\%$  recovery limit is exceeded for any analyte, then the *mean percent difference* of all calibrated analytes can be used. Attach a completed GC Calibration Check Criteria Form. (For 608 analysis, re-calibrate the instrument for the analytes that failed to meet recovery specifications). Provide the following documentation:
- 7.4.6.1. If the sample results are non-detect and the analytes outside of the acceptance limits in the ICV/CCV are  $>115\%$ , the analyst must flag the data with the appropriate qualifier (C1).
- 7.4.6.2. For any analyte which is non-detect in a sample and the ICV/CCV percent recovery is below the acceptance limits ( $<85\%$ ), compare the sample area count to that of the reporting limit standard. If the sample area count is lower, report the sample result as ND with a 'C2' qualifier.
- 7.4.6.3. If any analyte which is a positive hit in a sample and the ICV/CCV percent recovery is outside of the acceptance limits (high or low):
- Reanalyze the sample one time with a passing opening QC.
  - If the ending CCV is still out because of matrix effect, flag the results with the appropriate qualifier and submit a CAR.
- 7.4.6.4. The client will be notified using the form found in Attachment A. The form is completed during the reporting of data using the ELMNT program. The form automatically prints out with the final report.
- 7.4.7. If the CCV result is  $> 115\%$  of the expected value and all samples are ND for the compound then report the results with a C qualifier (C1 if mean percent recovery is used).
- 7.4.8. If the CCV result is  $< 85\%$  of the expected value, evaluate the Reporting Limit standard and compare the sample area counts to the reporting limit standard area counts. If the sample area is  $<$  the RL standard area, report the results as ND with a C5 qualifier (C2 if mean percent recovery is used).

NOTE: If any compound in a sample has a result above the RL, it must be reanalyzed against a calibration that meets the  $\pm 15\%$  CCV criteria.

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- 7.4.9. When a particular sample or project causes any CCV to fail the recovery verification two times, matrix interference is confirmed, the data is reported and flagged with "C7" qualifier and a CAR shall be filed.
- 7.4.9.1. When a suspected matrix interfered sample was bracketed by failing CCVs (opening and ending) in a previous analysis sequence, it only needs to be re-run once with a passing opening CCV.
- 7.4.10. Recalibrate the system in all other cases.
- 7.4.11. All of the criteria listed above for CCV also apply to the analysis sequence from the confirmation column if it is used to report data.
- 7.5. Analysis Procedure
- 7.5.1. Store extracts in crimp-top vials at 4°C until analysis.
- 7.5.2. Verify that the extract has not exceeded its 40 day holding time.
- 7.5.3. Create the daily sequence and retrieve the correct calibration method.
- 7.5.4. Refer to Figure 1 for a typical analysis sequence and to load the autosampler tray.
- 7.5.5. Verify that the solvent bottles in the autosampler are full, and the solvent waste vials are empty.
- 7.5.6. Verify that the instrument blank (hexane) daily is free of contamination.
- 7.5.7. Prime or deactivate the GC column (step 7.2.1).
- 7.5.8. Load and inject an instrument blank (IB) after priming.
- 7.5.9. Analyze a degradation standard containing DDT and Endrin and check for breakdown of DDT and Endrin. Repeat this step every 12-hour shift. (Not applicable for Toxaphene and Chlordane).
- 7.5.10. Proceed with a CCV analysis for single component analytes. Analyze a CCV for Toxaphene or Chlordane only if they are to be reported. Otherwise, analyze a standard at RL for pattern recognition
- NOTE: If the degradation check fails, but the the CCV immediately following this check passes, all subsequent sample results for DDT, DDD, DDE and Endrin, Endrin Aldehyde, Endrin Ketone must have their area counts compared to those of a bracketing reporting limit (RL) check standard analyzed immediately after the CCV. Sample area counts below the RL standard area counts are reported as ND with a proper qualifier (C2). Samples with area counts above the RL standard area count must be re-analyzed.
- 7.5.11. Analyze a RL check standard after every CCV. This is to verify sample results when sample matrix causes low CCV recoveries.
- 7.5.12. Analyze the method blank (MB) and verify that it is free of contamination (<RL).
- 7.5.13. Analyze the LCS and verify that it meets the QC limits.

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- 7.5.14. Follow with up to 10-20 sample extracts.
- 7.5.15. Load an instrument blank after a dirty extract to prevent carry-over. Instrument blanks (and standards) do not count towards the 10-20 samples between CCVs.
- 7.5.16. Analyze the matrix spike (MS) and matrix spike duplicate (MSD) sometime during the sequence. The recoveries of the MS/MSD should be within in-house control limits.

1.	Instrument Blank (Hexane)
2.	Prime
3.	Instrument Blank
4.	Degradation Standard (DDT and Endrin)
5.	CCV1
6.	RL
7.	CCV or RL - Toxaphene
8.	CCV or RL - Chlordane
9.	MB
10.	LCS
11.	10-20 samples (incl. MS/MSD)
12.	CCV2
13.	RL

Figure 1 - A typical analysis sequence

- 7.5.17. Dilute and re-analyze the extract if the responses exceed the calibration range of the system.

Record the sequence in the logbook, and edit the sequence in the computer file.

7.6. Identification of Single-Component Analytes

- 7.6.1. Batch out the samples and QA/QC with the appropriate method file.
- 7.6.2. Identify a single-component analyte by its daily retention time and confirm it on the confirmation column.
  - 7.6.2.1. If the analyte is confirmed, verify that the result from the confirmation column agrees with the primary column ( $RPD \leq 40\%$ ). Otherwise, check for co-elution or other error.
  - 7.6.2.2. If there is an explanation for the discrepancy in quantitated results, report the result from the unaffected column.

NOTE: Confirmation analysis for detected analytes on a secondary column is only required by Method 8081A, but strongly recommended for all pesticides analyses.

- 7.6.3. If no discrepancy is found, re-run the sample on a different instrument or report the higher of the two results.

7.7. Identification of Multi-Component Analytes (Chordane and Toxaphene)

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- 7.7.1. Multi-component pesticides are identified using pattern recognition and characteristic “fingerprint” retention time. Visually compare the Chlordane or Toxaphene chromatogram from the CCV or RL standard to the extract chromatogram. Overlay and expand the chromatograms using TurboChrom’s “compare” feature. Check for retention time shifting by verifying that the surrogates correspond. Expanding the baseline helps in identifying multi-component groups.
- 7.7.2. If Chlordane or Toxaphene is detected above the reporting limit, reanalyze the samples against the 3-5 pt. calibration curve of the identified compound.
- 7.7.3. Quantitate the chlordane or toxaphene based on the prior identification.
- 7.7.4. Submit the sample extract for Florisil cleanup whenever interferences prevent peak detection and identification. Soil samples always undergo the Florisil cleanup.
- 7.7.5. Re-analyze the cleaned extract and re-evaluate the extract chromatogram.
- 7.7.6. After calibrating for either toxaphene or chlordane, edit the sequence, then change the appropriate method file only for the extracts in question.
- 7.8. Quantitation of Multi-Component Analytes (Toxaphene/Chlordane)
  - 7.8.1. These analytes can be quantitated using the total area/time group of the analytes chromatographic pattern or using 3 to 6 major peaks.
  - 7.8.2. When deciding upon which approach to use, consider the sample extract:
    - 7.8.2.1. If there are still interferences present in the extract after cleanup, it is advised to use the 4 to 6 major peaks approach for quantitation.
    - 7.8.2.2. Otherwise, use the total area/time group of the Toxaphene or Chlordane pattern for quantitation.
    - 7.8.2.3. Indicate the quantitation method on the calibration sample file as well as the sample report printout.
  - 7.8.3. Total Area Measurement
    - 7.8.3.1. Construct the baseline of Toxaphene or Chlordane in the sample chromatogram between the retention times of the first and last eluting Toxaphene or Chlordane components in the standard.
    - 7.8.3.2. In order to use the total area/time group approach, the pattern in the sample chromatogram must be compared to that of the standard. Note: If the pattern does not match the standard pattern completely, the sample concentration may be significantly underestimated.

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- 7.8.3.3. **Chlordane Only:** When the GC pattern of a Chlordane residue in a sample differs considerably from that of the Technical Chlordane standard, the analyst may choose to report the sum of all the identifiable Chlordane components as “Chlordane (not otherwise specified)”.
- 7.8.4. Major peaks approach
- 7.8.4.1. Toxaphene and Chlordane may also be quantitated on the basis of 4 to 6 or 3 to 5 major peaks respectively.
- 7.8.4.2. The chosen peaks may not exactly match the sample with the standard in relative areas. However, analyst should not use peaks from the sample chromatogram whose areas appear to be disproportionately larger or smaller relative to the standard peaks.
- 7.8.4.3. If possible compare samples with standards from different suppliers to see if a better match is available.
- 7.8.4.4. The heights or areas of the selected peaks should be added together and used to determine the Toxaphene or Chlordane concentration.
- 7.8.4.5. Alternatively, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene or Chlordane in the standard.
- 7.8.4.6. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the resulting concentrations are averaged to provide the final result for the sample.
- 7.9. Chlordane by 8081A only
- 7.9.1. Method 8081A provides three options for the quantitation of Chlordane
- 7.9.1.1. These options depend upon the end use of the results and the analyst’s skill in interpreting chromatographic patterns for
- 7.9.1.2. These options depend upon the end use of the results and the analyst’s skill in interpreting chromatographic patterns for Chlordane).
- 7.9.1.3. In most cases, the analyst should quantitate Chlordane as Chlordane (not otherwise specified).
- 7.9.1.4. If specifically requested, alpha and gamma chlordanes may be reported individually as they are single component pesticides and may be quantitated directly from pesticide initial calibration.
- 7.9.1.5. If a specific project asks for either Technical Chlordane, or individual Chlordane components, refer to Method 8081A Sec 7.6.2 for detailed information.
- 7.10. Instrument Conditions

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- 7.10.1. The following are suggestive instrument conditions. These may vary slightly between instruments, or because of necessary instrument maintenance (e.g. column trimming) or because of column age.
- 7.10.2. Detectors: Dual Electron Capture Detector (ECD) or Dual Electron Capture Micro ECD.
- 7.10.3. Injection Volume: 1 µl
- 7.10.4. Refer to the following table for additional details:

	Option 1	Option 2
Column A:	J&W, DB-XLB (30m x 530µm)	Restek CLP (30m x 530µm)
Column B:	J&W, DB-608 (30m x 530µm)	Restek CLP2 (30 m x 530µm)
Helium flow rate:	10 mL/min	10 mL/min
ECD make-up Nitrogen:	75 mL/min	75 mL/min
Electronic pressure control:	20	20
Injector t °C:	265 °C	265 °C
Detector t °C:	310 °C	310 °C
Oven initial t °C:	175 °C	110 °C
Initial Hold time:	0 minute	1 minute
Ramp A:	8 °C/min	9 °C/min
Ramp B:	8 °C/min	10 °C/min
Intermediate t °C:	240 °C	285 °C
Intermediate Hold time:	9 minutes	4.5 minutes
Final t °C:	280 °C	280 °C
Final Hold Time:	4 minutes	4 minutes
Run Time:	25 minutes	25 minutes

7.11. Preventative Maintenance

- 7.11.1. If degradation of either DDT or Endrin exceeds 15%, take corrective action (See 7.2.4).
- 7.11.2. To prevent carryover from a dirty extract, load one or two instrument blanks immediately after all dirty extracts. In addition, place an instrument blank before each CCV to help prevent carryover.
- 7.11.3. If an instrument is unusable or has limitation to its use (bad port, not for low level samples, etc), it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution and verification of proper operation into the instrument maintenance logbook.

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7.11.4. Record all performed maintenance in the instrument maintenance logbook.

**8. QUALITY CONTROL**

8.1. Instrument Blank

8.1.1. Analyze an instrument blank daily before analyzing samples and after analyzing highly concentrated samples. Prepare the instrument blank with hexane. Verify that the instrument blank is free of target analytes before analyzing any samples.

8.2. Degradation Standard

8.2.1. Analyze a degradation standard initially according to step 7.2. Verify that the percent degradation of DDT and Endrin does not exceed 15%.

8.3. Surrogate Standard

8.3.1. Calculate the recovery of the surrogates, tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB). Both surrogates should be within the historically generated acceptance limits, however if one surrogate is out in a QC sample (e.g. CCV, LCS, MS/MSD) and the target analytes are within the QC limits, the data shall be deemed acceptable as the target analytes are more critical. Use the recovery of the surrogates to monitor for unusual matrix effects, gross sample processing errors, etc.

8.3.2. Calculate the surrogate recoveries on all samples, blanks, and spikes. Verify that both of the surrogate recoveries are within in-house control limits.

8.3.3. If the recoveries are not within limits, then:

8.3.3.1. Verify that there are no errors in calibration, calculations, or surrogate standard solutions in use.

8.3.3.2. Check instrument performance.

8.3.3.3. Recalculate the data and/or re-analyze the extract if any of the above checks reveals a problem.

8.3.3.4. Re-extract and re-analyze the sample if none of the above are a problem or flag the surrogate with an appropriate 'Z' qualifier and provide a corrective action report.

8.4. ICV/CCV Standard

8.4.1. Analyze an ICV after the initial degradation standard at the beginning of the analytical sequence. Analyze a CCV at a different level after every 10 samples (preferably less than 20 samples) or every 12-hour shift – whatever comes first - and at the end of the analytical sequence.

8.4.2. Verify that the ICV and CCV meet the  $\pm 15\%$  limit. See section 7.4 for corrective actions.

8.4.3. Re-inject all the extracts that were not bracketed by acceptable ICV/CCV.



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- 8.5. Method Blank (MB)
- 8.5.1. Analyze a MB that has gone through the extraction procedure for every batch of 20 samples or less. The method blank results must be below the reporting limit (RL).
- 8.5.2. If a detection above the RL is observed:
- 8.5.2.1. If samples results are greater than 20x the level found in the method blank, the results may be reported and must be flagged with a 'B1' qualifier.
- 8.5.2.2. Samples containing the compound(s) of contamination must be re-extracted/re-analyzed if they are < 20x the level found in the blank. If re-extraction is not feasible, all associated samples results must be flagged "B".
- 8.6. LCS Standard
- 8.6.1. Analyze a LCS that has gone through the extraction procedure for every batch of 20 samples or less. Verify that it meets the laboratory established acceptance limits.
- 8.6.2. Re-analyze the LCS once if any reported parameter fails to meet the acceptance criteria.
- 8.6.3. If the second run of LCS is still outside the acceptance limits, then determine as follows:
- 8.6.3.1. If both runs of LCS result in above the acceptance limits and the sample results are ND, fill out a CAR and report the data with a flag indicating the high LCS recovery. (L qualifier).
- 8.6.3.2. If both runs of LCS result in above the acceptance limits and the sample results are positive, re-extract and re-analyze the affected samples with acceptable QC criteria.
- 8.6.3.3. If both runs of LCS result in below the acceptance limits, re-extract and re-analyze the affected samples.
- 8.6.3.4. Notify Project Manager immediately if there is insufficient sample left to re-extract. Flag samples results and fill out a Corrective Action Report (CAR) if sample results are still to be reported with failed QC.
- 8.6.4. For 8081A only: "Oil" and "product" matrix samples are extracted by waste dilution (EPA 3580 A) with a surrogate-spiked method blank. Bracketing CCVs shall be used in that case as LCS and LCS duplicate.
- 8.7. Matrix spikes (MS/MSD)
- 8.7.1. Analyze a pair of MS/MSD that has gone through the extraction procedure. Verify that it meets the laboratory established determined acceptance limits.
- 8.7.2. If the MS/MSD are outside of the acceptance limits due to matrix effect, flag the reported results with the appropriate 'M' qualifier.

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- 8.7.3. If the MS/MSD are outside of the acceptance limits due to non-matrix related causes (instrument problems, analyst error, etc), re-analyze the samples after taking corrective action. If re-analyzing the MS/MSD is impossible, fill out a CAR with detailed explanation and corrective action and flag the data with the appropriate qualifier.
- 8.7.4. If the sample extract(s) require any cleanup procedure, the associated batch QC (i.e. MB, MS/MSD and LCS) must also undergo the same cleanup procedure with the sample extract(s).
- 8.8. Perform a method detection limit (MDL) study at least initially and on a yearly basis afterwards. Repeat the study after extensive instrument maintenance or a significant change in the method. Perform a method detection limit study by analyzing at least seven extracts over two different days.
- 8.9. Perform a retention time window study every six months by analyzing a triplicate run of a check standard (ICV/CCV) within three consecutive days.
- 8.10. Perform an Initial Demonstration of Capability (IDOC) for each analyst by preparing and analyzing 4 LCS samples with an average recovery within the established acceptance limits. Repeat the process (CDOC) for each analyst on an annual basis.
- 8.11. Calculations
- 8.11.1. Calculate the LCS and MS/MSD percent recoveries (PR) and MS/MSD relative percent difference (RPD) as follows:

$$PR = \frac{(Sp-S)}{Sa} \times 100$$

$$RPD = \frac{|R1-R2|}{(R1+R2)/2} \times 100$$

Where: PR = Percent Recovery  
Sp = Spike Result  
S = Sample Result  
Sa = Spike Amount

R1 = Conc. of MS  
R2 = Conc. of MSD

- 8.11.2. Water and Soil Samples:

$$C_f = C_i \times PF \times DF$$

where C<sub>f</sub> = Final concentration in µg/L or µg/Kg  
C<sub>i</sub> = Concentration in µg/L from instrument  
PF = Preparation Factor  
DF = Any additional Dilution Factor (post-extraction)

- 8.11.3. Determine the % Difference of the CCV using the following equation:

$$\% \text{ Difference} = \frac{[\text{Apparent conc}(\mu\text{g/L}) - \text{True conc}(\mu\text{g/L})]}{\text{True conc}(\mu\text{g/L})} \times 100$$

**9. PAPERWORK FLOW**

- 9.1. Daily

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- 9.1.1. Query the data system ("Elmnt") for the desired analysis codes to determine what needs to be analyzed and print out worklist.
- 9.1.2. Check the work list for sample status and organize the day's schedule.
- 9.1.3. Record the actual run sequence in the 608/8081A Logbook. Use the Comments section to note whether or not a sample is a confirmation, a re-analysis, or a rush. Also include a dilution factor or any other important information.
- 9.1.4. Edit the sequence in the computer. Record the correct analyst and method name in the sequence file.
- 9.1.5. Print reports and their corresponding chromatograms. Perform a manual integration when a chromatogram shows co-elution or interferences. All data should be initialed and dated. (including QC sample and reprocessed samples)
- 9.1.6. Complete an EPA 608/8081A data checklist form during second level review.
- 9.1.7. Complete the GC Calibration Check Criteria form for each analytical sequence.
- 9.1.8. Print the Sequence File Header Information sheet.
- 9.1.9. File the Extraction Logsheet and the Sequence File Header Information sheet together with the raw data.
- 9.2. As Needed
  - 9.2.1. Complete the Standard Log and update the standard section in the data system. Include the following: preparation date, supplier, standard name, lot number, initial concentration, solvent, volume used, final volume, code, final concentration, date disposed or depleted, and your initials.
  - 9.2.2. Complete the standard log in the data system when new standards are prepared. Have it reviewed and signed.
  - 9.2.3. Print the calibrations for both channels.
  - 9.2.4. Print the process files and place them with their corresponding calibrations.
  - 9.2.5. Submit a Corrective Action Report for any non-matrix related analytical problems or variances.
  - 9.2.6. Complete the Instrument Maintenance Logbook whenever necessary by documenting the date that the instrument became inoperable, the steps taken to fix the instrument, any parts replaced, the initials of the analyst, the name of the service company that performed the maintenance, and the date that the instrument became operable again. Refer to Section 7.8 in Method 8081A for suggested chromatographic system maintenance.
- 9.3. Reporting results
  - 9.3.1. Extraction department will create a batch for soil, water and oil samples.

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- 9.3.2. Use "data tool" to electronically transfer the analytical data into the data system.
- 9.3.3. Verify that all sample results and QC results transferred correctly and verify QC acceptance prior to updating the status in the data system to "analyzed".
- 9.3.4. Submit the raw data for second level review. Verify all sample results and QC results transferred correctly and verify QC acceptance prior to updating the status in the data system to "reviewed".

**10. METHOD PERFORMANCE**

- 10.1. Refer to the data system for method detection limits, reporting limits, and control limits.

**11. POLLUTION PREVENTION**

- 11.1. Samples that are hazardous should be segregated and disposed of as hazardous waste.

**12. WASTE MANAGEMENT**

- 12.1. See Waste Management Section of the Laboratory Safety Plan (LSP).
- 12.2. Preserved samples neutralized prior to disposal.
- 12.3. Unused standards are drummed and shipped out for incineration.

**13. METHOD REFERENCES**

- 13.1. EPA Method 608, 40 CFR part 136, Appendix A
- 13.2. EPA Method 8081A, EPA SW-846 Update III, December 1996
- 13.3. EPA Method 8000B, EPA SW-846 Update III, December 1996
- 13.4. Arizona DHS Information Update #37, June 13, 1997

**14. REFERENCE VARIANCES**

- 14.1. EPA Method 608 requires a calibration verification daily while EPA Method 8081A specifies it at the beginning of each 12-hour shift. The laboratory complies to the more stringent criteria specified in EPA method 8081A for this SOP.
- 14.2. Surrogate spikes are not discussed in EPA Method 608. The laboratory follows the criteria specified in EPA Method 8081A in this SOP.
- 14.3. EPA Method 608 does not require the use of confirmation from a secondary column. The laboratory follows the criteria specified in EPA 8081A in this SOP.
- 14.4. Method EPA 608 does not require the breakdown check of DDT and Endrin. The laboratory follows the criteria specified in EPA 8081A in this SOP.

## GC INITIAL CALIBRATION CHECK LIST

### EPA 608/8081A - Organochlorine Pesticides

Analyst: _____	2 <sup>nd</sup> Level Review: _____
Calibration Date: _____	Date: _____
Calibration File: _____	GC #: _____
Method Compliance: <input type="checkbox"/> EPA 608 <input type="checkbox"/> EPA 8081A	

**Analyst Rev    2<sup>nd</sup> Level Rev**


Minimum 5-point calibration – lowest standard at Reporting Limit

**Calibration Factors (CF):**

- RSD <= 10% for each compound or if
- RSD >10% (but <50%): Generate a linear or quadratic curve,  $r^2 \geq 0.99$   
(Do NOT use linear calibration curve for Negative or abnormally Positive results).
- For 8081A only if needed: Average RSD is used and <=20% (Attach GC Initial Cal. Criteria, GC\_ICC.doc).

**2<sup>nd</sup> Source ICV:**

- Immediately after initial calibration
- %Recovery: 85 – 115

Check and verify calibration data for:

- Correct retention times
- Correct area counts
- Saturated chromatographic peaks
- Manual integration (initialed and dated)
- Printed data before and after integration
- Transcription errors
- Calibration print out initialed and dated

Note:                    Attach this form to all daily runs  
                              GC Initial Calibration Check attached (GC\_ICC.doc) if average CF RSD is used

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

# DAILY DATA CHECKLIST

## EPA 608/8081A – Pesticides, Toxaphene, Chlordane

Analyst: _____	2 <sup>nd</sup> Level Review: _____
Analysis Date: _____	Date: _____
Method Compliance: <input type="checkbox"/> EPA 608 <input type="checkbox"/> EPA 8081A	GC #: _____
QC Batches: _____	Primary Channel (A/B): _____
_____	Confirm. Channel (A/B): _____

**Analyst Rev    2<sup>nd</sup> Level Rev**

- |       |       |  |
|-------|-------|--|
| _____ | _____ | Instrument blank before sample analysis : <=Reporting Limit  |
| _____ | _____ | Pesticides: Endrin (aldehyde & ketone) / DDT(DDE & DDD) Breakdown: <=15%   |
| _____ | _____ | <b>ICV/CCV</b> (1 <sup>st</sup> or 2 <sup>nd</sup> source)   |
| _____ | _____ | • At the beginning of every 12-hour shift, every 10-20 samples and at the end of analysis  |
| _____ | _____ | • Two different levels during the daily analysis.  |
| _____ | _____ | • %Recovery = 85 - 115   |
| _____ | _____ | • For Chlordane & Toxaphene, ICV/CCV outside of limits: valid for qualification only   |
| _____ | _____ | Method blank every extraction batch: <= Reporting limit, or  |
| _____ | _____ | Method blank contaminated, but samples ND or > 20 times MB.  |
| _____ | _____ | LCS every extraction batch of 20 samples or less (refer to in-house limits in Elmnt)   |
| _____ | _____ | MS/MSD every extraction batch of 20 samples or less (refer to in-house limits in Elmnt)  |
| _____ | _____ | <u>All samples checked for:</u>  |
| _____ | _____ | • Dilution Factor  |
| _____ | _____ | • Manual integration, transcription errors   |
| _____ | _____ | • Surrogates within limits (refer to in-house limits)  |
| _____ | _____ | • Precision between channels: <= 40 % (or otherwise justified)   |
| _____ | _____ | • First order (linear) regression is NOT used for Negative results or abnormally Positive results (above RL) with low response. Use CF or 2 <sup>nd</sup> order instead. |
| _____ | _____ | • Frequency of 10 (recommended) to 20 between compliant ICV/CCV  |
| _____ | _____ | GC Initial Calibration Checklist attached (8081_Cal.doc)   |
| _____ | _____ | GC Calibration Check Criteria form attached (if average % recovery if ICV/CCV is used)   |
| _____ | _____ | Instrument Blank and Sequence file prints out attached   |
| _____ | _____ | Corrective Action Report   |

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>8081A-Pesticides in Water (EPA 3510C/8081A)</b>								
Prep Method: EPA 3510C		Preservation: 4 C, Cool						
Container: 1 L Amber		Amount Required: 2000 ml			Hold Time: 7 days			
Aldrin	0.030	0.10 ug/l			35 - 120	30	35 - 120	30
alpha-BHC	0.020	0.10 ug/l			45 - 120	30	45 - 120	30
beta-BHC	0.040	0.10 ug/l			50 - 120	30	50 - 120	30
delta-BHC	0.020	0.20 ug/l			50 - 120	30	50 - 120	30
gamma-BHC (Lindane)	0.030	0.10 ug/l			40 - 120	30	40 - 120	30
Chlordane	0.20	1.0 ug/l						
4,4'-DDD	0.030	0.10 ug/l			55 - 125	30	55 - 120	30
4,4'-DDE	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
4,4'-DDT	0.030	0.10 ug/l			55 - 125	30	55 - 120	30
Dieldrin	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
Endosulfan I	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
Endosulfan II	0.040	0.10 ug/l			55 - 120	30	55 - 120	30
Endosulfan sulfate	0.050	0.20 ug/l			55 - 120	30	60 - 120	30
Endrin	0.030	0.10 ug/l			55 - 120	30	55 - 120	30
Endrin aldehyde	0.050	0.10 ug/l			50 - 120	30	55 - 120	30
Endrin ketone	0.040	0.10 ug/l			55 - 120	30	55 - 120	30
Heptachlor	0.030	0.10 ug/l			40 - 115	30	40 - 115	30
Heptachlor epoxide	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
Methoxychlor	0.040	0.10 ug/l			55 - 125	30	55 - 120	30
Toxaphene	1.5	5.0 ug/l						
surr: Tetrachloro-m-xylene					35 - 115			
surr: Decachlorobiphenyl					45 - 120			

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>8081A-Pesticides in Soil (EPA 3545/8081A)</b>								
Prep Method:EPA 3545		Preservation: 4 C, Cool						
Container:4 oz Jar		Amount Required: 100 grams			Hold Time: 14 days			
Aldrin	1.5	5.0 ug/kg			35 - 120	30	45 - 120	30
alpha-BHC	1.5	5.0 ug/kg			35 - 120	30	55 - 120	30
beta-BHC	3.0	5.0 ug/kg			40 - 120	30	55 - 120	30
delta-BHC	1.5	10 ug/kg			40 - 120	30	60 - 120	30
gamma-BHC (Lindane)	1.5	5.0 ug/kg			35 - 120	30	50 - 120	30
Chlordane	10	50 ug/kg						
4,4'-DDD	1.5	5.0 ug/kg			40 - 125	30	60 - 120	30
4,4'-DDE	1.5	5.0 ug/kg			40 - 125	30	60 - 120	30
4,4'-DDT	3.5	5.0 ug/kg			40 - 125	30	60 - 120	30
Dieldrin	1.5	5.0 ug/kg			40 - 120	30	60 - 120	30
Endosulfan I	1.5	5.0 ug/kg			40 - 120	30	60 - 120	30
Endosulfan II	2.5	5.0 ug/kg			45 - 120	30	60 - 120	30
Endosulfan sulfate	2.0	10 ug/kg			40 - 120	30	65 - 120	30
Endrin	2.5	5.0 ug/kg			45 - 120	30	60 - 120	30
Endrin aldehyde	1.5	5.0 ug/kg			25 - 120	30	55 - 120	30
Endrin ketone	2.0	5.0 ug/kg			35 - 125	30	65 - 120	30
Heptachlor	2.0	5.0 ug/kg			35 - 115	30	55 - 115	30
Heptachlor epoxide	2.0	5.0 ug/kg			40 - 120	30	55 - 120	30
Methoxychlor	3.0	5.0 ug/kg			35 - 130	30	60 - 120	30
Toxaphene	75	200 ug/kg						
surr: Tetrachloro-m-xylene					35 - 115			
surr: Decachlorobiphenyl					45 - 120			



Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>608-Pesticides in Water (EPA 608)</b>								
Prep Method: EPA 3510C		Preservation: 4 C, Cool						
Container: 1 L Amber		Amount Required: 2000 ml			Hold Time: 7 days			
Aldrin	0.030	0.10 ug/l			35 - 120	30	35 - 120	30
alpha-BHC	0.020	0.10 ug/l			45 - 120	30	45 - 120	30
beta-BHC	0.040	0.10 ug/l			50 - 120	30	50 - 120	30
delta-BHC	0.020	0.20 ug/l			50 - 120	30	50 - 120	30
gamma-BHC (Lindane)	0.030	0.10 ug/l			40 - 120	30	40 - 120	30
Chlordane	0.20	1.0 ug/l						
4,4'-DDD	0.030	0.10 ug/l			55 - 125	30	55 - 120	30
4,4'-DDE	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
4,4'-DDT	0.030	0.10 ug/l			55 - 125	30	55 - 120	30
Dieldrin	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
Endosulfan I	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
Endosulfan II	0.040	0.10 ug/l			55 - 120	30	55 - 120	30
Endosulfan sulfate	0.050	0.20 ug/l			55 - 120	30	60 - 120	30
Endrin	0.030	0.10 ug/l			55 - 120	30	55 - 120	30
Endrin aldehyde	0.050	0.10 ug/l			50 - 120	30	55 - 120	30
Endrin ketone	0.040	0.10 ug/l			55 - 120	30	55 - 120	30
Heptachlor	0.030	0.10 ug/l			40 - 115	30	40 - 115	30
Heptachlor epoxide	0.030	0.10 ug/l			50 - 120	30	50 - 120	30
Methoxychlor	0.040	0.10 ug/l			55 - 125	30	55 - 120	30
Toxaphene	1.5	5.0 ug/l						
surr: Tetrachloro-m-xylene					35 - 115			
surr: Decachlorobiphenyl					45 - 120			

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
<b>608-Pesticides (LowRL) in Water (EPA 608)</b>								
Prep Method: EPA 3510C		Preservation: 4 C, Cool						
Container: 1 L Amber		Amount Required: 2000 ml			Hold Time: 7 days			
Aldrin	0.0015	0.0050 ug/l			35 - 120	30	35 - 120	30
alpha-BHC	0.0025	0.0050 ug/l			45 - 120	30	45 - 120	30
beta-BHC	0.0040	0.010 ug/l			50 - 120	30	50 - 120	30
delta-BHC	0.0035	0.0050 ug/l			50 - 120	30	50 - 120	30
gamma-BHC (Lindane)	0.0030	0.010 ug/l			40 - 120	30	40 - 120	30
Chlordane	0.030	0.10 ug/l						
4,4'-DDD	0.0020	0.0050 ug/l			55 - 125	30	55 - 120	30
4,4'-DDE	0.0030	0.0050 ug/l			50 - 120	30	50 - 120	30
4,4'-DDT	0.0040	0.010 ug/l			55 - 125	30	55 - 120	30
Dieldrin	0.0020	0.0050 ug/l			50 - 120	30	50 - 120	30
Endosulfan I	0.0020	0.0050 ug/l			50 - 120	30	50 - 120	30
Endosulfan II	0.0030	0.0050 ug/l			55 - 120	30	55 - 120	30
Endosulfan sulfate	0.0030	0.010 ug/l			55 - 120	30	60 - 120	30
Endrin	0.0020	0.0050 ug/l			55 - 120	30	55 - 120	30
Endrin aldehyde	0.0020	0.010 ug/l			50 - 120	30	55 - 120	30
Endrin ketone	0.0030	0.010 ug/l			55 - 120	30	55 - 120	30
Heptachlor	0.0030	0.010 ug/l			40 - 115	30	40 - 115	30
Heptachlor epoxide	0.0025	0.0050 ug/l			50 - 120	30	50 - 120	30
Methoxychlor	0.0035	0.0050 ug/l			55 - 125	30	55 - 120	30
Toxaphene	0.070	0.10 ug/l						
surr: Decachlorobiphenyl					45 - 120			
surr: Tetrachloro-m-xylene					35 - 115			



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**DEL MAR ANALYTICAL  
 STANDARD OPERATING PROCEDURE  
 GASOLINE RANGE ORGANICS (GRO)/BTEX AND MTBE  
 EPA METHOD 8015B/8021B**

Approved By: [Signature] Date: 5/12/05  
**Department Manager**

Approved By: [Signature] Date: 5/12/05  
**Quality Assurance Manager**

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**DEL MAR ANALYTICAL  
STANDARD OPERATING PROCEDURE  
GASOLINE RANGE ORGANICS (GRO)/BTEX AND MTBE  
EPA METHOD 8015B/8021B**

**1. SCOPE AND APPLICATION**

- 1.1. Method 8015B is used to determine the concentration of Gasoline Range Organics (GRO), also known as Total Volatile Petroleum Hydrocarbons (TVPH or TPH), in wide variety of matrices (e.g. ground, surface, and waste water, soil, sediment, sludge, leachates, etc.) by gas chromatography using a Flame Ionization Detector (FID).
  - 1.1.1. Gasoline range organics correspond to the range of alkanes from C<sub>6</sub> to C<sub>12</sub> and cover a boiling point range of approximately 60°C - 200°C.
  - 1.1.2. Gasoline range organics may also be requested in the range of alkanes from C<sub>4</sub> to C<sub>12</sub>.
  - 1.1.3. Each alkane range can be reported by requesting Hydrocarbon Distribution.
  - 1.1.4. Other requested carbon ranges will be quantitated against the C<sub>4</sub>-C<sub>12</sub> calibration
- 1.2. Method 8021B is used to determine volatile organic compounds in a wide variety of matrices (e.g. ground, surface, and waste water, soil, sediment, sludge, leachates etc.) by gas chromatography using a photoionization detector (PID).
  - 1.2.1. For the purposes of this SOP, the compound list has been reduced to: Benzene, Toluene, Ethylbenzene, and Xylenes (o-xylene and m,p-xylenes).
  - 1.2.2. Methyl tert-Butyl Ether (MTBE) is also a target analyte though not specifically listed in method 8021B.
- 1.3. Water samples (Method 5030B) and soils samples (Method 5030A and Method 5035) with low levels of target analytes are loaded onto a purge and trap autosampler. Water samples containing high levels of target analytes are diluted prior to being loaded onto an autosampler. High level soil samples are extracted with Methanol (MeOH) and high level water samples are diluted with ultrapure water.
- 1.4. The samples are purged with Helium, which extracts the target compounds into a vapor phase. The vapor concentrates on to the trap, which is then heated to release the target compounds into the gas chromatograph column.
- 1.5. Analytes are detected with a photoionization detector (PID) and flame ionization detector (FID) in series.
- 1.6. The standard Reporting Limits (RLs) for this method are as follows (other RLs may be specified based on client or regulatory program needs):

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Compound (units from adjusted amount)	Water RL ( $\mu\text{g/L}$ or ppb)	Low Soil RL (mg/kg or ppm)	High Soil RL (MeOH Extract) (mg/kg or ppm)
Benzene	0.30	0.005	0.10
Ethylbenzene	0.30	0.005	0.10
Toluene	0.30	0.005	0.10
o-Xylene			
m,p-xylenes			
Total Xylene	0.60	0.015	0.30
MTBE	10	0.035	0.70
TPH as gasoline	50	1.0	20

**2. DEFINITIONS**

- 2.1. Carbon Range  $\text{C}_6\text{-C}_{12}$  integration starts immediately before the elution of  $\text{C}_6$  and ends immediately before the elution of a  $\text{C}_{13}$  standard.
- 2.2. Carbon Range  $\text{C}_4\text{-C}_{12}$  integration starts immediately after the elution of the methanol peak and before the elution of tert-Butyl alcohol (TBA) and ends immediately after the elution of a  $\text{C}_{13}$  standard.
- 2.3. There are no additional specific definitions associated with this test. See the QAPM and EPA 8000B for general definitions.

**3. APPARATUS AND MATERIALS**

- 3.1. 5, 10, and class A volumetric flasks with stoppers
- 3.2. 10  $\mu\text{L}$ , 50  $\mu\text{L}$ , 100  $\mu\text{L}$ , 250  $\mu\text{L}$ , 500  $\mu\text{L}$ , and 1000  $\mu\text{L}$  syringes
- 3.3. 1 mL disposable (Pasteur) pipettes
- 3.4. Parafilm
- 3.5. Pipettor
- 3.6. 1 mL mininert vials
- 3.7. 9 mL screw top vials
- 3.8. Organic-free reagent water (Ultrapure water)
- 3.9. 10 mL glass syringe with a Luerlock tip
- 3.10. Disposable glass culture tubes (18mm x 150mm)
- 3.11. 40 mL VOA vials
- 3.12. Stir bars
- 3.13. Analytical pan balance
- 3.14. Vortex mixer
- 3.15. JT Baker Sand (Purified, baked and ignited) or equivalent (e.g. other sand Baked @ 400°C for at least 1 hr)

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- 3.16. Scoopula
- 3.17. Mud-Dawgs™ (OI Analytical)
- 3.18. GC HP 5890 or equivalent
- 3.19. OIC 4460A or OIC 4560 Concentrator
- 3.20. DPM-16 or MPM-16 Autosampler (for samples in Sparge Tubes)
- 3.21. EST 8100 Archon Autosampler (for samples in VOA Vials)

**4. REAGENTS AND STANDARDS**

- 4.1. Stock Standards (vendors: Supelco, Restek, Ultra, Absolute, or equivalent):
  - 4.1.1. TPH (5000 µg/mL) – calibrations, and LCS/LCSD
  - 4.1.2. TPH (20000 µg/mL) – CCV
  - 4.1.3. BTEX (2000 µg/mL)/MTBE (30000 µg/mL) Mix – calibrations, LCS and MS/MSD
  - 4.1.4. BTEX (2000 µg/mL)/MTBE (30000 µg/mL) - CCV
  - 4.1.5. BTEX (2000 µg/mL) Mix – Calibrations and LCS and MS/MSD for any samples that do not require analysis for MTBE and for all samples that require analysis for C<sub>4</sub>.
  - 4.1.6. Methanol (MeOH), purge and trap grade, lot tested by GCMS
  - 4.1.7. a, a, a-Trifluorotoluene (aaa-TFT) (2000µg/mL).
  - 4.1.8. 4-Bromofluorobenzene (4-BFB) (2000µg/mL).
  - 4.1.9. 4-BFB/aaa-TFT (2000 µg/mL) mix
- 4.2. C<sub>4</sub>-C<sub>13</sub> Carbon range standard (Alkanes)
- 4.3. C<sub>12</sub> standard (used to identify peaks)
- 4.4. TBA standard (used to identify C<sub>4</sub> Start peaks)
- 4.5. Sodium Bisulfate

**5. SAFETY**

- 5.1. Since all of the hazards of samples and chemicals used in this procedure are not entirely known, strict adherence to safety rules and use of prescribed personal protection equipment is mandatory. The health hazards of the standards, reagents and samples are not entirely known so caution must be exercised in all cases.
- 5.2. Employees performing this procedure must be familiar with the Chemical Hygiene Plan (CHP), and the precautions stated on the appropriate Material Safety Data Sheets (MSDS).
- 5.3. Personal Protective Equipment Required: Safety Glasses, Labcoat, Gloves
- 5.4. Keep Sodium Bisulfate away from Methanol.

**6. INTERFERENCES**

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- 6.1. Contamination may occur when a low concentration sample is analyzed immediately after a high concentration sample. To help prevent contamination, rinse the purge needles and purge valves two to three times with ultrapure water. After rinsing with ultrapure water, analyze a blank. If the target compounds are not present in the blank above the reporting limit then analysis may continue.
- 6.2. Contamination may also occur when a sample contains surfactants. Signs of surfactant are foaming when the sample is purged. After a sample containing surfactants is analyzed, run a rinse. Look carefully for signs of carry over in the samples that are analyzed immediately after the surfactant sample and in the same port the next time it is used. Attaching 1-3 Mud-Dawgs helps minimize the effects of surfactants.
- 6.3. The sample storage area must be free of organic solvent vapors. This is verified bi-weekly with the use of refrigerator blanks analyzed by GC.
- 6.4. MTBE in the spiking solution will interfere with TPH % recovery calculations in the MS/MSD when analyzing down to C<sub>4</sub>. When TPH (C<sub>4</sub>-C<sub>12</sub>) is being reported, use the spike mix without MTBE. See Standard preparation in 8.1.

**7. SAMPLE HANDLING AND PRESERVATION**

- 7.1. Notice that the most common containers for soil samples are brass boring rings, Encore Sampling devices, Sodium Bisulfate preserved VOAs and Methanol preserved VOAs, or 4 oz. jars. For water samples, the required sample containers are 40 mL VOA vials with no headspace.
- 7.2. Check that the sample has not exceeded its holding time.
  - 7.2.1. Soil samples have a holding time of 14 days if in brass sleeves or jars (5030) or if in refrigerated VOA vials preserved with Sodium Bisulfate, or frozen VOA vials with water (5035).
  - 7.2.2. When using Encore samples, EPA Method 5035 states that soil samples must be either analyzed or extracted within 48 hrs. The holding time may be extended to 7 days by storing the samples in a freezer until the time of analysis.
  - 7.2.3. The holding time for soils under the LA Regional Water Quality Control Board Well Investigation Program (WIP) is 7 days regardless of preservation.
  - 7.2.4. The holding time for water samples is fourteen (14) days from the date of collection. Samples should be preserved with HCl to a pH < 2.
    - 7.2.4.1. Using pH test strips, measure the pH of the sample after the sample aliquot is removed for analysis to ensure it was properly preserved.
    - 7.2.4.2. Document the pH on the run log. If the pH >2, document on the run log that the sample was analyzed though improperly preserved (pH >2). Flag the results with a 'P1' qualifier if the pH is between 5-8 or 'P and pH' qualifiers for any other pH.

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7.2.4.3. If, due to the matrix, the sample cannot be preserved with HCl, the holding time is seven (7) days.

- 7.3. If there is any headspace present in a 40 mL VOA vial, document this on the run log and. Flag the results with a 'HS' qualifier. Samples may be re-loaded from a VOA with Headspace without this qualifier if the VOA was opened for the first time within two hours.
- 7.4. Document on the run log if there are any materials present that could affect or contaminate the sample (e.g. black particulates or tape of any kind). Flag the results with a 'A-01' qualifier describing the material.
- 7.5. Flag the results with a 'H' qualifier if the method holding time has been exceeded.
- 7.6. Submit an Internal Record of Communication (IRC) to the project manager and document on the track sheet that the PM was notified if any of these qualifiers are needed, once the run is deemed reportable.

**8. PROCEDURE**

**8.1. Standard Preparation**

Standard	Conc (ppm)	Standard Amount Added	Supplier / Parent Standard	MeOH Added	Use
Primary BTEX/M Stock	2000, 30000	--	Restek	--	--
Primary BTEX/M	10, 150	50 µL	1 <sup>st</sup> BTEX/M Stock	10 mL	Calibration, LCS
Intermediate BTEX/M	0.1, 1.5	10 µL	1 <sup>st</sup> BTEX/M	990 µL	Calibration
Secondary BTEX/M Stock	2000, 30000	--	Supelco	--	--
Secondary BTEX/M	10, 150	50 µL	2 <sup>nd</sup> BTEX/M Stock	10 mL	CCV
BTEX only Stock	2000	--	Restek	--	--
BTEX	10	50 µL	BTEX only Stock	10 mL	LCS
Primary TPH Stock	20000	--	Supelco	--	--
Primary TPH	1000	50 µL	Primary TPH Stock	950 µL	Calibration, LCS
Intermediate TPH	100	100 µL	Primary TPH	900 µL	Calibration
Secondary TPH Stock	5000	--	Restek	--	--
Secondary TPH	1000	200 µL	Secondary TPH Stock	800 µL	CCV
TFT, BFB Stock	2000	--	Accustandard	--	--
TFT, BFB	20	10 µL	TFT, BFB Stock	990 mL	IS/SURR
TFT Stock	2000	--	Supelco	--	--
TFT	20	10 µL	TFT Stock	990 µL	Calibration, Extracts
BFB stock	2000	--	Supelco	--	Extracts
BFB	20	10 µL	BFB Stock	990 mL	Calibration
C12 Stock	200	--	Supelco	--	RT
TBA Stock	1000	--	Supelco	--	RT
C4-C13 Stock	200	--	Restek	--	RT



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- 8.1.1. Prepare Stock Solutions
  - 8.1.1.1. Empty manufacturer ampoule into a clean 1mL mini-inert vial.
  - 8.1.1.2. Label the vials according to the SOP titled “Reagent and Standard Control and Documentation”. Store the standards in the freezer for no more than 6 month or until the volume in the 9 mL vial reaches approximately 2 mL.
- 8.1.2. Prepare 10mL Standards
  - 8.1.2.1. Rinse a pre-cleaned 10 mL class A volumetric flask with MeOH. Fill the flask approximately half filled with MeOH.
  - 8.1.2.2. Using a 50  $\mu$ L or 500  $\mu$ L syringe, add the required volume of standard to the flask. Mix to the flask.
  - 8.1.2.3. With a 1 mL disposable pipette, dilute to 10 mL mark with MeOH. The bottom of the meniscus should be at the line on the volumetric flask. The final concentration is listed in the column above.
  - 8.1.2.4. Transfer the standard to one 9 mL screw cap vial and one 1-mL mininert vial.
  - 8.1.2.5. Label the vials according to the SOP titled “Reagent and Standard Control and Documentation”. Store the standards in the freezer for no more than 1 month or until the volume in the 9 mL vial reaches approximately 2 mL.
  - 8.1.2.6. Enter standard information into ELMNT. If a previous standard is copied, ensure the proper standard name, expiration, analyte concentrations, and solvent and standard lot numbers are included.
- 8.1.3. Prepare 1 mL Standards
  - 8.1.3.1. Using a pre-rinsed 1 mL syringe, inject the amount of MeOH listed above into a 1 mL mininert vial.
  - 8.1.3.2. With a 10  $\mu$ L or 100  $\mu$ L syringe, add the volume of standard listed above into the mininert vial
  - 8.1.3.3. Label the vials according to the SOP titled “Reagent and Standard Control and Documentation”. Store the standards in the freezer for no more than 1 month.
  - 8.1.3.4. Enter standard information into ELMNT. If a previous standard is copied, ensure the proper standard name, expiration, analyte concentrations, and solvent and standard lot numbers are included.
- 8.2. General Instrument Use
  - 8.2.1. Preparing a Sample (QC or Client) for Analysis
    - 8.2.1.1. On a Sparge Tube Based Autosampler

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8.2.1.1.1. Using a 10 mL gas tight syringe, rinse the port with ultrapure water.

8.2.1.1.2. Rinse appropriate syringes (surrogate/IS, spike, CCV or client) with methanol.

8.2.1.1.3. The amount of soil (if any) and 10 mL of water should be put in a sparge tube. This can be all client sample for a low level water analysis, all ultrapure water for a QC analysis, or a mixture of both for a client sample dilution. From here on the tube should be prepared as quickly as possible to prevent loss of analytes through volatilization.

8.2.1.1.4. Any required standards are added to the 10mL solution.

8.2.1.1.5. Soil extracts only need 5.0 µl of the aaa-TFT internal standard (without surrogate standard) to the 10 mL.

8.2.1.1.6. Attach tube to the autosampler.

8.2.1.2. On a VOA Vial Based Autosampler

8.2.1.2.1. Rinse appropriate syringes (surrogate/IS, spike, CCV or client) with methanol.

8.2.1.2.2. The amount of soil (if any) and 10mL of water should be put in an unused VOA vial. This can be all client sample for a low level water analysis, all ultrapure water for a QC analysis, or a mixture of both for a client sample dilution. From here on the vial should be prepared as quickly as possible to prevent loss of sample through volatilization.

8.2.1.2.3. Any required standards are added to the 10mL solution.

8.2.1.2.4. Soil extracts only need 5.0 µl of the aaa-TFT internal standard (without surrogate standard) to the 10 mL.

8.2.1.2.5. Place vial in the autosampler.

8.2.2. Starting Analysis

8.2.2.1. On a 4460A Concentrator

8.2.2.1.1. To run one sample analysis, advance the autosampler to the correct port by pressing the "ADV" button. Ensure that the red light is lit on the black "Auto" button. Press the red "Start" button. Analysis should begin and a green light will come on.

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8.2.2.1.2. To run several analyses automatically, press the black "Auto Set" button. Toggle the switch up once for two consecutive analyses, up twice for three, etc. Then press the black "Auto View" button. Ensure the red light is lit on the black "Auto" button. Press the red "Start" button. Analysis should begin and a green light will come on.

8.2.2.2. On a 4560 Concentrator

8.2.2.2.1. Press the "SPL" button and use the up and down arrows (also say "ON" and "OFF") to switch the flashing cursor between the port to start and the port to end on. To change the port to start or end on, type in the port number and press enter. Once you have set these two fields, the total number of samples to be analyzed will be displayed on the second line. Press the "Clear" button and then the "Start" button to begin analysis. The green light next to the "Start" button will turn on.

8.2.2.2.2. To change a sequence in progress, press the "Hold" button and the red light next to it should come on. Then press the "SPL" button to edit the runs, then press "Start".

8.2.2.3. On the Archon Concentrator

8.2.2.3.1. To run analyses automatically, edit the method by pressing the "Method" button and the "Enter" button until the edit method screen comes up. Then press "Enter" until you are able to put in the "First Vial" and "Last Vial". Press "Method" twice to exit the method editor. To start the autorun, press "Auto" and then "Enter" twice.

8.3. Initial Calibrations

- 8.3.1. Perform initial calibrations and as needed based upon the CCV percent recovery.
- 8.3.2. Prepare new standards prior to each initial calibration if there are not standards available that were made within the 3 weeks prior to calibration.
- 8.3.3. Verify initial calibrations after a change in concentrator parameters and after major maintenance. Recalibrate if verification does not pass.
- 8.3.4. Re-calibrate the instrument after a change in GC parameters and any time the PID lamp or O-ring is replaced. If re-calibration is needed, clean or replace PID lamp and replace O-ring first.
- 8.3.5. Analyze several blanks after performing maintenance to ensure that the instrument is not contaminated.
- 8.3.6. Process a total of 5 calibration points for the TPH and BTEX components (6 points for MTBE, aaa-TFT, and 4-BFB).

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- 8.3.6.1. It is highly recommended that BTEX/MTBE and TPH calibrations are performed consecutively, however calibration for BTEX/MTBE may be re-calibrated without TPH re-calibration if necessary.
- 8.3.6.2. 4-BFB and aaa-TFT calibrations for TPH are taken from the FID readings during the BTEX/MTBE calibration.
- 8.3.6.3. If BTEX/MTBE is calibrated without TPH, then the surrogate calibrations need to be updated in the TPH calibrations (method files) and clearly documented to ensure traceability.
- 8.3.6.4. If TPH requires re-calibration, also recalibrate BTEX/MTBE.
- 8.3.6.5. The lowest standard must support the reported detection limit.
- 8.3.6.6. The RFs from the initial calibration curve must have an RSD  $\leq$  20 % for all reported compounds or the calibration must be repeated.

BTEX+MTBE Calibration Standards									
Calibration Level (ng)			Intermediate Standard Conc. ( $\mu\text{g/mL}$ )				Standard Volume ( $\mu\text{L}$ )		
BTEX	MTBE	4-BFB	BTEX/MTBE		4-BFB	aaa-TFT	BTEX/MTBE	4-BFB	aaa-TFT
--	15	40	0.1	1.5	20	20	10	2	5
2	30	60	0.1	1.5	20	20	20	3	5
20	300	100	10	150	20	20	2	5	5
200	3000	200	10	150	20	20	20	10	5
400	6000	400	10	150	20	20	40	20	5
600	9000	600	10	150	20	20	60	30	5

TPH Calibration Standards		
Calibration Level (ng)	Stock Standard Conc. ( $\mu\text{g/mL}$ )	Standard Volume ( $\mu\text{L}$ )
500	100	5
1500	100	15
5000	1000	5
10000	1000	10
20000	1000	20

8.3.7. Perform the Initial Calibration for BTEX + MTBE

- 8.3.7.1. Inject 5.0  $\mu\text{L}$  of the 20  $\mu\text{g/mL}$  aaa-TFT internal standard solution into 10mL.

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- 8.3.7.2. Inject the appropriate volume of the 20 µg/mL 4-BFB surrogate standard solution.
- 8.3.7.3. Inject the appropriate volume of the 0.1 µg/mL BTEX + 1.5 µg/mL MTBE standard or the 10 µg/mL BTEX + 150 µg/mL MTBE standard.
- 8.3.8. Perform the Initial Calibration for TPH
  - 8.3.8.1. Inject the appropriate amount of the following standards into 10mL for separate analyses.
    - 8.3.8.1.1. Load 0.25 µL of the C<sub>12</sub> marker. This compound often carries over, so analyze first so that instrument will be clean by the time calibration is analyzed.
    - 8.3.8.1.2. Inject 0.5 µL of the C<sub>4</sub>-C<sub>13</sub> carbon range standard. This standard will be compared to the C<sub>12</sub> standard to determine which peak is which.
    - 8.3.8.1.3. Inject 10 µL of the TBA standard.
    - 8.3.8.1.4. Inject 5 µL of the TBA standard. This standard is loaded at two different volumes so the chromatograms will show two different areas to verify the peak.
  - 8.3.8.2. Inject 5 µL internal standard/surrogate mixed solution in 10 mL of water. Then, inject the appropriate volume of the 100 µg/mL and 1000 µg/mL TPH standards.
- 8.3.9. Document Calibrations
  - 8.3.9.1. Fill out Track sheet with all standards used in calibration and the proper sequence of runs. A copy of this will be filed with the calibration and the original will be bound with all track sheets for that instrument.
  - 8.3.9.2. Fill out the calibration checklists (See Attachments) and include all requirements listed on them in the calibration folder. Store calibration folder in the Data Deliverables department until they no longer need it filed. Then return to the file cabinets.
- 8.4. Preparation of a low-level soil sample
  - 8.4.1. Discard one-half inch to one inch of the top layer of the soil sample before removing a portion to analyze. This top layer has lost any volatile analytes it may have contained and therefore should be discarded.
  - 8.4.2. Using a scoopula, weigh 0.25 g to 5.00 g of the sample into a sparge tube or VOA vial. To avoid cross contamination between samples, clean the scoopula between samples and do not touch the soil with gloves.
  - 8.4.3. Mark the tube or vial with the sample number, using a permanent marker. Record the amount of soil to two decimal places.

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- 8.4.4. Immediately, add 10 mL of ultrapure water.
- 8.4.5. Cover the sparge tube with parafilm. Prepare up to 5 samples before loading onto the autosampler. There is no limit to the number of VOA vials that can be prepared before loading onto the autosampler.
- 8.5. Low level Soil samples – From an Encore™ sampling device
  - 8.5.1. Weigh each Encore™ sampling device containing samples to the nearest 0.01 g and record the weight.
  - 8.5.2. Immediately, add 10-mL of ultrapure water
  - 8.5.3. Cover the sparge tube with parafilm. Prepare up to 5 samples before loading onto the autosampler. There is no limit to the number of VOA vials that can be prepared before loading onto the autosampler.
  - 8.5.4. Reweigh the empty Encore™ sampling devices. The difference between the initial weight and the final weight is the actual sample weight used to calculate final results.
- 8.6. Preparation of a low-level water sample
  - 8.6.1. Inject 5.0 µl of the internal standard/surrogate to the 10 mL.
- 8.7. Analysis
  - 8.7.1. Before beginning a batch, analyze a continuing calibration verification (CCV).
  - 8.7.2. Analyze a method blank at the beginning of each batch. Analyze a water blank with water samples, a soil (sand) blank with direct purge soil samples and a MeOH blank with soil samples extracted with MeOH.
  - 8.7.3. Analyze a method blank, LCS, matrix spike and matrix spike duplicate with every batch of 20 samples or less per matrix. If the LCS meets CCV limits, it can also serve as a CCV.
  - 8.7.4. Rinse each port with a 10 mL gas-tight syringe full of ultrapure water by injecting the water through the port and collecting the waste water in a test tube.
  - 8.7.5. Analyze the quality control samples (e.g., LCS) at the beginning of the sequence to validate the analytical batch.
  - 8.7.6. For Aqueous samples, dip pH test paper into the sample remaining in the VOA vial after the sample has been loaded. Record the pH on the sample track sheet ('<2' is acceptable if the pH is 2 or less). If the sample has a pH >2 and was sampled more than seven days earlier, flag the results with a 'P1' qualifier and notify the project manager.
  - 8.7.7. Re-analyze samples on a different GC column or on a GC/MS, when you suspect analytical interferences or when the samples require confirmation, according to section 8.12.
  - 8.7.8. Dilute a sample if:
    - 8.7.8.1. It is a neat solution.

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- 8.7.8.2. It has a strong gasoline odor.
- 8.7.8.3. The chromatogram shows saturation of a target analyte peak.
- 8.7.8.4. A target analyte peak is higher than the highest calibration point.
- 8.7.8.5. Historical or PID data shows high recoveries.
- 8.7.8.6. Perform a smaller dilution, if the target analyte peak is lower than the lowest calibration point.

8.8. Dilutions

- 8.8.1. Dilution of soil samples containing high levels of target analytes
  - 8.8.1.1. Remove another one-half inch of soil from the boring and weigh between 0.25 g and 5.00 g of the sample into a 40 mL VOA vial, using a scoopula.
  - 8.8.1.2. Immediately add 5 mL of MeOH.
  - 8.8.1.3. Label the VOA vial with the sample number and the date.
  - 8.8.1.4. Using a 10 µl syringe, add 5 µl of the 2000 µg/mL 4-BFB surrogate standard (without internal standard) into the 40 mL VOA vial.
  - 8.8.1.5. Cap the VOA vial and mix with a vortex on the fast setting. Let the mixture settle for 10 minutes. If the sample and MeOH have not separated, then centrifuge.
  - 8.8.1.6. To avoid cross contamination between samples, wipe the scoopula clean with a clean Kimwipe before weighing another sample, and do not touch the soil with gloves.
  - 8.8.1.7. Record all information in the Extract logbook.
- 8.8.2. Dilution of soil samples containing high levels of analytes -- From an Encore™ sampling device
  - 8.8.2.1. Weigh each Encore™ sampling device containing samples to the nearest 0.01 g and record the weight.
  - 8.8.2.2. Extrude the sample directly into a VOA vial.
  - 8.8.2.3. Reweigh the empty Encore™ sampling devices. The difference between the initial weight and the final weight is the actual sample weight used to calculate final results.
  - 8.8.2.4. Immediately add 5 mL of MeOH.
  - 8.8.2.5. Label the VOA vial with the sample amount, client code and sample number.
  - 8.8.2.6. Using a 10 µl syringe, add 5 µl of the 2000 µg/mL 4-BFB surrogate standard (without internal standard) into the 40 mL VOA vial.

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- 8.8.2.7. Cap the VOA vial and mix with a vortex on the fast setting. Let the mixture settle for 10 minutes. If the sample and MeOH have not separated, then centrifuge.
- 8.8.2.8. Record all information in the Extract logbook.
- 8.8.3. Prepare the diluted soil sample for analysis
- 8.8.3.1. Depending on the dilution ratio, withdraw 1 µl to 100 µl of the MeOH layer of the diluted soil sample, using the appropriate size syringe and add to the 10 mL.
- 8.8.4. Dilute a water sample containing a high level of target analytes with ultrapure water.
- 8.8.4.1. Using a 10 mL syringe, withdraw the appropriate amount of the high-level water sample to attain a final volume of 10 mL. For example, for a 10:1 dilution, the appropriate amount of ultrapure water is 9.0 mL and the appropriate amount of sample water is 1.0 mL. When adding less than 1 mL of sample use a smaller appropriate syringe.
- 8.8.4.2. Add 5.0 µl of the internal standard/surrogate mixed solution using a 10 µl syringe to the 10mL.
- 8.8.5. Use the Dilution Calculation Chart (See Attachment) to determine the dilution factors, reporting limits, and MDLs for water, soil and methanol extract samples. It is to be used as a quick reference based on standard base reporting limits for high level samples.
- 8.9. Instruments conditions:
- 8.9.1. The following chromatographic columns or an equivalent are used. Each GC has a different column so confirmations may be run on any other GC.
- 8.9.1.1. DB-MTBE 30m .447mm
- 8.9.1.2. RTX-5 30m 0.53mm
- 8.9.1.3. DB-1 30m 0.53mm
- 8.9.1.4. DB-624 30m 0.53mm
- 8.9.1.5. ZB-1 30m 0.53mm
- 8.9.1.6. DB-MTBE 30m 0.83mm
- 8.9.1.7. DB-VRX 30m 0.45mm
- 8.9.2. The following are general instrument conditions. These may vary slightly between instruments due to specific column differences, or because of necessary instrument maintenance (e.g. column trimming) or because of column age.
- 8.9.2.1. Concentrator Conditions

Pre-Heat Time	1.5 min	Pre-Heat Temp.	40°C	Ext Temp	100°C
Purge Time	11 min	Purge Temp	22°C	Inj. Time	0 min



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Dry Purge Time	1.5 min	Pre-Heat Desorb	175°C	Pump Time	0 min
Desorb Time	4 min	Desorb Temp.	185°C	Xfer Temp.	100°C
Bake Time	5 min	Bake Temp.	210°C	Valve Temp.	120°C

8.9.2.2. GC Conditions

Initial Temp.	40°C	Final Temp. A	170°C
Initial Time	2.5 min	Final Time A	1.0 min
Rate	25°C/min	Rate B	25°C/min
Final Temp.	90°C	Final Temp. A	220°C
Final Time	1.0 min	Final Time A	0.17 min
Rate A	15°C/min	Total Run Time	14.00 min

8.10. Maintenance

8.10.1. Preventative

8.10.1.1. Prior to analyzing samples, rinse each port with a 10 mL syringe full of ultrapure water by injecting the water through the port and collecting the waste water in a test tube.

8.10.1.2. Attach a Mud-Dawg to the sparger for Encore samples or samples with surfactants.

8.10.2. General Maintenance

8.10.2.1. After a high concentration sample is analyzed, tag the port for possible contamination and:

8.10.2.1.1. Bake out the trap and system

8.10.2.1.2. Rinse each port with a 10 mL syringe full of ultrapure water by injecting the water through the port and collecting the waste water in a test tube.

8.10.2.1.3. Run a methanol blank with 100 µl of MeOH. If compound is less than the reporting limit the sample port is ready to use. Otherwise repeat from step 8.10.2.1.1.

8.10.2.1.4. If contamination still persists, major instrument maintenance must be performed (e.g. change trap, check port valves, cleaning PID lamp, etc.).

8.10.2.1.5. Record all performed maintenance in the instrument maintenance logbook.

8.10.2.1.6. Replace traps if necessary.

8.10.2.1.7. Replace the carrier gas trap at least annually and record on the maintenance schedule posted on the instrument.

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8.10.2.2.If an instrument is unusable or has limitation to its use (e.g. bad port, not usable for BTEX, not for low-level sample, etc), it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution, and verification of proper operation into the instrument maintenance logbook.

8.11. GRO (TPH) Carbon Ranges

8.11.1. For GRO (TPH), Carbon ranges may be determined by altering the start and end points of the chromatographic integration based upon the retention times of the appropriate carbon marker.

8.11.1.1.A C<sub>6</sub>-C<sub>12</sub> Calibration curve is used for quantitating the analyte "Volatile Fuel Hydrocarbons (C<sub>6</sub>-C<sub>12</sub>)". Crystal Reports' is set up to automatically add the qualifier "Volatile Fuel Hydrocarbons (C<sub>6</sub>-C<sub>12</sub>) are quantitated against a gasoline standard."

8.11.1.2.A separate C<sub>4</sub>-C<sub>12</sub> Calibration curve is used for quantitating the Carbon ranges reported as C<sub>4</sub>-C<sub>12</sub> as well as C<sub>5</sub>-C<sub>12</sub>. Crystal Reports is set up to automatically add the qualifier "GRO (C<sub>4</sub>-C<sub>12</sub>) is quantitated against a gasoline standard. Quantitation begins immediately following the methanol peak."

8.11.1.3.Carbon Range C<sub>6</sub>-C<sub>12</sub> integration starts immediately before the elution of C<sub>6</sub> and ends immediately before the elution of a C<sub>13</sub> standard.

8.11.1.4.Carbon Range C<sub>4</sub>-C<sub>12</sub> integration starts immediately after the elution of the methanol peak and before the elution of tert-Butyl alcohol (TBA) and ends immediately after the elution of a C<sub>13</sub> standard.

8.11.2. Analyze a carbon Range Standard prior to all initial calibrations and following any RT shifts.

8.11.3. Hydrocarbon Distributions

8.11.3.1.HC distribution is a semi-quantitative analysis that a client uses to determine the sources of contamination. Determining specific carbon ranges and quantitating each range based on the peak areas in the carbon range helps to identify the source of the fuel.

8.11.3.2. In general, the quantitation begins with C<sub>4</sub>-C<sub>5</sub>, which begins immediately before the TBA peak and ends immediately before C<sub>6</sub>. C<sub>4</sub> and C<sub>5</sub> co-elute, so are reported in the same range. Quantitation ends with C<sub>12</sub>, which ends immediately before the C<sub>13</sub> peak.

8.11.3.3. There are limitations with this method as there are no practical ways to calibrate each carbon range. Therefore the quantitation is based on the area counts of the total carbon range from C<sub>4</sub>-C<sub>12</sub>. Upon request quantitation can begin with C<sub>6</sub>.

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8.11.3.4. Because the quantitation is based on a total range, the individual ranges do not provide good recovery data. Therefore only the total recovery will be reported for the LCS, MS, and MSD. The Method Blank however will be reported to show contamination of the system in the individual ranges.

8.11.3.5. Each reported sample will have a total C<sub>4</sub>-C<sub>12</sub> range within 15% of the calibrated method. This calculation will not be performed if the total range is below the total reporting limit.

8.12. Confirmations

8.12.1. Positive hits for 8021B (BTEX/MTBE) must be confirmed on an instrument with a different column or by GC/MS.

8.12.1.1. Confirm at least one sample containing each of the analytes per workorder that has the same pattern.

8.12.1.2. Confirmation should agree within 40% of the original result or report the higher of the two values.

8.12.1.3. Confirmations for BTEX analytes are not required if there is a fuel pattern in the sample. This is defined as two BTEX compounds above the calibration level and a TPH result above the RL, or three of the four BTEX compounds above the calibration level. MTBE always needs to be confirmed.

8.12.1.4. Historical data may be used in lieu of confirmation analysis. Attach a copy of historical data for documentation.

8.12.2. Matrix interference on internal standards and surrogates must be confirmed on a GC with a different column if the acceptance criteria in Section 9.3.2. has not been met.

8.12.2.1. Confirm at least one sample per workorder containing similar interference pattern.

8.13. Results Reporting

8.13.1. Create a batch in ELMNT prior to loading the instrument and enter the Surrogate ID. (it can always be edited later if something changes).

8.13.2. Create a bench sheet in ELMNT. Enter the initial sample volumes and verify the final volumes. Enter the QC information: initial and final volumes, spike source, sample source for MS/MSD.

8.13.2.1. Water samples have an initial and final volume of 10mL. TCLP, STLC, SPLP, and DIWET samples have an initial volume of 1mL and a final volume of 10mL. Low level soils will have the weight to two decimal places as the initial weight, and 10mL as the final volume. Soil extracts will have the weight to two decimal places as the initial weight, and 5mL as the final volume.

8.13.3. Use Data tool to transfer the data electronically into ELMNT.

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8.13.4. Verify all sample results and QC results transferred correctly, verify QC acceptance, and add the appropriate qualifiers prior to updating the status in ELMNT to "analyzed".

**9. QUALITY CONTROL**

- 9.1. Analyze a method blank per matrix for every batch of 20 samples, or less.
- 9.1.1. Prepare a water method blank
- 9.1.1.1. Inject 5.0 µl of the internal standard/surrogate spike solution to 10 mL.
- 9.1.2. Prepare a soil method blank
- 9.1.2.1. Weigh 5.0 g or of pre-dried clean sand into a sparge tube or VOA vial.
- 9.1.2.2. Add 10 mL of ultrapure water and 5.0 µl of the internal standard/surrogate solution.
- 9.1.2.2.1. Since a soil method blank contains reagents for both water and soil matrices, it may be used to control combined water and soil batches, not to exceed 20 samples total.
- 9.1.3. Prepare a MeOH blank
- 9.1.3.1. Measure 5 mL of MeOH and 5 g of sand into a 40 mL VOA vial. Add 5 µl of the 2000 µg/mL 4-BFB surrogate solution.
- 9.1.3.2. Add 5.0 µl of the internal standard and 100 µl of MeOH with the 4-BFB added previously to 10 mL.
- 9.1.4. The method blank must be free of contamination below the reporting limit for all target analytes in order to be acceptable. If the method blank shows contamination, reanalyze all samples in the batch unless:
- 9.1.4.1. The samples are ND (fill out a CAR) or
- 9.1.4.2. The sample result is >20x the blank level. Fill out a corrective action report and flag the sample results with a 'B1'.
- 9.1.4.3. The L.A. Water Board testcodes require blank results below the MDL.
- 9.2. Continuing Calibration Verification (CCV)
- 9.2.1. Verify the initial calibration by analyzing a BTEX and/or TPH CCV at the beginning of each day prior to sample analysis.
- 9.2.2. Verify the continuing calibration by analyzing a BTEX and/or TPH CCV after every 10 samples or 12 hours, which ever is sooner, varying the amount of spike used. (The blank and LCS are not included as samples, the MS/MSD are included as samples).
- 9.2.3. Analyze a BTEX CCV
- 9.2.3.1. Inject 5.0 µl of the internal standard/surrogate into the 10mL.

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- 9.2.3.2. For the high level CCV, inject 40 µl of the 10 µg/mL BTEX + 150 µg/mL MTBE or 10 µg/mL BTEX standard for the 400 ng BTEX + 6000 ng MTBE or 400 ng BTEX CCV.
- 9.2.3.3. For the low level CCV, inject 20 µl of the 10 µg/mL BTEX + 150 µg/mL MTBE or 10 µg/mL BTEX standard for the 200 ng BTEX + 3000 ng MTBE or 200 ng BTEX CCV.
- 9.2.4. Analyze a TPH CCV
  - 9.2.4.1. Inject 5.0 µl of the internal standard/surrogate into the 10mL.
  - 9.2.4.2. For the high level, inject 8.0 µl of the 1000 µg/mL TPH standard for the 8000 ng TPH CCV.
  - 9.2.4.3. For the low level, inject 5.0 µl of the 1000 µg/mL TPH standard for the 5000 ng TPH CCV.
- 9.2.5. Compare the CCV results to the predicted value from the initial calibration.
  - 9.2.5.1. If the response for any target analyte, including BFB surrogate for 8021B, varies from the predicted response by more than ± 15% (20% for MTBE), take any necessary corrective action, prepare a new CCV standard, and reanalyze.
  - 9.2.5.2. If the second CCV fails, perform any needed corrective action and either recalibrate or analyze two consecutive acceptable CCV.
  - 9.2.5.3. If the CCV fails high (> 115%) and some samples are ND for the failing compound, the ND data may be reported. The results must be flagged with a 'C' qualifier.
- 9.3. Surrogates are added to each sample, CCVs, method blank, LCS, MS and MSD. Sample surrogate recoveries must fall within the acceptance limits established semi-annually by in-house statistical analysis.
  - 9.3.1. BFB is the surrogate for 8021B and the primary surrogate for TPH (8015B). aaa-TFT is the secondary surrogate for TPH.
  - 9.3.2. If any surrogates are outside of the acceptance limits, determine the cause of the problem and take corrective including reanalysis. Exceptions are:
    - 9.3.2.1. If samples are ND and surrogates are biased high. Reanalysis is not required. Flag surrogate results with 'Z2'.
      - 9.3.2.1.1. Note: The surrogate recoveries are not evaluated in the TPH CCV due to excessive co-elutions from the fuel standard.
    - 9.3.2.2. For TPH, both BFB and aaa-TFT must pass for clean samples and blanks or corrective action must take place, which may include reanalysis. If after reanalysis, one or both surrogates fail in the Method blank, recalibrate the instrument.

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- 9.3.2.2.1. Due to co-elution, LCS-TPH will only be reprocessed for 4-BFB. Co-elution primarily occurs with aaa-TFT, and this compound will not be reprocessed to keep the processing as similar to the calibration as possible.
- 9.3.2.3. If TPH samples have BFB surrogates out due to obvious chromatographic matrix interference and aaa-TFT is acceptable, reanalysis is not required. Flag surrogate results with 'Z5'
  - 9.3.2.3.1. If TPH samples have aaa-TFT surrogate out due to obvious chromatographic matrix interference and BFB is acceptable, reanalysis is not required. Report results unqualified.
- 9.3.2.4. TCE, a common contaminate, elutes very close to aaa-TFT, often co-eluting. When this happens, a sample from the workorder must be confirmed either from another instrument, historical data or from GCMS.
- 9.3.2.5. If both surrogates co-elute, it must be confirmed on another column and posted with a 'Z' or 'ZX' qualifier.
- 9.3.2.6. On occasion, one confirmation may have surrogates within the acceptance limits, but cannot be reported due to QC problems. When this occurs, the sample can be posted from the first analysis if the analyte recovery is within 40%.
- 9.3.3. If necessary (non-matrix related), fill out a corrective action form.
- 9.3.4. If after the reanalysis, the surrogate is still out due to sample matrix, flag the results in ELMNT when reporting results, and attach confirmation to reported chromatogram
- 9.4. Internal Standards are added to each sample, CCVs, method blank, LCS, MS/MSD for 8021B.
  - 9.4.1. Internal standard (aaa-TFT) area counts must fall within 70%-130% of the average area of the calibration standard for Blanks, LCS, and CCVs or corrective action needs to take place. Corrective action may include reanalysis and/or recalibration (if reanalysis fails).
  - 9.4.2. Internal standard recoveries in samples must fall within -50% to +100% of the average area of the calibration standard or corrective action must take place. Samples must be reanalyzed to confirm matrix effects.
- 9.5. Analyze a matrix spike and matrix spike duplicate with every batch of 20 samples or less per matrix.
  - 9.5.1. After analyzing about 8 to 10 samples, if possible, select a sample that does not require a dilution to use for the matrix spike and matrix spike duplicate.
  - 9.5.2. Prepare a soil matrix spike

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- 9.5.2.1. Weigh about the same amount as used for the initial unspiked sample into a labeled sparge tube or VOA vial. Remember to discard the top layer of the soil sample.
- 9.5.2.2. Add 5.0  $\mu$ l of the internal standard/surrogate mixed solution, and add 20  $\mu$ l of the BTEX/MTBE or BTEX standard solution to the 10mL.
- 9.5.2.3. Cover the sparge tube with parafilm or cap the vial.
- 9.5.2.4. Repeat the procedure to prepare a matrix spike duplicate.
- 9.5.3. Prepare a water matrix spike
  - 9.5.3.1. Measure the same volume of sample as used for the initial unspiked sample into a labeled test tube.
  - 9.5.3.2. Add 5.0  $\mu$ l of the internal standard/surrogate mixed solution, and add 20  $\mu$ l of the BTEX/MTBE or BTEX standard solution.
  - 9.5.3.3. Repeat the procedure to prepare a matrix spike duplicate.
- 9.5.4. The acceptance limits for the MS/MSD are determined semi-annually by in-house statistical analysis.
- 9.5.5. If the MS/MSD are outside of the acceptance limits due to matrix effects note on the track sheet and flag the MS/MSD and source sample in ELMNT with the appropriate 'M' qualifier when reporting results.
- 9.5.6. If the MS/MSD are outside of the acceptance limits due to no matrix related issues (e.g. instrument problems or analyst error), re-analyze the MS/MSD if possible, otherwise fill out a CAR with detailed explanation and corrective action.
- 9.5.7. If the RPD between the MS and MSD are outside of the acceptance limits due to matrix effects note on the track sheet and flag the MSD in ELMNT with the appropriate 'R' qualifier when reporting results.
- 9.6. Analyze a Laboratory Control Sample (LCS) with every batch of 20 samples or less per matrix.
  - 9.6.1. Prepare a LCS-BTEX
    - 9.6.1.1. To the 10 mL of water (and sand if for a soil batch), add 5.0  $\mu$ l of the internal standard/surrogate mixed solution, and add 20  $\mu$ l of the BTEX/MTBE or BTEX standard solution.
  - 9.6.2. Prepare a LCS-TPH
    - 9.6.2.1. To the 10 mL of water (and sand if for a soil batch), add 15.0  $\mu$ l of the internal standard/surrogate mixed solution, and add 8  $\mu$ l of the LCS TPH standard solution.
  - 9.6.3. Prepare a LCSD for each LCS if there is not enough sample to perform an MS/MSD. Flag the LCS with a 'M-NR1' qualifier.
  - 9.6.4. Prepare the LCS-BTEX and LCSD-BTEX standard for methanol extractions with each extraction batch.

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- 9.6.4.1. Add 5 mL of MeOH and 5 g sand into two separate 40 mL VOA vial. And add the following.
  - 9.6.4.1.1. Add 5 µl of the 4-Bromofluorobenzene (2000 µg/mL)/mixture
  - 9.6.4.1.2. Add 10 µl of the BTEX standard (2000 µg/mL / 30000 µg/mL) unless MTBE is requested then use BTEX/MTBE mix.
- 9.6.4.2. Vortex as a sample.
- 9.6.4.3. Add 50 µl of MeOH-LCS /LCSD with the 4-BFB added previously into the 10 mL syringe and 5 uL of the aaa-TFT internal standard (without surrogate standard) to the 10 mL.
- 9.6.5. Prepare the LCS-TPH and LCSD-TPH standard for methanol extractions with each extraction batch.
  - 9.6.5.1. Add 5 mL of MeOH and 5 g sand into two separate 40 mL VOA vial. And add the following:
    - 9.6.5.1.1. Add 15 µl of the 4-Bromofluorobenzene (2000 µg/mL) mixture
    - 9.6.5.1.2. Add 40 µl of the TPH standard (20000 µg/mL) unless MTBE is requested then use BTEX/MTBE mix.
  - 9.6.5.2. Vortex as a sample.
  - 9.6.5.3. Add 50 µl of MeOH-LCS /LCSD with the 4-BFB added previously into the 10 mL syringe and 15 uL of the aaa-TFT internal standard (without surrogate standard) to the 10 mL.
- 9.6.6. The acceptance limits for the LCS are determined semi-annually by in-house statistical analysis.
  - 9.6.6.1. If the LCS does not meet the acceptance limits, the samples in the batch must be re-prepared and reanalyzed unless all samples are ND for the failing compound and the LCS recovery is above the acceptance limit. In that case the data may be reported with a CAR and the results must be flagged with an 'L' qualifier.
- 9.7. TCLP, STLC, SPLP and DIWET analysis requires a Method blank to be prepared with the samples. For analysis, load the method blank and then load another method blank with LCS spike as a LCS. Load a sample with LCS spike in addition to the sample as an MS. A MSD is not required for these analyses.
- 9.8. Sample Reanalysis Requirements:
  - 9.8.1. Whenever the internal standard or surrogate is out of acceptance range due to co-elution or matrix interference (see 9.3.2 for exceptions). The acceptance range for the internal standard is in section 9.4, and the acceptance range for the surrogate in samples is determined by in-house statistical analysis ( $\pm 15\%$  for 8021B CCV).
  - 9.8.2. Whenever a sample requires dilution (see step 8.7.10).



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- 9.8.3. Whenever cross contamination (or carryover) exists due to instrument saturation.
- 9.8.4. Whenever results are at the reporting limits thereby requiring confirmation.
- 9.8.5. Refer to client data history (if available) and section 8.12.1.4 when deciding if a sample, containing low level BTEX results, requires re-analysis.
- 9.9. Perform a method detection limit (MDL) study for each matrix (Aqueous and Solid and Extract) initially and after extensive instrument maintenance. An MDL study is accomplished by analyzing 7-8 replicates (over a minimum of two days) of the lowest calibration standard and multiplying the SD by the student T factor (e.g. 3.143 for 7 replicates). In lieu of an annual MDL study, a quarterly MDL verification can be performed by loading one standard at half the reporting limit for each analyte and matrix. It is considered verified as long as the method considers it a detect. See MDL SOP.
- 9.10. Perform retention time window studies at the same time as the MDL study.
  - 9.10.1. Use the CCV check standard from the three previous days to determine the retention time windows.
- 9.11. Perform an initial Demonstration of Capability (DOC) before performing analyses by analyzing 4 LCS samples were the average recovery meets the in-house acceptance limits. If the average does not meet the requirement identify the problem and repeat the process.
- 9.12. Calculations
  - 9.12.1. The formulas for calculating average RF and CF as well as % RSD can be found in Method 8000B.
  - 9.12.2. Calculate the LCS and MS/MSD percent recoveries (PR) and MS/MSD relative percent difference (RPD) as follows:

$$PR = \frac{(Sp-S)}{Sa} \times 100 \qquad RPD = \frac{|R1-R2|}{(R1+R2)/2} \times 100$$

Where: PR =Percent Recovery                      R1 =Conc. of MS  
 Sp = Spike Result                                      R2 = Conc. of MSD  
 S = Sample Result  
 Sa = Spike Amount

- 9.13. Water and Soil Samples
  - 9.13.1.  $C_f = C_i \times PF \times DF$ 

$C_f$  = Final concentration in µg/L or µg/Kg  
 $C_i$  = Concentration in µg/L from instrument  
 PF = Preparation Factor  
 DF = Any additional Dilution Factor
  - 9.13.2. The water RL is based on a 10 mL sample

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9.13.3. The soil (low level) RL is based on a 2 g soil sample.

9.13.4. High Level Soils (MeOH extracts)

$$9.13.4.1. C_f = \frac{C_i \times V_{id}}{S \times V_{inj}}$$

$C_f$  = Final concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$

$C_i$  = Concentration in ng (raw amount) from instrument

$V_{id}$  = Volume used for initial dilution in mL (usually 10 mL)

$S$  = Initial sample weight in g (10g maximum)

$V_{inj}$  = Volume of MeOH injected in mL (0.1 mL maximum)

9.13.4.2. The RL for high level soils (MeOH extracts) is based on a 5g sample, 5mL of MeOH and a 0.1 mL injection volume. Any reduction in the sample size or injection volume will result in a proportional increase in the RL.

9.13.5. Determine the % Difference of the CCV using the following equation:

$$\% \text{ Difference} = \frac{[\text{Apparent conc. } (\mu\text{g/l}) - \text{True conc. } (\mu\text{g/L})] \times 100}{\text{True conc. } (\mu\text{g/l})}$$

## 10. PAPERWORK FLOW

10.1. Daily

10.1.1. Query ELMNT for the desired analysis codes to determine what needs to be analyzed.

10.1.2. Maintain reshot list in the reshot book, marking off samples when reshots are completed.

10.1.3. Complete the Sample Track Sheet as you load each sample onto the autosampler. Use the comments section to note whether or not a sample is a confirmation, a re-analysis or a rush. Also include a dilution amount or any other important information in the comments section.

10.1.4. After each individual sample analysis, the computer automatically prints the corresponding chromatogram. Manually integrate when a chromatogram shows obvious co-elution or interferences. Submit all manual integrations for second level review. See SOP on Manual Integration.

10.1.5. Match the chromatograms with the corresponding Sample Track Sheets and file them by ascending date and ascending gas chromatograph number.

10.1.6. File a copy of the work list with the corresponding daily sequence logs.

10.2. As Needed

10.2.1. Print initial calibrations and file them in separate colored folders for easy retrieval.

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- 10.2.2. Enter the standards information into ELMNT. Check that the supplier, methanol lot, expiration date, and amount are correct. Store the solution in the refrigerator.
- 10.2.3. Complete a Corrective Action Report (CAR) when any QC samples (internal standard, surrogate, MS or MSD) are out of acceptable limits due to non-matrix related causes.
  - 10.2.3.1.A CAR is also required in the event of method blank contamination or surrogate failure and unacceptable LCS recovery.
  - 10.2.3.2. Complete the CAR, and include a printout with the data.
- 10.2.4. Document all other analytical problems in a Corrective Action Report also.
- 10.2.5. Complete an instrument log for each instrument. Include the date that the instrument became inoperable, the steps taken to fix the instrument, replacement parts, the initials of the analyst or the name of the service company that performed the maintenance, and the date that the instrument became operable again and what was used to indicate proper performance (e.g. ICAL).

**11. METHOD PERFORMANCE**

- 11.1. See attached analysis codes for information regarding control limits, reporting limits, and method detection limits.

**12. POLLUTION PREVENTION**

- 12.1. Samples that are considered hazardous should be segregated and disposed of as hazardous waste.
- 12.2. Samples that are acidic should be neutralized prior to sample disposal.

**13. WASTE MANAGEMENT**

- 13.1. See Waste Management Section of the Laboratory Safety Plan (LSP).
- 13.2. Preserved samples neutralized prior to disposal.
- 13.3. Unused standards are drummed and shipped out for incineration.

**14. METHOD REFERENCES**

- 14.1. EPA Method 8015B, EPA SW-846 Update III, December 1996
- 14.2. EPA Method 8021B, EPA SW-846 Update III, December 1996
- 14.3. EPA Method 8000B, EPA SW-846 Update III, December 1996
- 14.4. EPA Method 5030A, EPA SW-846 Update I, December 1992
- 14.5. EPA Method 5030B, EPA SW-846 Update III, December 1996
- 14.6. EPA Method 5035, EPA SW-846 Update III, December 1996
- 14.7. DHS-GC-FID, CADHS LUFT Manual, October 1989

**15. REFERENCE VARIANCES**

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- 15.1. Method 8015B has the GRO carbon range from C<sub>6</sub>-C<sub>10</sub>, however, the carbon range of C<sub>4</sub>-C<sub>12</sub> is used as the standard range as it is consistent with the DHS LUFT method.
- 15.2. The California Regional Water Quality Control Board requires a Carbon Range of C<sub>4</sub>-C<sub>12</sub> to be reported. This SOP allows for that requirement.
- 15.3. Method 8021B does not have MTBE in the compound list, however, MTBE is being analyzed using this method.

SOP Number: GCMS-SVOA.SOP  
Revision: New

Effective Date: 07/13/07  
Supersedes: 8270C.SOP, rev. 1 (03/06/01)  
625.SOP, rev. 3 (03/06/01)

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EPA METHODS SW8270C & 625**

Approved By:  Date: 7/11/07  
Department Manager

Approved By:  Date: 7/10/07  
Quality Assurance Manager

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**1. SCOPE AND APPLICATION**

- 1.1. Method 8270C is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Method 625 is used to determine the concentration of semivolatile organic compounds in extracts prepared from waste water and ground water only.
- 1.2. These methods can be used to quantitate most basic, neutral and acidic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a slightly polar fused silica capillary column.
- 1.3. Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup procedures. For Method 625, samples are extracted in accordance with the procedure outlined in the determinative method. For Method 8270C, solids and waters are extracted in accordance with appropriate extraction and clean-up procedures detailed in the EPA Method 3500 and 3600 series.
- 1.4. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract and for their qualitative and quantitative analysis by mass spectrometry.
- 1.5. See attached Analysis codes for applicable analyte list and reporting limits.

**2. DEFINITIONS**

- 2.1. There are no additional specific definitions associated with this test. See the QA Manual and methods SW8270C and 625 for general definitions.

**3. APPARATUS AND MATERIALS**

- 3.1. Equipment and Supplies
  - 3.1.1. 1 ml vials with PTFE-lined screw caps
  - 3.1.2. Fume hood
  - 3.1.3. 10 µl, 100 µl, 250 µl, 500 µl, and 1000 µl micro syringes
  - 3.1.4. Class A volumetric flask with ground glass stopper (10 ml)
  - 3.1.5. Disposable Pasteur Pipets and Bulbs
  - 3.1.6. Target vials with crimp tops
  - 3.1.7. Target vial crimper and cap remover
- 3.2. Reagents and Standards

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- 3.2.1. The vendors indicated below currently supply the following standards and reagents. In the instance where a different vendor might be used, an equivalent product will be purchased. As long as the item is documented clearly in a logbook or on the raw data, this SOP will remain valid though the vendor may have changed.
- 3.2.2. Methylene Chloride (MeCl<sub>2</sub>), lot-tested
- 3.2.3. Crescent Chemical Benzidines Mix 2 (2000 µg/ml), different lot number for LCS
- 3.2.4. Crescent Chemical Acids Mix (2000 µg/ml), different lot number for LCS
- 3.2.5. Crescent Chemical Base/Neutrals Mix 1 (2000 µg/ml), different lot number for LCS
- 3.2.6. Crescent Chemical Semi-Volatile Mix (2000 µg/ml), different lot number for LCS
- 3.2.7. Restek Internal Standard Mix (2000 µg/ml)
- 3.2.8. Restek Acid Surrogate Standard Mix (10,000 µg/ml)
- 3.2.9. Restek Base/Neutrals Surrogate Standard Mix (5,000 µg/ml)
- 3.2.10. UltraScientific, DFTPP, Pentachlorophenol, Benzidine and DDT Tuning Standard Mix (1000 µg/ml)

**4. SAFETY**

- 4.1. Since all of the hazards of samples and chemicals used in this procedure are not entirely known, strict adherence to safety rules and use of prescribed personal protection equipment is mandatory. The health hazards of the standards, reagents and samples are not entirely known so caution must be exercised in all cases.
- 4.2. Employees performing this procedure must be familiar with the Chemical Hygiene Plan (CHP), and the precautions stated on the appropriate Material Safety Data Sheets (MSDS).
- 4.3. Personal Protective Equipment Required: Safety Glasses, Labcoat, Gloves

**5. INTERFERENCES**

- 5.1. Raw GC/MS data from all blanks, samples and spikes must be evaluated for interferences. Determine if the source of the interferences is from the preparation and/or instrument, and take corrective action to eliminate the problem. Most interferences occur when analyzing samples with high hydrocarbon contamination. Another source of interferences could result if samples have large amounts of non-target analytes.

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- 5.2. Contamination may occur when a low concentration sample is analyzed immediately after a high concentration sample. To help prevent contamination, analyze a blank before and/or after the highly contaminated sample. If the target compounds are not present in the blank then analysis may continue.

**6. SAMPLE HANDLING AND PRESERVATION**

6.1. Sample Containers and Preservation

6.1.1. Waters are to be collected in 1 liter glass amber bottles without preservation.

6.1.2. Soils are to be collected in brass or stainless steel boring tubes, and/or glass 4-ounce jars.

6.2. Storage temperature and Holding times

6.2.1. Both waters and soils are to be stored at  $>0^{\circ}\text{C}$  to  $6^{\circ}\text{C}$  until extracted. Extracts must be stored at  $-10^{\circ}\text{C}$  to  $-20^{\circ}\text{C}$  until analyzed.

6.2.2. The holding time for waters is 7 days from collection to extraction and 40 days from extraction to analysis.

6.2.3. The holding time for soils is 14 days from collection to extraction and 40 days from extraction to analysis.

**7. PROCEDURE**

7.1. Standard Preparation

7.1.1. Prepare the calibration standards, LCS standard, internal standard and surrogate standard every twelve months or sooner.

7.1.2. Transfer the vendor-supplied stock standards from their 1 ml ampules to 1 ml vials with a PTFE-lined screw caps. Label the vials and enter the information into Element and ensure each is peer-reviewed. Store the stock standards in the freezer ( $-10$  to  $-20^{\circ}\text{C}$ ) for no longer than twelve months. Replace the standards when a change in response is observed.

7.1.3. Work under a hood when handling stock standards containing high concentrations of toxic analytes. Note that many of the compounds present in the standard mixes are classified as known or suspected carcinogens and should be treated as potential health hazards. Keep exposure to these chemicals to a minimum. Treat all compounds, not just carcinogens, as potential health hazards.

7.1.4. Tuning standard

7.1.4.1. Inject 950  $\mu\text{l}$  of  $\text{MeCl}_2$  into a target vial.

7.1.4.2. Add 50  $\mu\text{l}$  of 1000  $\mu\text{g}/\text{ml}$  UltraScientific Tuning Standard Mix to the target vial to obtain a final concentration of 50  $\mu\text{g}/\text{ml}$ .

7.1.4.3. Label the vial and enter the information into Element. Store the vial in the freezer. Set expiration date at 12 months.



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7.1.5. Calibration standards

7.1.5.1. Intermediate ICAL Stock (200ppm)

A 200ppm ICAL stock solution, 10ml final volume, is prepared as follows:

1ml Benzidine St.

1ml Acid Mix Std

1ml Base/Neutral Mix

1ml Semi-Vol Mix

200ul Acid Surrogate

400ul Base/Neutral Surrogate

5.4ml MeCl<sub>2</sub>

7.1.5.2. Inject MeCl<sub>2</sub>, the ICAL stock standard and the internal standard into 1-ml target vials with screw cap tops.

7.1.5.3. Refer to Table 1 for the appropriate volumes.

7.1.5.4. Check the calibration standards frequently for signs of degradation or evaporation.

**Table 1: Calibration Standards**

<b>Std Conc (mg/L):</b>	<b>2</b>	<b>5</b>	<b>10</b>	<b>50</b>	<b>80</b>	<b>120</b>	<b>160</b>
MeCl <sub>2</sub> (μl):	970	955	930	730	580	380	180
200ppm ICAL Stock	10	25	50	250	400	600	800
<b>Internal Std. (μl):</b>	<b>20</b>	<b>20</b>	<b>20</b>	<b>20</b>	<b>20</b>	<b>20</b>	<b>20</b>

7.2. Prepare the Laboratory Control Sample (LCS) standard at a concentration of 50 mg/L in the same way that the 50 mg/L calibration standard is prepared except use different vendor standards and/or a different lot number than those used for the calibration standards.

7.3. Calibration and Verification

7.3.1. Tune

7.3.1.1. Perform a DFTPP tune on the mass spectrometer prior to performing the initial calibration.

7.3.1.2. Tune the mass spectrometer by injecting 50 ng of DFTPP (1 μl of 50 μg/ml Tuning Standard).

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- 7.3.1.3. The mass spectrometer must produce a mass spectrum that meets all the relative ion abundance criteria for DFTPP. See Table 14.1.
- 7.3.1.4. If the DFTPP mass spectrum does not meet all the criteria, then replace the septa and injection liner, bake the column and try the DFTPP tune again.
- 7.3.1.5. If the criteria are still not met, then perform a FC-43 (perfluorotributylamine) autotune followed by a DFTPP tune. Tuning with FC-43 is a last resort since this compound prematurely contaminates the ion source.
- 7.3.1.6. Benzidine and pentachlorophenol tailing factors must be  $\leq 3$  and  $\leq 5$  respectively.
- 7.3.1.7. In addition, check for DDT degradation. It must be  $\leq 20\%$ . If poor chromatography is noted, clean the injection port and trim off the first 6-12 inches of the column. Record in the maintenance logbook.
- 7.3.1.8. **Note:** All subsequent standards, MS/MSD, samples and blank associated with the DFTPP tune must use identical mass spectrometer instrument conditions as the DFTPP tune.
- 7.3.2. Initial Calibration
- 7.3.2.1. Perform initial calibrations on an as needed basis, after a FC-43 tune, and after major instrument maintenance. The surrogates are calibrated in conjunction with the calibration standards.
- 7.3.2.2. After analysis of all standards, remove the pierced screw cap of the target vial and replace it with a new screw cap. Place all standards back in the freezer as soon as possible.
- 7.3.2.3. ICAL Requirements
- 7.3.2.3.1. The ICAL must consist of a minimum of 5 points (6 for quadratic) with a %RSD for the average response factor  $\leq 15\%$  (except CCCs) and all SPCC and CCC criteria met.
- 7.3.2.3.2. If one or more of the compounds, other than the CCCs, has a %RSD  $> 15\%$ :
- If the compounds  $> 15\%$  RSD are known historically to be Not Detected (ND), calculate the average RSD for all compounds and if  $< 15\%$  quantitate using the average RF. (ND samples only)
  - Alternatively, generate a calibration curve using a linear or second-order regression formula

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- If the compounds that are > 15% RSD are known historically to have positive hits, a first order (linear) or second order (quadratic) regression may be used for quantitation (not forced through the origin).

7.3.2.3.3. The System Performance Check Compounds (SPCCs) are analyzed with the initial calibration curve, and must meet acceptance criteria before the calibration curve is used. The SPCCs are checked for a minimum average relative response factor. The SPCCs are the first analytes to show poor performance. If the minimum RF are not met, the system must be evaluated and corrective action taken before sample analysis.

7.3.2.3.4. The Calibration Check Compounds (CCCs) are used to evaluate the calibration from the standpoint of the integrity of the system. If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

#### 7.3.2.4. Linear and Quadratic curves

7.3.2.4.1. Print out a linear curve (5pts minimum) and a quadratic curve (6 pts minimum).

7.3.2.4.2. The Coefficient of Determination ( $r^2$ ) must be  $\geq 0.99$  (Correlation Coefficient ( $r$ )  $\geq 0.995$ ) for the curve to be acceptable. If  $r^2$  is  $< 0.99$  then the instrument must be recalibrated for that compound.

7.3.2.4.3. If the software does not calculate  $r^2$  quadratic curves, use the linear curve  $r^2$  for validation.

7.3.2.4.4. If  $r^2$  for the linear curve is at least 0.99 (rounded to 2 sig. figs.) then use the curve that gives the y intercept closest to zero (where applicable).

#### 7.3.2.5. Special Criteria for low-end of curves

7.3.2.5.1. Since the curve is not forced through the origin, inaccuracies may be present near the low end of the curve or negative values may be obtained at the reporting limit.

7.3.2.5.2. The cause is sometimes related to the slight bending in the curve at higher analyte concentrations and a 1<sup>st</sup> order curve is used. If this is the case the a 2<sup>nd</sup> order fit will take care of the problem.

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7.3.2.5.3. If "J" flags are required and the quantitated result from a calibration curve is  $< 0$  (negative value), requantitate the concentration using the average RF from the calibration curve.

7.3.3. Initial Calibration Verification

7.3.3.1. Verify the calibration initially, immediately following the calibration procedure, using a second-source standard.

7.3.3.2. Analyze a standard at the midpoint calibration range using a secondary source standard that has not been processed through the extraction procedure. The second source midpoint standard must meet the following criteria:

7.3.3.2.1. All recoveries within statistically-derived in-house historical limits. (Calculate acceptance limits for the non-CCC analytes by performing a statistical evaluation of 20 ICVs. Use  $\pm 3$  SD for the acceptance limits)

7.3.3.2.2. CCCs and SPCCs within acceptance limits

7.3.4. Continuing Calibration Verification

7.3.4.1. Perform a DFTPP tune and analyze a midpoint check standard every 12 hours, before any samples are analyzed. After analysis of all standards, remove the pierced crimp top of the target vial and replace it with a new crimp top. Place all standards back in the freezer as soon as possible.

7.3.4.2. Perform tune and any necessary corrective action in the same manner as outlined above for initial calibration.

7.3.4.3. Analyze a midpoint check standard by injecting 1 uL of the 50 mg/L calibration standard.

7.3.4.4. Check that the performance criteria of the SPCCs and CCCs of the midpoint check standard are met prior to sample analysis. (See Table 3.)

7.3.4.5. Check that the percent recoveries of all non-CCC analytes are within acceptance limits. (Calculate acceptance limits for the non-CCC analytes by performing a statistical evaluation of 20 CCVs. Use  $\pm 3$  SD for the acceptance limits.)

7.3.4.5.1. The non-CCC analytes should have an average percent difference within the acceptance limits previously established. This would include the SPCCs.

7.3.4.5.2. If any midpoint analyte result is greater than the high acceptance limit of the expected value and all samples are ND for the compound then report the results with a CAR and flag the results with a 'C' qualifier.

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7.3.4.5.3.If any midpoint analyte result is below the acceptance limit, reanalyze the standard one time and recalibrate if the criteria is still not met.

7.3.4.5.4.**FOR SW8270C ONLY**--If any analyte exceeds the criteria, an average percent recovery < 15% may be used to accept the analytical run. A GCMS calibration Check Criteria form must be utilized if this condition exists (see appendix).

7.3.4.5.5.Determine that the retention times for any internal standard have not changed by more than 30 seconds from the mid-point standard level of the most recent initial calibration.

7.3.4.5.5.1.If the retention times for any internal standard exceeds the 30 second change, inspect the system for error. If the shift is due to routine guard column trimming only, analysis can continue. Update the IS RT as needed.

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7.3.4.5.6. Determine that the absolute areas of the quantitation ions of the internal standards in the CCV check standard have not changed by a factor of two (-50% to +100%) from the areas measured during the most recent initial calibration (mid-point standard level). If the areas have changed by more than these amounts, perform necessary maintenance and recalibrate.

7.4. Analysis Procedure

- 7.4.1. Notice that 1000 ml or 15 g of sample is typically used for the extraction of water and soil samples respectively. A smaller sample aliquot may be used for highly contaminated soils.
  - 7.4.1.1. Soil samples are extracted with a 1:1 mix of MeCl<sub>2</sub>/acetone.
  - 7.4.1.2. Water samples are extracted with methylene chloride.
  - 7.4.1.3. The final volume for soil extracts is 1.0 ml and water extracts is 2.0 ml.
  - 7.4.1.4. A larger final volume (e.g.: 5.0 ml or 10.0 ml) may be used for sample extracts which contain a high concentration of hydrocarbons and can not be completely reduced to 1.0 ml.
- 7.4.2. Store sample extracts in the refrigerator until they are analyzed. Allow the extracts to warm to room temperature before analysis.
- 7.4.3. Perform a DFTPP tune and analyze a midpoint and a method blank before analyzing any samples.
- 7.4.4. Add 40 µg/ml (20 µl of 2000 µg/ml) internal standard solution to each 1.0 ml sample, blank, spike and LCS extract prior to analysis.
- 7.4.5. If an extract has a final volume of 2.0 ml, split the extract into two 1.0 ml portions.
  - 7.4.5.1. Add 20 µl of internal standard to one 1.0 ml portion of the extract prior to analysis.
  - 7.4.5.2. Save the other 1.0 ml portion for subsequent analysis or dilutions.
  - 7.4.5.3. Alternatively, a 1.0 ml sample extract may be split into two 500 µl portions.
  - 7.4.5.4. Add 10 µl of internal standard to one 500 µl portion of the extract prior to analysis.
  - 7.4.5.5. Save the other 500 µl portion for subsequent analysis or dilutions.

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- 7.4.6. Analyze sample and QC extracts by placing the extracts in the autosampler tray and programming the autosampler to analyze all samples and QC extracts in the tray. Analyze extracts in the following order:

DFTPP tune (50 ng)  
Midpoint Check Sample (50 µg/L)  
Method Blank  
Laboratory Control Sample (50 µg/L)  
Samples  
Matrix Spike  
Matrix Spike Duplicate

- 7.4.7. Instrument blanks of MeCl<sub>2</sub> may be analyzed after dirty samples or at the end of the sequence as needed to check for instrument contamination.

- 7.4.8. If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

- 7.4.9. If an extract is dark in color or otherwise appears highly concentrated, use a micro syringe to dilute the extract with MeCl<sub>2</sub> into another 2.0 ml target vial. Add 20 µl of internal standard to the diluted extract prior to analysis. If a highly contaminated extract needs dilution after internal standards are added, use a micro syringe to dilute the extract with MeCl<sub>2</sub> into another 2.0 ml target vial. Calculate the appropriate volume of 2000 µg/ml internal standard mix needed to adjust the concentration of internal standard in the diluted extract to 40 µg/ml by using the following formula:

internal standard volume = 20 µl x (1-(1/D)), where D = the dilution factor

- 7.5. Instrument Conditions

- 7.5.1. Instrument Conditions may vary slightly between units. See Table 14.3 for typical settings used for current laboratory instrumentation.

- 7.6. Preventative Maintenance

- 7.6.1. Record all performed maintenance in the instrument maintenance logbook.

- 7.6.2. Daily, or as needed, a standard at the midpoint level and/or a tune standard are analyzed to evaluate preventative maintenance needs. These injections are for diagnostic purposes only and are not used in any analytical sequences.

- 7.6.3. Clean injector port with methylene chloride when necessary. Use a cotton swab to reach inside the injector chamber.

- 7.6.4. Replace septum at least once a week.

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- 7.6.5. Inspect O-ring seal and replace if necessary.
- 7.6.6. Trim column 2-6 inches depending on samples analyzed.
- 7.6.7. Replace gold seal if necessary.
- 7.6.8. Replace pre-column if necessary.
- 7.6.9. If an instrument is unusable or has limitation to its use, it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution, and verification of proper operation into the instrument maintenance logbook.
- 7.6.10. Replace injector septa and deactivate liners when necessary.
- 7.6.11. Clean the ion source and replace filaments when necessary.
- 7.6.12. Change the oil in the foreline pump as needed.
- 7.6.13. Check the diffusion pump oil at least annually and replace as necessary and record on the maintenance schedule posted on the instrument.
- 7.6.14. Replace the carrier gas trap as needed.
- 7.6.15. If an instrument is unusable or has limitation to its use (bad port, not for low level samples, etc), it must be tagged accordingly until such a time the problem has been corrected.

**8. QUALITY CONTROL**

8.1. QC samples

- 8.1.1. Analyze a method blank with every batch of 20 samples or less. The method blank results should be below the practical quantitation limit. Analyze a MeCl<sub>2</sub> instrument blank as needed to check for instrument or solvent contamination (i.e. method blank for solvent dilution analyses).
  - 8.1.1.1. The method blank results must be below the reporting limit (RL). If a detection above the RL is observed, and associated samples detect the same compound, flag results with "B". Samples containing the compound(s) of contamination must be reextracted if they are < 10x the level found in the blank.
- 8.1.2. Analyze an LCS with every batch of 20 samples or less. An LCS is a second source standard to monitor the accuracy of the calibration and the efficiency of the analytical method without potential sample interferences.
  - 8.1.2.1. The acceptance limits for the LCS are determined semi-annually by in-house statistical analysis. If any reported LCS compound exceeds the pre-established limits:
  - 8.1.2.2. If the LCS is out above the acceptance limits and the sample results are ND, fill out a CAR and flag with an 'L' qualifier and report the data.



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- 8.1.2.3. If the LCS is out of the acceptance limits and the sample results are positive, re-extract and reanalyze the samples with acceptable QC. Investigate the source of the problem and take corrective action.
- 8.1.3. Analyze a matrix spike/matrix spike duplicate (MS/MSD) with every batch of twenty samples, or less.
- 8.1.3.1. The acceptance limits for the MS/MSD are determined semi-annually by in-house statistical analysis.
- 8.1.3.2. If the MS/MSD are outside of the acceptance limits due to matrix effects note on the run log "matrix effects" and flag the MS/MSD results in ELMNT with the appropriate 'M' qualifier.
- 8.1.3.3. If the MS/MSD are outside of the acceptance limits due to instrument problems or due to analyst error, re-analyze the MS/MSD if possible, otherwise fill out a CAR with **detailed** explanation and corrective action. Flag the MS/MSD with the appropriate 'M' qualifier.
- 8.1.4. Calculate the surrogate recoveries on all samples, blanks, and spikes. The surrogates must be within the historical acceptance limits. Use the recovery of the surrogates to monitor for unusual matrix effects, gross sample processing errors, etc.
- 8.1.4.1. Verify that the surrogate recoveries are within in-house control limits.
- 8.1.4.2. Sometimes the surrogates are low or unrecoverable due to dilution, cleanup, and/or emulsion formation during extraction.
- 8.1.4.3. If the recoveries are not within limits, then
- Verify that there are no errors in calculations, surrogate solutions and calibration
  - Check instrument performance
  - Recalculate the data and/or re-analyze the extract if any of the above checks reveals a problem.
  - If any surrogates are outside of the acceptance limits, determine the cause of the problem and take corrective action. If the cause is not due to obvious chromatographic matrix interference, the sample must be reanalyzed to confirm matrix effects.
  - Re-extract and re-analyze the sample if none of the above are a problem or flag the data and provide a corrective action report.
- 8.1.5. If the sample extract(s) require any cleanup procedure, the associated batch QC (i.e. MB, MS/MSD and LCS) must also undergo the same cleanup procedure with the sample extract(s).

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8.2. Calculations

8.2.1. Final Results

$$C_f = C_i \times PF \times DF$$

$C_f$  = Final concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$

$C_i$  = Concentration in  $\text{ng}/\mu\text{L}$  from instrument

PF = Preparation Factor

DF = Any additional Dilution Factor

8.2.2. Determine the % Difference of the CCV using the following equation:

$$\% \text{ Difference} = \frac{[\text{Apparent conc. } (\mu\text{g/l}) - \text{True conc. } (\mu\text{g/L})] \times 100}{\text{True conc. } (\mu\text{g/l})}$$

8.2.3. Determine the % recovery for the LCS and MS/MSD as follows:

$$\% \text{ Recovery} = \frac{(S_p - S)}{S_a} \times 100 \quad \text{RPD} = \frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100$$

Where:  $S_p$  = Spike result

$S$  = Sample result

$S_a$  = Spike amount

$R_1$  = Conc. of MS

$R_2$  = Conc. of MSD

8.2.4. Determine response factor (RF)

$$RF = \frac{A_s \times C_{IS}}{A_{IS} \times C_s}$$

Where:

$A_s$  = Peak area (or height) of the analyte or surrogate

$A_{IS}$  = peak area (or height) of the internal standard

$C_s$  = concentration of the analyte or surrogate, in  $\mu\text{g/L}$

$C_{IS}$  = concentration of the internal standard, in  $\mu\text{g/L}$

8.2.5. Degradation checks

$$\% \text{ breakdown for 4,4'-DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)} \times 100}{\text{Total DDT peak area (DDT + DDE + DDD)}}$$

$$\% \text{ breakdown for Endrin} = \frac{\text{Total endrin degradation peak area (endrin aldehyde + endrin ketone)} \times 100}{\text{Total endrin peak area (endrin + endrin aldehyde + endrin ketone)}}$$

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**9. PAPERWORK FLOW**

9.1. Daily

- 9.1.1. Query ELMNT for the desired analysis codes.
- 9.1.2. Compare the worklist with the previous day's Daily Log Summary sheet to determine which sample numbers to cross off. Cross off the sample numbers of those analyses that have been completed and update the status in ELMNT.
- 9.1.3. Complete the Daily Log Summary sheet and instrument logbook as each sample is loaded onto the autosampler. Use the Comments section to note whether or not a sample is a confirmation, a re-analysis, or a rush. Also include a dilution amount or any other important information in the Comments section.
- 9.1.4. Reports and their corresponding chromatograms automatically print out as each sample is analyzed. Perform a manual integration when a chromatogram has a co-elution or interferences. All manual integrations must undergo second level review.
- 9.1.5. Print out the mass spectra that corresponds to positive results on the quantitation report. Compare these spectra with the calibration spectra. If there is no spectral match (NSM), draw a line through the result on the report and write NSM beside it.
- 9.1.6. Staple behind each chromatogram, its corresponding quantitation report and mass spectra. Match the chromatograms, quantitation reports, and spectra to their corresponding Daily Log Summary sheet and file them in the cabinet by ascending date.
- 9.1.7. Fill out a Continuing Calibration Check Criteria form daily. Indicate whether or not the CCV criteria was met.
- 9.1.8. Complete the Standard Log in ELMNT when new standards are prepared and have it peer-reviewed prior to use.
- 9.1.9. Check the study name and analyst on each sequence file to ensure the correct method name and analyst are entered.
- 9.1.10. All raw data must be submitted for second level review and must be initialed and dated.

9.2. As Needed

- 9.2.1. Document on a Corrective Action Report, any non-matrix related analytical problems or variances.
- 9.2.2. Complete an instrument maintenance log for each instrument and include the date that the instrument became inoperable, the steps taken to fix the instrument, replacement parts, the initials of the analyst the name of the service company that performed the maintenance, and the date that the instrument became operable again.

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9.2.3. Fill out a GCMS Calibration Criteria form whenever any compound exceeds the ICAL or CCV criteria. Include a copy of the form with each data set affected daily.

9.2.4. Complete a Preparation Bench Sheet whenever samples are analyzed. Be sure "spike witnessed by" and "extracts received by" are appropriately signed or marked as "not applicable."

**10. METHOD PERFORMANCE**

10.1. See attached analysis codes for information regarding control limits, reporting limits, and method detection limits.

10.2. Perform a method detection limit study at least once a year, and after extensive instrument maintenance. A method detection limit study is accomplished by analyzing seven replicates over a minimum of two days.

10.3. Every analyst must perform an Initial Demonstration of Capability (IDOC) before performing analyses on any client samples. An IDOC can be 1) 4 consecutive LCS samples with an average recovery and RSD within the in-house statistical limits, or 2) passing results on a blind or PT study.

**11. POLLUTION PREVENTION**

11.1. Acid waste should be disposed of in the acid waste drum.

11.2. Basic waste should be disposed of in the basic waste drum.

11.3. Samples that are hazardous should be segregated and disposed of as hazardous waste.

**12. WASTE MANAGEMENT**

12.1. Methylene chloride waste/rinsate is poured into a hazardous waste container under the hood.

12.2. The waste container must be labeled as "Hazardous Waste, Contents: Methylene Chloride -Liq., Toxic" and marked with an accumulation start date or frequency emptied.

12.3. Satellite waste containers may only be opened while adding or removing waste. When filling a container with liquid waste, always leave a 5% headspace to allow for expansion of the contents.

12.4. Safety glasses, gloves and lab coat must be worn while handling waste.

12.5. When the waste container is full, it is removed from the hood and placed in the sample archive storage area where a trained waste management technician will dispose the waste into the appropriate waste drum.

12.6. Target vials and unused standards are disposed of into a step-on can in extractions which then is drummed and shipped out for incineration by a trained waste management technician.

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- 12.7. Samples that are known to be hazardous prior to analysis are placed in a pink bag by sample control technicians and stored in the hazardous sample refrigerator. After analysis, the samples must be returned to the hazardous sample refrigerator in the pink bag. A trained sample disposal technician removes samples on a scheduled basis and disposes the samples according to the Hazardous Waste Disposal SOP.
- 12.8. For more detail, see the Waste Management Section of the Safety Manual & Chemical Hygiene Plan (SMCHP).

**13. METHOD REFERENCES**

- 13.1. EPA Method 8270C, EPA SW-846 Update III, December 1996
- 13.2. EPA 625, 40 CFR Part 136, Appendix B

**14. APPENDICES**

- 14.1. Table of DFTPP Tuning Criteria
- 14.2. Table of SPCC and CCC acceptance criteria
- 14.3. Table of Instrument Conditions
- 14.4. GCMS Initial Calibration Criteria Form
- 14.5. GCMS calibration Check Criteria Form
- 14.6. ICAL Checklist
- 14.7. Daily Run Sequence Checklist
- 14.8. Table of Internal Standard-Analyte Associations
- 14.9. Table of Characteristic Ions

**15. REFERENCE VARIANCES**

- 15.1. EPA 625
  - 15.1.1. NOTE: The laboratory has merged the method 8270C and 625. If 8270C criteria are stricter than those listed in 625, 8270C criteria will generally be adhered to and are not addressed below as variances.
  - 15.1.2. ICAL acceptance criteria are based on those for SW8270C.
  - 15.1.3. Control limits for ICV, CCV and LCS are based upon statistically-derived in-house limits, provided they are tighter than those specified in Table 6 of the source method.
  - 15.1.4. Standards are diluted to volume with MeCl<sub>2</sub>, not acetone. Samples and standards are prepared with MeCl<sub>2</sub>. MeCl<sub>2</sub> is used for dilutions to maintain consistency with the solvents.

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- 15.1.5. Only 1  $\mu\text{l}$  of each standard and sample is analyzed, not 2 to 5  $\mu\text{l}$ . Acceptable response and repeatability is achieved with a 1  $\mu\text{l}$  injection performed with the autosampler. The concentration of the DFTPP tuning standard is prepared at a higher concentration so that the required 50 ng of DFTPP is analyzed. All sample analyses, calibrations and method detection limit studies are also performed with 1  $\mu\text{l}$  injections
- 15.2. In order to add confidence to the non-CCC analytes' quantitation, though not specified in the method, the average % D for all non-CCC (including the SPCCs) analytes in the daily CCV must be within historically generated control limits based on  $\pm 3$  times the standard deviation from the mean of 20 CCV standards.

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**Table 14-1: DFTPP KEY IONS AND ION ABUNDANCE CRITERIA**

M/z	Required Intensity (relative abundance)
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

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**Table 14-2: SPCC & CCC Criteria**

SPCCs		CCCs	
Analyte	Avg RF Acceptance Criteria	Analyte	%RSD Acceptance Criteria
nitrosodipropylamine	>0.050	acenaphthene	<30
hexachlorocyclopentadiene		1,4-dichlorobenzene	
2,4-dinitrophenol		hexachlorobutadiene	
4-nitrophenol		diphenylamine	
		di-n-octyl phthalate	
		fluoranthene	
		benzo(a)pyrene	
		4-chloro-3-methylphenol	
		2,4-dichlorophenol	
		2-nitrophenol	
		phenol	
		pentachlorophenol	
		2,4,6-trichlorophenol	

**Table 14-3: Instrument Conditions**

Parameter	Setup-1 (#4, #8)	Setup-2 (#50)	Setup-3 (#49, low-level)
Column	HP5MS 30m x 0.25µm x 0.25mm	HP5MS 20m x 0.18µm x 0.18mm	HP5MS 20m x 0.18µm x 0.18mm
Carrier gas/flow rate	He, 1 mL/min	He, 0.7 mL/min	He, 0.7 mL/min
Initial temp	50 °C, hold for 2.9 minutes	50 °C, hold for 2.5 minutes	40 °C, hold for 1.5 minutes
Temp program	Rate A: 10 °C/min to 200 °C, hold for 0 minute Rate B: 20 °C/min to 275 °C, hold for 4 minute Rate C: 25°C/min to 300°C, hold until benzo (g,h,I) perylene is eluted	Rate A: 18 °C/min to 190 °C, hold for 0 minute Rate B: 25 °C/min to 275 °C, hold for 3 minute Rate C: 35°C/min to 310°C, hold until benzo (g,h,I) perylene is eluted	Rate A: 18 °C/min to 190 °C, hold for 0 minute Rate B: 25 °C/min to 275 °C, hold for 1 minute Rate C: 35°C/min to 300°C, hold until benzo (g,h,I) perylene is eluted
Injector Temp	250 °C	250 °C	210 °C







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**FIGURE 14-6: Initial Calibration Checklist**

**GCMS INITIAL CALIBRATION CHECK LIST**  
EPA 8270C/625 – Semivolatile Organic Analysis

2 <sup>nd</sup> Level Review: _____ Date: _____ _____ _____	Analyst: _____ Analysis Date: _____ GCMS #: _____
--	---

<u>2<sup>nd</sup> Level Rev</u>	<u>Analyst Rev</u>	
_____	_____	<b>DFTPF Tuning :</b> Benzidine tailing <=3; Pentachlorophenol tailing <=5; DDT degradation <=20%
_____	_____	<b>Calibration :</b>
_____	_____	• Minimum 5-point calibration – lowest standard at RL (= 6-point for quadratic regression)
_____	_____	• Minimum Response Factors (RF) for SPCCs: >=0.050
_____	_____	• RSD of RF: <= ±30 % for CCCs; <= ±15 % for non-CCCs.
_____	_____	• If RSD >± 15 % and r <sup>2</sup> >= 0.99: use linear or quadratic regression
_____	_____	<b>LCS check :</b>
_____	_____	• After initial calibration
_____	_____	• SPCC: Minimum RF and % recovery met (refer to in-house limits)
_____	_____	• CCC: % difference from initial calibration <= 20%
_____	_____	• Other compounds: % recovery met (refer to in-house limits)
_____	_____	<b>Datafile:</b>
_____	_____	Print out calibration curve (include software or calculator-generated r <sup>2</sup> coefficient)
_____	_____	<b>Generate:</b>
_____	_____	• Response Factor summary sheet
_____	_____	• List compound summary sheet
_____	_____	• List compound history summary sheet
_____	_____	Print all levels with area count for all compounds
_____	_____	Check all levels for:
_____	_____	• Manual integration
_____	_____	• Print data before and after integration
_____	_____	• Transcription errors
_____	_____	Calibration performed within 12-hour tuning period
_____	_____	<b>Mini Miner Check</b>
_____	_____	Corrective Action Report attached (if applicable)
Comments: _____ _____ _____		

QualityData\_rev741614\GCMS8270\_CAL.doc  
 Rev 01/06/05

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**FIGURE 14-7: Daily Run Sequence Checklist**

**GCMS DATA CHECK LIST**  
EPA 8270C/625 – Semivolatile Organic Analysis

2 <sup>nd</sup> Level Review: _____	Analyst: _____
Date: _____	Analysis Date: _____
QC Batches: _____	GCMS #: _____

<u>2<sup>nd</sup> Level Rev</u>	<u>Analyst Rev</u>	
_____	_____	<b>DFTPP Tuning :</b>
_____	_____	Benzidine tailing <=3; Pentachlorophenol tailing <=5; DDT degradation <=20%
_____	_____	<b>Calibration :</b>
_____	_____	• Minimum 3-point calibration – lowest standard at RL
_____	_____	(>= 6-point for quadratic regression)
_____	_____	• Minimum Response Factors (RF) for SPCCs: >=0.050
_____	_____	• RSD of RF: <= +30 % for CCCs; <= +15 % for non-CCCs.
_____	_____	• If RSD >= 15 % and r <sup>2</sup> >= 0.99: use linear or quadratic regression.
_____	_____	For negative or "below-cal" results by regression: reprocess using RFs.
_____	_____	<b>Mid-point check (ICV/CCV) :</b>
_____	_____	• After initial calibration and every 12-hour shift
_____	_____	• SPCC: Minimum RF and % recovery met (refer to in-house limits)
_____	_____	• CCC: % difference from initial calibration <= 20%
_____	_____	• Other compounds: % recovery met (refer to in-house limits)
_____	_____	<b>Method blank :</b> every extraction batch of 20 samples (< RL or flag accordingly)
_____	_____	<b>LCS :</b> every extraction batch of 20 samples or less (checked against in-house limits)
_____	_____	<b>MS/MSD :</b> every extraction batch of 20 samples or less (checked against in-house limits)
_____	_____	<b>All samples check for :</b>
_____	_____	• Unit, Dilution Factor,
_____	_____	• Manual Integration, Transcription Errors,
_____	_____	• Spectra Match
_____	_____	• IS areas (-50% to +100 % first four IS; -20% to +100% last two IS)
_____	_____	• Surrogates within limits (refer to in-house limits)
_____	_____	• All samples analyzed within tuning period (EPA 8270C: 12hr, EPA 625: 24hr)
_____	_____	GCMS Initial Calibration Criteria Form attached (if averaged calibration RFs used)
_____	_____	GCMS Calibration Check Criteria Form attached (if averaged %REC of ICV/CCV used)
_____	_____	Mint Miner Check
_____	_____	Corrective Action Report attached (if applicable)
<b>Comments:</b>		_____
		_____
		_____

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**Table 14-8: Internal Standard-Analyte Association, 1 of 2**

TABLE 5 SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION		
1,4-Dichlorobenzene-d <sub>2</sub>	Naphthalene-d <sub>8</sub>	Acenaphthene-d <sub>10</sub>
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethylphenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d <sub>8</sub> (surr)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d <sub>6</sub> (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

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**Table 14-8: Internal Standard-Analyte Association, 2 of 2**

TABLE 5 (continued)		
Phenanthrene-d <sub>10</sub>	Chrysene-d <sub>12</sub>	Perylene-d <sub>12</sub>
4-Aminobiphenyl Anthracene 4-Bromophenyl phenyl ether Di-n-butyl phthalate 4,6-Dinitro-2-methyl- phenol Diphenylamine Fluoranthene Hexachlorobenzene N-Nitrosodiphenylamine Pentachlorophenol Pentachloronitrobenzene Phenacetin Phenanthrene Pronamide	Benzidine Benzo(a)anthracene Bis(2-ethylhexyl) phthalate Butyl benzyl phthalate Chrysene 3,3'-Dichlorobenzidine p-Dimethylaminoazobenzene Pyrene Terphenyl-d <sub>14</sub> (surr) 7,12-Dimethylbenz- (a)anthracene Di-n-octyl phthalate Indeno(1,2,3-cd) pyrene 3-Methylchol- anthrene	Benzo(b)fluor- anthrene Benzo(k)fluor- anthrene Benzo(g,h,i)- perylene Benzo(a)pyrene Dibenz(a,i)acridine Dibenz(a,h)- anthracene
(surr) = surrogate		

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**Table 14-9: Characteristic Ions, 2 of 6**

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosopyrrolidine	12.65	100	41,42,68,69
Acetophenone	12.67	105	71,51,120
4-Methylphenol	12.82	107	108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	107,77,51,79
3-Methylphenol	12.93	107	108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,55,56,41
1,4-Phenylenediamine	13.62	108	80,53,54,52
1-Chloronaphthalene	13.65 <sup>a</sup>	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	76,50,148
o-Anisidine	15.00	108	80,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d <sub>10</sub> (IS)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	164,126,98,63
4-Chloroaniline	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00 <sup>a</sup>	143	115,116
1,4-Naphthoquinone	16.23	158	104,102,76,50,130
p-Cresidine	16.45	122	94,137,77,93
Dichlorovos	16.48	109	185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	135,134,91,77
N-Nitrosodi-n-butylamine	16.73	84	57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

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**Table 14-9: Characteristic Ions, 3 of 6**

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethyl phosphoramide	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54 <sup>a</sup>	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro-o-toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d <sub>10</sub> (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosaffrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

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**Table 14-9: Characteristic Ions, 4 of 6**

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramidate	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
α,α-Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d <sub>12</sub> (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyriene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147

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**Table 14-9: Characteristic Ions, 5 of 6**

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate <sup>b</sup>	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d <sub>12</sub> (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,i)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

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**Table 14-9: Characteristic Ions, 6 of 6**

TABLE 1  
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
α-BHC	--	183	181,109
β-BHC	--	181	183,109
δ-BHC	--	183	181,109
γ-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d <sub>5</sub> (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d <sub>5</sub> (surr)	--	99	42,71
Terphenyl-d <sub>14</sub> (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard

surr = surrogate

\*Estimated retention times

<sup>b</sup>Substitute for the non-specific mixture, tricresyl phosphate

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## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
<b>8270C-Default in Water (EPA 8270C)</b>								
Preservation:4 C, Cool								
Container:1 L Amber								
Amount Required:2000 ml								
Hold Time:7 days								
Acenaphthene	2.0	10 ug/l			55 - 120	25	55 - 120	20
Acenaphthylene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Aniline	2.5	10 ug/l			40 - 120	25	40 - 120	30
Anthracene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Benzidine	8.5	20 ug/l			25 - 160	35	25 - 160	35
Benzoic acid	8.5	20 ug/l			25 - 125	30	25 - 120	30
Benzo(a)anthracene	2.0	10 ug/l			60 - 120	20	60 - 120	20
Benzo(b)fluoranthene	2.0	10 ug/l			55 - 125	25	55 - 125	25
Benzo(k)fluoranthene	2.0	10 ug/l			55 - 125	30	50 - 125	20
Benzo(g,h,i)perylene	3.0	10 ug/l			40 - 130	30	45 - 130	25
Benzo(a)pyrene	2.0	10 ug/l			55 - 125	25	55 - 125	25
Benzyl alcohol	2.5	20 ug/l			40 - 120	30	50 - 120	20
Bis(2-chloroethoxy)methane	2.0	10 ug/l			50 - 120	25	55 - 120	20
Bis(2-chloroethyl)ether	2.5	10 ug/l			50 - 120	25	50 - 120	20
Bis(2-chloroisopropyl)ether	2.5	10 ug/l			45 - 120	25	45 - 120	20
Bis(2-ethylhexyl)phthalate	4.0	50 ug/l			60 - 125	25	60 - 125	20
4-Bromophenyl phenyl ether	2.5	10 ug/l			50 - 120	25	55 - 120	25
Butyl benzyl phthalate	4.0	20 ug/l			50 - 125	25	50 - 125	20
4-Chloroaniline	2.0	10 ug/l			50 - 120	25	50 - 120	25
2-Chloronaphthalene	2.0	10 ug/l			55 - 120	20	55 - 120	20
4-Chloro-3-methylphenol	2.0	20 ug/l			55 - 120	25	55 - 120	25
2-Chlorophenol	2.0	10 ug/l			45 - 120	25	45 - 120	25
4-Chlorophenyl phenyl ether	2.0	10 ug/l			55 - 120	25	60 - 120	20
Chrysene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Dibenz(a,h)anthracene	3.0	20 ug/l			45 - 135	30	50 - 135	25
Dibenzofuran	2.0	10 ug/l			60 - 120	25	60 - 120	20
Di-n-butyl phthalate	2.0	20 ug/l			55 - 120	25	55 - 125	20
1,3-Dichlorobenzene	3.0	10 ug/l			35 - 120	25	35 - 120	25
1,4-Dichlorobenzene	2.5	10 ug/l			35 - 120	25	35 - 120	25
1,2-Dichlorobenzene	3.0	10 ug/l			40 - 120	25	40 - 120	25
3,3-Dichlorobenzidine	3.0	20 ug/l			45 - 130	25	50 - 135	25
2,4-Dichlorophenol	2.0	10 ug/l			50 - 120	25	50 - 120	20
Diethyl phthalate	2.0	10 ug/l			50 - 120	30	50 - 120	30
2,4-Dimethylphenol	3.5	20 ug/l			35 - 120	25	35 - 120	25
Dimethyl phthalate	2.0	10 ug/l			25 - 120	30	25 - 120	30
4,6-Dinitro-2-methylphenol	4.0	20 ug/l			40 - 120	25	40 - 120	25
2,4-Dinitrophenol	4.5	20 ug/l			35 - 120	25	35 - 120	25
2,4-Dinitrotoluene	2.0	10 ug/l			60 - 120	25	60 - 120	20
2,6-Dinitrotoluene	2.0	10 ug/l			55 - 120	20	60 - 120	20
Di-n-octyl phthalate	2.0	20 ug/l			60 - 130	20	60 - 130	20
Fluoranthene	2.0	10 ug/l			55 - 120	25	55 - 120	20
Fluorene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Hexachlorobenzene	2.5	10 ug/l			50 - 120	25	55 - 120	20
Hexachlorobutadiene	3.5	10 ug/l			40 - 120	25	40 - 120	25
Hexachlorocyclopentadiene	5.0	20 ug/l			20 - 120	30	20 - 120	30
Hexachloroethane	3.0	10 ug/l			35 - 120	25	35 - 120	25

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
Indeno(1,2,3-cd)pyrene	3.0	20 ug/l			40 - 135	30	45 - 135	25
Isophorone	2.0	10 ug/l			50 - 120	25	50 - 120	20
2-Methylnaphthalene	2.0	10 ug/l			50 - 120	20	50 - 120	20
2-Methylphenol	2.0	10 ug/l			50 - 120	25	50 - 120	20
4-Methylphenol	2.0	10 ug/l			50 - 120	25	45 - 120	20
Naphthalene	2.5	10 ug/l			50 - 120	25	50 - 120	20
2-Nitroaniline	2.0	20 ug/l			60 - 120	25	60 - 120	20
3-Nitroaniline	2.0	20 ug/l			55 - 120	25	55 - 120	25
4-Nitroaniline	2.5	20 ug/l			50 - 125	25	50 - 125	20
Nitrobenzene	2.5	20 ug/l			50 - 120	25	50 - 120	25
2-Nitrophenol	3.5	10 ug/l			45 - 120	25	45 - 120	25
4-Nitrophenol	5.5	20 ug/l			40 - 120	25	40 - 120	30
N-Nitrosodiphenylamine	2.0	10 ug/l			55 - 120	25	55 - 120	20
N-Nitroso-di-n-propylamine	2.5	10 ug/l			45 - 120	25	45 - 120	20
Pentachlorophenol	3.5	20 ug/l			45 - 125	25	45 - 125	25
Phenanthrene	2.0	10 ug/l			55 - 120	25	60 - 120	20
Phenol	2.0	10 ug/l			45 - 120	25	45 - 120	25
Pyrene	2.0	10 ug/l			50 - 120	25	50 - 125	25
1,2,4-Trichlorobenzene	2.5	10 ug/l			45 - 120	20	45 - 120	20
2,4,5-Trichlorophenol	3.0	20 ug/l			50 - 120	30	50 - 120	30
2,4,6-Trichlorophenol	3.0	20 ug/l			50 - 120	30	50 - 120	30
1,2-Diphenylhydrazine/Azobenzene	2.0	20 ug/l			55 - 120	25	55 - 120	25
surr: 2-Fluorophenol			30 - 120					
surr: Phenol-d6			35 - 120					
surr: 2,4,6-Tribromophenol			40 - 120					
surr: Nitrobenzene-d5			40 - 120					
surr: 2-Fluorobiphenyl			45 - 120					
surr: Terphenyl-d14			45 - 120					

## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
<b>8270C/3545 (Default) in Soil (EPA 8270C)</b>								
Preservation:4 C, Cool								
Container:4 oz Jar								
Amount Required:100 grams								
Hold Time:14 days								
Acenaphthene	60	330 ug/kg			45 - 120	25	55 - 120	20
Acenaphthylene	70	330 ug/kg			60 - 120	20	55 - 120	20
Aniline	70	420 ug/kg			40 - 110	30	25 - 120	20
Anthracene	50	330 ug/kg			60 - 120	25	60 - 120	20
Benzidine	660	660 ug/kg			25 - 120	30	10 - 160	30
Benzoic acid	70	830 ug/kg			25 - 120	30	25 - 120	30
Benzo(a)anthracene	50	330 ug/kg			50 - 120	25	60 - 120	20
Benzo(b)fluoranthene	50	330 ug/kg			55 - 125	30	55 - 125	25
Benzo(k)fluoranthene	50	330 ug/kg			55 - 120	30	50 - 125	25
Benzo(g,h,i)perylene	110	330 ug/kg			30 - 120	30	40 - 130	25
Benzo(a)pyrene	40	330 ug/kg			55 - 120	25	60 - 120	20
Benzyl alcohol	100	330 ug/kg			40 - 120	30	35 - 120	25
Bis(2-chloroethoxy)methane	70	330 ug/kg			50 - 120	25	50 - 120	20
Bis(2-chloroethyl)ether	60	170 ug/kg			35 - 110	25	40 - 120	25
Bis(2-chloroisopropyl)ether	60	330 ug/kg			40 - 120	25	40 - 120	20
Bis(2-ethylhexyl)phthalate	90	330 ug/kg			50 - 125	25	55 - 125	20
4-Bromophenyl phenyl ether	60	330 ug/kg			45 - 120	20	50 - 120	20
Butyl benzyl phthalate	80	330 ug/kg			50 - 120	25	50 - 120	20
4-Chloroaniline	80	330 ug/kg			10 - 120	30	20 - 120	30
2-Chloronaphthalene	50	330 ug/kg			45 - 120	20	50 - 120	20
4-Chloro-3-methylphenol	70	330 ug/kg			55 - 120	25	55 - 120	20
2-Chlorophenol	60	330 ug/kg			40 - 120	20	45 - 120	20
4-Chlorophenyl phenyl ether	70	330 ug/kg			50 - 120	25	60 - 120	20
Chrysene	40	330 ug/kg			55 - 120	25	55 - 120	20
Dibenz(a,h)anthracene	100	420 ug/kg			25 - 135	30	45 - 135	25
Dibenzofuran	60	330 ug/kg			55 - 120	25	60 - 120	20
Di-n-butyl phthalate	60	330 ug/kg			50 - 120	25	55 - 120	20
1,3-Dichlorobenzene	90	330 ug/kg			35 - 120	25	35 - 120	25
1,4-Dichlorobenzene	50	330 ug/kg			35 - 120	25	35 - 120	25
1,2-Dichlorobenzene	60	330 ug/kg			40 - 120	25	40 - 120	20
3,3-Dichlorobenzidine	150	830 ug/kg			15 - 120	25	20 - 135	25
2,4-Dichlorophenol	60	330 ug/kg			50 - 120	25	50 - 120	20
Diethyl phthalate	70	330 ug/kg			50 - 120	25	55 - 120	20
2,4-Dimethylphenol	100	330 ug/kg			35 - 120	25	40 - 120	20
Dimethyl phthalate	50	330 ug/kg			25 - 120	25	55 - 120	20
4,6-Dinitro-2-methylphenol	110	420 ug/kg			40 - 120	25	45 - 120	25
2,4-Dinitrophenol	80	660 ug/kg			35 - 120	25	25 - 120	25
2,4-Dinitrotoluene	80	330 ug/kg			50 - 120	25	60 - 120	20
2,6-Dinitrotoluene	60	330 ug/kg			55 - 120	20	60 - 120	20
Di-n-octyl phthalate	60	330 ug/kg			55 - 120	25	50 - 130	20
Fluoranthene	70	330 ug/kg			45 - 120	25	60 - 120	20
Fluorene	70	330 ug/kg			50 - 120	25	60 - 120	20
Hexachlorobenzene	70	330 ug/kg			50 - 120	25	55 - 120	20
Hexachlorobutadiene	60	330 ug/kg			40 - 110	25	40 - 120	20
Hexachlorocyclopentadiene	80	830 ug/kg			20 - 120	30	30 - 120	25
Hexachloroethane	40	330 ug/kg			35 - 120	30	40 - 120	20

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
Indeno(1,2,3-cd)pyrene	130	330 ug/kg			20 - 135	30	35 - 135	25
Isophorone	50	330 ug/kg			40 - 120	25	45 - 120	20
2-Methylnaphthalene	70	330 ug/kg			45 - 120	20	50 - 120	20
2-Methylphenol	80	330 ug/kg			50 - 120	25	45 - 120	20
4-Methylphenol	60	330 ug/kg			50 - 120	25	50 - 120	20
Naphthalene	60	330 ug/kg			40 - 120	25	50 - 120	20
2-Nitroaniline	60	330 ug/kg			50 - 120	25	55 - 120	20
3-Nitroaniline	70	330 ug/kg			30 - 120	25	35 - 120	25
4-Nitroaniline	90	830 ug/kg			40 - 120	30	50 - 120	20
Nitrobenzene	70	330 ug/kg			40 - 120	25	45 - 120	20
2-Nitrophenol	60	330 ug/kg			45 - 120	25	50 - 120	20
4-Nitrophenol	140	830 ug/kg			40 - 120	30	45 - 120	20
N-Nitrosodiphenylamine	80	330 ug/kg			50 - 120	25	50 - 120	20
N-Nitroso-di-n-propylamine	70	250 ug/kg			35 - 120	25	45 - 120	20
Pentachlorophenol	150	830 ug/kg			30 - 125	25	45 - 125	20
Phenanthrene	60	330 ug/kg			50 - 120	25	55 - 120	20
Phenol	40	330 ug/kg			45 - 120	25	40 - 120	20
Pyrene	80	330 ug/kg			45 - 120	30	45 - 125	25
1,2,4-Trichlorobenzene	50	330 ug/kg			40 - 120	25	40 - 120	20
2,4,5-Trichlorophenol	130	330 ug/kg			50 - 120	20	55 - 120	20
2,4,6-Trichlorophenol	70	330 ug/kg			50 - 120	25	50 - 120	20
1,2-Diphenylhydrazine/Azobenzene	60	330 ug/kg			55 - 125	25	60 - 120	20
surr: 2-Fluorophenol			25 - 120					
surr: Phenol-d6			35 - 120					
surr: 2,4,6-Tribromophenol			35 - 125					
surr: Nitrobenzene-d5			30 - 120					
surr: 2-Fluorobiphenyl			35 - 120					
surr: Terphenyl-d14			40 - 135					



## Analytical Method Information

Analyte	MDL	Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
		Limit	%R	RPD	%R	RPD	%R	RPD
<b>625-Default in Water (EPA 625)</b>								
Preservation:4 C, Cool								
Container:1 L Amber								
Amount Required:2000 ml								
Hold Time:7 days								
Acenaphthene	2.0	10 ug/l			55 - 120	25	55 - 120	20
Acenaphthylene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Aniline	2.5	10 ug/l			40 - 120	25	40 - 120	30
Anthracene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Benzidine	8.5	20 ug/l			25 - 160	35	25 - 160	35
Benzoic acid	8.5	20 ug/l			25 - 125	30	25 - 120	30
Benzo(a)anthracene	2.0	10 ug/l			60 - 120	20	60 - 120	20
Benzo(b)fluoranthene	2.0	10 ug/l			55 - 125	25	55 - 125	25
Benzo(k)fluoranthene	2.0	10 ug/l			55 - 125	30	50 - 125	20
Benzo(g,h,i)perylene	3.0	10 ug/l			40 - 130	30	45 - 130	25
Benzo(a)pyrene	2.0	10 ug/l			55 - 125	25	55 - 125	25
Benzyl alcohol	2.5	20 ug/l			40 - 120	30	50 - 120	20
Bis(2-chloroethoxy)methane	2.0	10 ug/l			50 - 120	25	55 - 120	20
Bis(2-chloroethyl)ether	2.5	10 ug/l			50 - 120	25	50 - 120	20
Bis(2-chloroisopropyl)ether	2.5	10 ug/l			45 - 120	25	45 - 120	20
Bis(2-ethylhexyl)phthalate	4.0	50 ug/l			60 - 125	25	60 - 125	20
4-Bromophenyl phenyl ether	2.5	10 ug/l			50 - 120	25	55 - 120	25
Butyl benzyl phthalate	4.0	20 ug/l			50 - 125	25	50 - 125	20
4-Chloroaniline	2.0	10 ug/l			50 - 120	25	50 - 120	25
2-Chloronaphthalene	2.0	10 ug/l			55 - 120	20	55 - 120	20
4-Chloro-3-methylphenol	2.0	20 ug/l			55 - 120	25	55 - 120	25
2-Chlorophenol	2.0	10 ug/l			45 - 120	25	45 - 120	25
4-Chlorophenyl phenyl ether	2.0	10 ug/l			55 - 120	25	60 - 120	20
Chrysene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Dibenz(a,h)anthracene	3.0	20 ug/l			45 - 135	30	50 - 135	25
Dibenzofuran	2.0	10 ug/l			60 - 120	25	60 - 120	20
Di-n-butyl phthalate	2.0	20 ug/l			55 - 120	25	55 - 125	20
1,3-Dichlorobenzene	3.0	10 ug/l			35 - 120	25	35 - 120	25
1,4-Dichlorobenzene	2.5	10 ug/l			35 - 120	25	35 - 120	25
1,2-Dichlorobenzene	3.0	10 ug/l			40 - 120	25	40 - 120	25
3,3-Dichlorobenzidine	3.0	20 ug/l			45 - 130	25	50 - 135	25
2,4-Dichlorophenol	2.0	10 ug/l			50 - 120	25	50 - 120	20
Diethyl phthalate	2.0	10 ug/l			50 - 120	30	50 - 120	30
2,4-Dimethylphenol	3.5	20 ug/l			35 - 120	25	35 - 120	25
Dimethyl phthalate	2.0	10 ug/l			25 - 120	30	25 - 120	30
4,6-Dinitro-2-methylphenol	4.0	20 ug/l			40 - 120	25	40 - 120	25
2,4-Dinitrophenol	4.5	20 ug/l			35 - 120	25	35 - 120	25
2,4-Dinitrotoluene	2.0	10 ug/l			60 - 120	25	60 - 120	20
2,6-Dinitrotoluene	2.0	10 ug/l			55 - 120	20	60 - 120	20
Di-n-octyl phthalate	2.0	20 ug/l			60 - 130	20	60 - 130	20
Fluoranthene	2.0	10 ug/l			55 - 120	25	55 - 120	20
Fluorene	2.0	10 ug/l			60 - 120	25	60 - 120	20
Hexachlorobenzene	2.5	10 ug/l			50 - 120	25	55 - 120	20
Hexachlorobutadiene	3.5	10 ug/l			40 - 120	25	40 - 120	25
Hexachlorocyclopentadiene	5.0	20 ug/l			20 - 120	30	20 - 120	30
Hexachloroethane	3.0	10 ug/l			35 - 120	25	35 - 120	25

## Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
					%R	RPD	%R	RPD
Indeno(1,2,3-cd)pyrene	3.0	20 ug/l			40 - 135	30	45 - 135	25
Isophorone	2.0	10 ug/l			50 - 120	25	50 - 120	20
2-Methylnaphthalene	2.0	10 ug/l			50 - 120	20	50 - 120	20
2-Methylphenol	2.0	10 ug/l			50 - 120	25	50 - 120	20
4-Methylphenol	2.0	10 ug/l			50 - 120	25	45 - 120	20
Naphthalene	2.5	10 ug/l			50 - 120	25	50 - 120	20
2-Nitroaniline	2.0	20 ug/l			60 - 120	25	60 - 120	20
3-Nitroaniline	2.0	20 ug/l			55 - 120	25	55 - 120	25
4-Nitroaniline	2.5	20 ug/l			50 - 125	25	50 - 125	20
Nitrobenzene	2.5	20 ug/l			50 - 120	25	50 - 120	25
2-Nitrophenol	3.5	10 ug/l			45 - 120	25	45 - 120	25
4-Nitrophenol	5.5	20 ug/l			40 - 120	25	40 - 120	30
N-Nitrosodiphenylamine	2.0	10 ug/l			55 - 120	25	55 - 120	20
N-Nitroso-di-n-propylamine	2.5	10 ug/l			45 - 120	25	45 - 120	20
Pentachlorophenol	3.5	20 ug/l			45 - 125	25	45 - 125	25
Phenanthrene	2.0	10 ug/l			55 - 120	25	60 - 120	20
Phenol	2.0	10 ug/l			45 - 120	25	45 - 120	25
Pyrene	2.0	10 ug/l			50 - 120	25	50 - 125	25
1,2,4-Trichlorobenzene	2.5	10 ug/l			45 - 120	20	45 - 120	20
2,4,5-Trichlorophenol	3.0	20 ug/l			50 - 120	30	50 - 120	30
2,4,6-Trichlorophenol	3.0	20 ug/l			50 - 120	30	50 - 120	30
1,2-Diphenylhydrazine/Azobenzene	2.0	20 ug/l			55 - 120	25	55 - 120	25
surr: 2-Fluorophenol				30 - 120				
surr: Phenol-d6				35 - 120				
surr: 2,4,6-Tribromophenol				40 - 120				
surr: Nitrobenzene-d5				40 - 120				
surr: 2-Fluorobiphenyl				45 - 120				
surr: Terphenyl-d14				45 - 120				