

DRAFT
QUALITY ASSURANCE PROJECT PLAN
SOIL INVESTIGATION FOR
HISTORICAL STORMWATER PATHWAY - SOUTH
MONTROSE CHEMICAL SUPERFUND SITE

EPA CONTRACT NO. 68-W-98-225
EPA WORK ASSIGNMENT NO. 233-RICO-0926
CH2M HILL PROJECT NO. 336460.FI.01

Prepared for
U.S. Environmental Protection Agency
Region 9
75 Hawthorne Street
San Francisco, California 94105

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March 2006

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U.S. ENVIRONMENTAL PROTECTION AGENCY REGION 9

Plan Title: Draft Quality Assurance Project Plan, Soil Investigation for Historical Stormwater Pathway - South, Montrose Chemical Superfund Site, Los Angeles County, California
Site Name: Montrose Chemical Superfund Site
Site Location: 20201 Normandie Avenue
City/State/Zip: Torrance, California 90502
Site EPA ID#: CAD 008242711
Anticipated Sampling Dates: Starting in June 2006
Prepared By: Artemis Antipas March 2006
John Dolegowski Date

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Acronyms

ARAR	applicable or relevant and appropriate requirement
ASTM	American Society for Testing and Materials
bgs	below ground surface
BHC	benzene hexachloride
Cal-EPA	California Environmental Protection Agency
CCR	California Code of Regulations
CDWR	California Department of Water Resources
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	chain-of-custody
CRDL	contract-required detection level
DDD	4,4'-dichlorodiphenyldichloroethane
DDE	4,4'-dichlorodiphenyldichloroethene
DDT	4,4'-dichlorodiphenyltrichloroethane
DQO	data quality objective
DTSC	Department of Toxic Substances Control
ECI	Ecology Control Industries
EPA	U.S. Environmental Protection Agency
FAR	Federal Acquisition Regulations
FD	frequency of detection
FS	feasibility study
FSP	Field Sampling Plan
ft	foot/feet
GC	gas chromatography
GIS	geographic information system
HHRA	human health risk assessment

HSP	Health and Safety Plan
IDW	investigation-derived waste
LACFCD	Los Angeles County Flood Control District
LADWP	Los Angeles Department of Water and Power
LDR	land disposal restriction
m	meter(s)
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MS/MSD	matrix spike/matrix spike duplicate
msl	mean sea level
NFA	No Further Action
PCB	polychlorinated biphenyl
PD	Playa Deposits
PRG	Preliminary Remediation Goal
ppm	parts per million
PVS	Palos Verdes Sands
QA	quality assurance
QAO	Quality Assurance Officer
QAMS	Quality Assurance Management Section
QAPP	Quality Assurance Project Plan
QC	quality control
RAC	Response Action Contract
RCRA	Resource Conservation and Recovery Act
RI	remedial investigation
RPD	relative percent difference
RPM	Remedial Project Manager
RSCC	Regional Sample Control Center
RSD	relative standard deviation
RTL	Review Team Leader
SIM	selective ion monitoring

SM	Site Manager
SOW	statement of work
SRM	standard reference material
SSC	Site Safety Coordinator
STL	Sampling Team Leader
STLC	soluble threshold limit concentration
SVE	soil vapor extraction
TAL	target analyte list
TCL	target compound list
TTLC	total threshold limit concentration
TCLP	toxicity characteristic leaching procedure
TPH	total petroleum hydrocarbons
TPH-d	total petroleum hydrocarbons-diesel
TPH-g	total petroleum hydrocarbons-gasoline
TPH-o	total petroleum hydrocarbons-oil
TSDF	treatment, storage, or disposal facility
UBA	Upper Bellflower Aquitard
UCL	upper confidence limit
U.S.	United States
USC	United States Code
UST	underground storage tank
UTS	universal treatment standard
VOC	volatile organic compound
WAM	Work Assignment Manager

1.0 Introduction

This Quality Assurance Project Plan (QAPP) presents the policies, organizations, objectives, and functional activities/procedures associated with the remedial investigation (RI) soil sampling and analysis activities within a portion of the Historical Stormwater Pathway – South for the Montrose Chemical Superfund Site, Los Angeles County, California, by the United States (U.S.) Environmental Protection Agency (EPA). This QAPP includes the data quality objectives (DQOs), which are presented in Appendix A.

This QAPP follows EPA guidelines contained in *EPA Guidance for Quality Assurance Project Plans* (EPA, 2002a), and *EPA Requirements for Quality Assurance Project Plans* (EPA, 2001a). The development, review, approval, and implementation of the QAPP is part of EPA's mandatory Quality System, which requires all organizations to develop and operate management structures and processes to ensure that data used in agency decisions are of the type and quality needed for their intended use. The following sections of this document correlate with the subtitles found in the EPA guidelines (EPA, 2001a).

The document is organized into the following sections and appendixes:

- Section 1.0 Introduction.** Provides an introduction and describes the organization of the QAPP.
- Section 2.0 Project Management/Data Quality Objectives.** Describes project organization, background, goals, and DQOs (through reference to Appendix A); summarizes data needs, uses, performance criteria, and task descriptions.
- Section 3.0 Measurement Data Acquisition.** Defines the sampling methods, sample handling, chain-of-custody (COC), analytical methods, and quality control (QC) data to be acquired.
- Section 4.0 Assessment/Oversight.** Describes procedures to assess and oversee quality of data collection procedures.
- Section 5.0 Data Validation and Usability.** Describes the data quality assurance/quality control (QA/QC) procedures.
- Section 6.0 References.** Provides a list of references used in preparing this document.
- Appendix A Data Quality Objectives.** Presents the DQO process that identifies the specific objectives, the associated data needs, decisions, and subsequently the sampling design. The last two steps of the DQO process (Steps 6 and 7) provide details of the statistical analysis of historical data and statistical performance specifications that have been used in the formulation of the proposed sampling, particularly with respect to how the proposed sampling achieves the broadly defined DQOs identified in Section 2.0.

Appendix B Analytical Technical Specifications. Provides specification requirements for analytical techniques to be used in quantitation of samples collected.

This QAPP is accompanied by the Field Sampling Plan (FSP), for soil investigation of a portion of the Historical Stormwater Pathway - South, Montrose Chemical Superfund Site, Los Angeles County, California (EPA, 2006).

2.0 Project Management/Data Quality Objectives

2.1 Project Organization

This project is being conducted as Work Assignment No. 233-RICO-0926 under EPA Response Action Contract (RAC) No. 68-W-98-225. CH2M HILL has designated a Site Manager (SM) who works directly with the EPA Work Assignment Manager (WAM) and Remedial Project Manager (RPM) to complete the work assignment. The SM will manage the financial, schedule, and technical status of the work assignment. The key people interfacing with the SM are the EPA WAM and RPM, and the CH2M HILL Quality Assurance Officer (QAO), Review Team Leader (RTL), individual task managers for field sampling, and the Sampling Team Leader (STL).

The primary responsibility for project quality rests with the SM, while independent QC is provided by the RTL and QAO. The RTL/review team and QAO will review project planning documents, data evaluations, and deliverables.

The sampling team will implement the project in accordance with the Sampling and Analysis Plan (consisting of the QAPP and FSP) and the companion Health and Safety Plan (HSP). The CH2M HILL Site Safety Coordinator (SSC) is responsible for adherence to the HSP and field decontamination procedures. The entire field effort is directed by the STL.

The subcontract administrator is responsible for procuring subcontracts for EPA's RAC projects under the Federal Acquisition Regulations (FAR), and provides the interface with project subcontractors. Subcontractors will be utilized on this work assignment for concrete coring and direct-push sampling, and may be utilized for laboratory analyses, depending on the availability of the EPA regional laboratory or another laboratory designated by EPA.

Where quality assurance (QA) problems or deficiencies requiring special action are uncovered, the SM, RTL, and QAO, in coordination with the EPA representatives, will identify the appropriate corrective action to be initiated by the STL or the laboratory.

Project organization and the line of authority for CH2M HILL efforts are illustrated in Figure 2-1. Data users and recipients are shown in Figure 2-2. Both EPA and CH2M HILL technical personnel and QA personnel are shown.

The organizational functions noted above are consistent with the overall RAC 9 Program Plan; these functions are further detailed in the program plan.

2.2 Problem Definition/Background

2.2.1 Purpose

This QAPP presents the policies, organizations, objectives, and functional activities/procedures associated with the soil sampling and analysis activities to be conducted by EPA in a segment of the historical stormwater pathway, south of Torrance Boulevard (Historical

Stormwater Pathway – South). This QAPP includes the DQOs, which can be found in Appendix A.

This QAPP follows EPA guidelines contained in *EPA Guidance for the Data Quality Objectives Process* (EPA, 2000) and *EPA Requirements for Quality Assurance Project Plans* (EPA, 2001a). Thus, the section headings contained herein correlate with the subtitles found in the EPA guidelines (EPA, 2002a).

2.2.2 Problem Statement

Specific problem statements are prepared in the DQO process in Appendix A; following is a general description of the primary problems. The areas of initial investigation (the Study Area) include the commercial property occupied at 20846 Normandie Avenue, southeast of the intersection of Normandie Avenue and Torrance Boulevard, in Los Angeles County, California, and seven residential properties located directly east of 20846 Normandie Avenue. The commercial property is occupied by Ecology Control Industries (ECI). A map of the Study Area is provided in Figure 2-3. The historical stormwater pathway passed through a portion of each of these properties.

EPA believes that total DDT and other hazardous substances detected in soil from within the Study Area are from the former Montrose Chemical Plant property, and that the Study Area is, therefore, part of the Montrose Chemical Superfund Site. Additional sampling is necessary to characterize the nature and extent of Montrose-related contamination, as required by the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA). EPA does not believe that the sampling already performed at the ECI Property on behalf of the property owner (described below) adequately meets this objective. Additional surface soil and subsurface soil samples from the Study Area portion of the historical stormwater pathway need to be collected and analyzed for Montrose-related contamination to provide a more detailed characterization of this portion of the Historical Stormwater Pathway – South.

EPA understanding of the Study Area and the need for additional sampling are summarized below, and described in further detail in subsequent sections of this QAPP.

ECI, a California-registered hazardous waste transporter, utilizes the 20846 Normandie Avenue property as a dispatch yard for its truck fleet and for maintaining roll-off bins, containers, and other environmental-use equipment and vehicles (EPA, 2005b). In the spring of 2005, sampling at the ECI Property was conducted by Haley & Aldridge (under contract to ECI) as part of due diligence activities to prepare for sale of the property. That sampling found several constituents present in soil at elevated concentrations, including petroleum hydrocarbons, polychlorinated biphenyls (PCBs), and several organochlorine pesticides including chlordane and 4,4'-dichlorodiphenyltrichloroethane (DDT), 4,4'-dichlorodiphenyldichloroethene (DDE), and 4,4'-dichlorodiphenyldichloroethane (DDD). The property owner began excavating to remove soils containing these compounds.

DDT, DDE, and DDD (referred to hereafter as *total DDT*) were detected in soil samples from the eastern and southeastern portions of the ECI Property at concentrations up to 325 parts per million (ppm). These soil sample locations were excavated by the Property owner and the soil was stored on-property in soil piles, managed to prevent potential releases (e.g., via

fugitive dust and surface water runoff), until it could be properly disposed. EPA determined that soil in the piles and the open excavations presented a potential threat to public health, and issued a Unilateral Administrative Order (UAO, Docket No. 09-2006-02a) to ECI, the property owner, and Montrose Chemical Corporation of California (EPA, 2005b) requiring the transport and disposal of the excavated soil at the ECI Property, and the backfilling and covering of the open excavations. The soil piles were transported to a permitted hazardous waste landfill in January 2006 by the respondents. Backfilling of the excavations remains to be completed under the UAO.

EPA believes that total DDT was transported to the Study Area by the historical stormwater drainage pathway, which conveyed contaminants from the Montrose Chemical Corporation of California (Montrose) DDT manufacturing plant formerly located at 20201 Normandie Avenue, in Los Angeles County, California. The historical stormwater drainage pathway passes through the eastern portion of the ECI Property, where elevated concentrations of total DDT were detected (EPA, 2005a).

The historical drainage pathway was continuous from the former Montrose Plant property through the Study Area, and beyond, as indicated by aerial photographs (1947 to 1982). Stormwater leaving the former Montrose Plant property would collect just beyond the southeastern boundary of the former Montrose Plant property, an area referred to as the Normandie Avenue Ditch Ponding Area. From there, stormwater was conveyed across Normandie Avenue via an 18-inch culvert, and entered an "unimproved channel" that passed by houses along 204th Street, and continued south via a ditch along the west side of Kenwood Avenue (a.k.a. the Kenwood Ditch) to Torrance Boulevard. Stormwater then crossed under Torrance Boulevard, where the historical stormwater drainage pathway broadened to form a slough or swale within and beyond the Study Area.

In the late 1960s and early 1970s, the Los Angeles County Flood Control District (LACFCD) installed an underground concrete stormwater drainage system referred to as Project 685, Kenwood Avenue-Supplemental. The new system, Project 685, replaced both the ditch along Kenwood Avenue and the slough that was present in the Study Area and beyond. The LACFCD continues to have an easement for Project 685, including through the Study Area (along the eastern side of the ECI Property) and through the adjacent Royal Boulevard Landfill (the next segment of the historical stormwater pathway downstream of the Study Area). Portions of the Project 685 concrete box culvert are visible in the open excavations on the ECI Property.

In 2001, EPA addressed the portion of the historical stormwater pathway north of Torrance Boulevard, along Kenwood Avenue. This effort, referred to as the Kenwood Removal Action, addressed the west side of Kenwood Avenue, from Del Amo Alley to Torrance Boulevard. Sampling and excavation along Kenwood Avenue revealed elevated soil concentrations of total DDT (above 10 ppm), and up to 6,700 ppm total DDT. Additionally, in three yards, samples from fragments of a depositional layer left over from the bottom of the ditch contained total DDT concentrations in excess of 100,000 ppm. Areas with elevated total DDT were identified, and that soil was removed and replaced with clean fill.

2.2.3 Background

2.2.3.1 Site Location and Description

The Study Area is located in Los Angeles County, California, and includes portions of eight properties located along or within the Historical Stormwater Pathway – South, southeast of the intersection of Normandie Avenue and Torrance Boulevard. The eight properties include a commercial property located at 20846 Normandie Avenue, occupied by ECI, and seven residential properties located directly east of the ECI Property along Raymond Avenue and 209th Street. This area may be extended depending on data obtained under this study.

2.2.3.2 Operational History

In 1992, the ECI Property owner, Mr. Ron Flury, purchased approximately 4.7 acres of Azko Coatings, Inc. (Azko) property. Several years later, Mr. Flury purchased an additional 2.7 acres of Azko's remaining property. The current size of the ECI Property is approximately 7.5 acres.

During its ownership, Azko maintained two underground storage tank (UST) farms that stored petroleum-based solvents, in what is now the southern boundary of the ECI Property. A release of toluene from one of the tanks required soil and groundwater investigations and the installation of a soil vapor extraction (SVE) system. As part of the installation of the SVE system, the southern area of the property was graded, and the western portion paved in concrete following installation of the SVE system. Azko operated the SVE system for several years after it sold the property to ECI. ECI occupied the property while the SVE system was in operation. On July 22, 1996, the California Regional Water Quality Control Board (RWQCB) issued a closure letter confirming the completion of the UST remedial action.

2.2.3.3 Physical Description

This section provides a brief description of the regional geology and hydrogeology, the historic stormwater pathway, and LACFCD drainage easement for the Project 685 buried stormwater drainage channel.

Geology and Hydrogeology

The Study Area is located within the West Coast Basin of the Torrance Plain. The Ballona Escarpment bounds the basin to the north, the Newport-Inglewood Uplift to the east, Palos Verdes Hills to the southwest, and the Pacific Ocean to the west. There are four major structural features within the Torrance Plain, in the vicinity of the Montrose Chemical Superfund Site and the Study Area: the Charnock Fault, the Palos Verdes Fault, the Torrance Anticline, and the Gardena Syncline (EPA, 1998; and California Department of Water Resources [CDWR], 1961).

The stratigraphy of the West Coast Basin includes Quaternary-age continental and marine deposits and Tertiary-age marine sediments overlying a basement complex of igneous and metamorphic rocks. The geologic units of hydrogeologic interest are (in order from oldest to youngest) the Pico Formation; the San Pedro Formation; the Lakewood Formation; and older dune sand, alluvium, and active dune sand (EPA, 1998; CDWR, 1961).

Hydrogeologic units in the West Coast Basin include aquitards and aquifers of varying compositions and water-yielding properties. These units, in order from first water encountered to deeper units, include the Bellflower Aquitard, the Gage Aquifer, an unnamed aquitard, the Lynwood Aquifer, another unnamed aquitard, and the Silverado Aquifer. A detailed discussion of the regional geologic, hydrogeologic, and physiographic setting is presented in the *Final Remedial Investigation Report for the Montrose Superfund Site* (EPA, 1998).

There are three generalized, unsaturated soil layers in the vicinity of the Montrose Plant property, described as follows:

- **Upper Layer – Playa Deposits:** This layer is found near the surface to depths of approximately 25 feet below ground surface (bgs). According to grain-size analysis of soil samples collected in this layer, silt and clay comprise more than 65 percent of these soils.
- **Middle Layer – Palos Verdes Sands:** This layer is found between approximately 25 and 45 feet bgs and consists primarily of fine-grained sands. According to grain-size analysis of soil samples collected in this layer, fine- and medium-grained sand comprises more than 70 percent of these soils.
- **Lower Layer – Upper Bellflower Aquitard:** This layer is found between approximately 45 feet bgs and groundwater (approximately 65 feet bgs) and consists of multiple thin sand layers interbedded with layers of silts and clays. Grain-size analysis of soil samples collected in this layer ranged from more than 70 percent fine-grained sand to more than 60 percent silt. This soil layer varied from fine-grained sands to clays and silts with increasing depth.

The specific occurrence, depth, and thickness of these units in the vicinity of the ECI Property have not been well defined. The first-encountered groundwater beneath the area is at approximately 65 to 70 feet bgs, in the Upper Bellflower Aquitard (EPA, 1998).

Historical Stormwater Pathway

The historical stormwater pathway was a natural surface water drainage channel that originated as a drainage ditch from the former Montrose Plant property. It initially entered a drainage ditch on the west side of Normandie Avenue, crossed Normandie Avenue via an 18-inch corrugated iron culvert, entered an “unimproved channel” that passed by houses along 204th Street, and continued south via a ditch along the west side of Kenwood Avenue (a.k.a. the Kenwood Ditch) to Torrance Boulevard. Stormwater crossed under Torrance Boulevard and broadened into a slough, or swale. This includes the area now occupied by the ECI Property. Figure 2-3 shows the stormwater pathway from the Montrose property including the Kenwood Drain and Torrance Lateral.

To help determine the extent of the historical stormwater pathway within the Study Area, an analysis of historical aerial photographs taken of the historical stormwater pathway and farther downstream into the adjacent Royal Boulevard Landfill was conducted (EPA, 2005a). The interpreted extent of the historical stormwater pathway for this area is shown in Figure 2-4, and further described in Section 2.2.3.4.

During the late 1960s and early 1970s, much of the historical stormwater pathway was replaced by a new system of stormwater conveyances constructed by the LACFCD. The segment along Kenwood Avenue, through the Study Area and beyond is an underground concrete box culvert referred to as Project 685, or the Kenwood Avenue-Supplemental. .

Construction drawings for the segment of Project 685 in the Study Area show a ground elevation of approximately 16 feet above mean sea level (msl) prior to construction (Los Angeles County Department of Public Works As-Built Drawings). This is believed to be the lowest elevation of the historical stormwater pathway in the Study Area. Installation of the Project 685 storm drain required excavation within the existing historical stormwater drainage in order to place the large, concrete box drain. The Project 685 box drain (8 feet wide and 12.5 feet high) is shown on the as-built drawings as having an invert elevation (interior bottom of the drain) at approximately 11 feet msl. These as-built drawings also show 1 foot of fill above the box drain, for a finished surface elevation of approximately 24.5 feet msl within the ECI Property (EPA, 2005b). In the process of installing this box drain, the excavation and backfilling of soils would have significantly mixed the soil.

Study Area Topography

In 1998, the owner of the ECI Property had the northern portion of that property graded (ECI, 2005). The preconstruction drawings show a surface elevation exceeding 40 feet msl at the western edge of the northern parcel, a large mound of soil in the center of the northern portion (created from previous grading of the southern portion of the property) and an elevation of 35 to 36 feet msl along the eastern edge of the property, with a low of 31 feet msl in the northeastern corner along the LACFCD drainage easement. The regrading of the ECI Property pushed soil from west to east, to level the property. Soil from an earthen embankment along Torrance Boulevard and the soil mound were used for the grading (EPA, 1993). After grading, the surface of the ECI Property transitioned smoothly from approximately 40 feet msl at its western edge to approximately 36 feet msl at its eastern edge. Residential properties immediately east of the ECI Property are shown as having elevations between approximately 33 and 36 feet msl (EPA, 2005b).

Figure 2-5 shows a conceptual model of EPA's understanding of the history of the ditch elevation relative to the placement of the Project 685 box drain and the current ground elevation adjacent to and above the Los Angeles County drainage easement on the ECI Property.

2.2.3.4 Analysis of Extent of Historical Stormwater Pathway within Study Area

Historical aerial photographs, topographic maps, grading plans, and precipitation data were analyzed collectively to estimate the lateral extent of the historical stormwater pathway within the area analyzed (EPA, 2005a). These photographs, maps, and plans were scanned, rectified, and loaded into a geographic information system (GIS) database for this analysis.

Two main types of information were extracted from the historical aerial photographs: areas of historical inundation, and areas of potential wetland/riparian vegetation. Areas of inundation are directly representative of the extent of past stormwater flow events; however, the available aerial photographs do not necessarily capture the most significant or relevant events and only represent the flow conditions at the time of the photograph. Potential wetland/riparian vegetation, because of its dependence on wet environments, is

representative of areas subjected to frequent inundation and/or high groundwater levels; however, coverage may be limited by factors other than hydrology (e.g., soil type or land use). Nonetheless, delineation of historical inundation and potential wetland/riparian vegetation, when evaluated together, shows the approximate extent of historical stormwater flow. (Precipitation data from 1932 to 2005 for Torrance, California, were also evaluated to determine whether the flooding and apparent wet and dry conditions observed on the aerial photographs occurred during average, below average, or above average precipitation months or years.)

Figure 2-4 shows the cumulative (maximum) interpreted extent of potential wetlands/riparian areas (green areas) and ponded water (blue areas) delineated within the area analyzed, which included a portion of the adjacent Royal Boulevard Landfill for the years 1928 to 1965. These cumulative interpreted areas are used to define the extent of the historical stormwater pathway.

2.2.3.5 Previous Investigations and Regulatory Involvement

Previous Investigations

In the early 1980s, EPA and the State of California conducted investigations that documented the release of DDT from the Montrose Chemical Plant property, via several pathways, including storm water runoff.

Beginning in 1999 and continuing through 2002, as part of the ongoing investigations related to the Montrose Superfund Site, EPA conducted evaluations of total DDT in soils around the Montrose Chemical Superfund Site area, particularly in neighborhoods. An evaluation of background surface soil samples from areas in several directions from the former Montrose Plant property, including cross-wind and up-wind directions, found that regional background concentrations of total DDT in surface soil (up to 2 to 4 miles from the former Montrose Plant property) averaged between 1 and 3 ppm, and ranged up to 10 ppm.

Subsequent EPA investigations of soil in residential areas near the former Montrose Plant property found elevated levels of total DDT in the front yards of residential properties on the west side of Kenwood Avenue, along the pathway of the historical stormwater drainage pathway. The original stormwater ditch was in the low point of the front yards alongside Kenwood Avenue.

In 2001 and 2002, EPA conducted an investigation and removal action to remove DDT-contaminated soils associated with the portion of the historical ditch along Kenwood Avenue, from the Del Amo Alley to Torrance Boulevard. This effort is referred to as the Kenwood Storm Water Drainage Pathway Removal Action, or the Kenwood Removal Action (EPA, 2001d). In each yard, EPA collected and analyzed soil samples to define the extent of the old drainage ditch and areas of contamination affected by that former ditch. EPA took additional samples in several back yards, and found no elevated DDT concentrations in soil from the back yards of the properties. EPA determined the need for remediation at residences by using the concentration value corresponding to a one-in-one-hundred-thousand (1×10^{-5}) cancer risk for a residential exposure scenario (17 ppm). Remediation was then conducted to reach concentrations at or below 10 ppm (the upper end of the background range of total DDT in the South Los Angeles Area).

In the course of excavations, a layer (and layer fragments) of depositional material containing high levels of total DDT was clearly visible in subsurface soil at three properties. The southernmost finding of the depositional layer was at a residential property only three parcels north of Torrance Boulevard. This depositional layer is believed to have been the bottom of the former ditch along the historical stormwater pathway. Removal of the depositional layer resulted in deeper excavations at these properties.

Removal of soil was conducted at 22 properties and in 2 alleyways. With few exceptions, soils in this removal action were excavated to a maximum of 6 feet below the current ground surface elevation of Kenwood Avenue.

Regulatory Involvement

In the spring of 2005, sampling at the ECI Property was performed as part of due diligence activities to prepare the ECI Property for sale, as described in Section 2.2.3.6. That sampling indicated that several constituents, including petroleum hydrocarbons, PCBs, and DDT, were present in soil. These activities were performed without regulatory oversight (i.e., prior to EPA involvement).

EPA learned of the presence of DDT and the excavation activities at the ECI Property in early summer of 2005, and requested that the owner immediately stop excavation and implement protective measures to minimize water and wind erosion (e.g., fugitive dusts from excavated soil piles). EPA also requested ECI provide all information related to its soil sampling and excavation activities (i.e., locations, laboratory data sheets, etc.). A summary of data from this sampling effort is presented in Section 2.2.3.6.

Upon review of available information, EPA determined that soil in the piles and the open excavations presented a potential threat to public health, and a release or threat of release from the ECI Property. An EPA Removal Action Memorandum was prepared and approved November 2, 2005. To address the potential threat of release from these excavated soils, EPA issued a Unilateral Administrative Order (UAO, Docket No. 09-2006-02A) to ECI, the property owner, and Montrose Chemical Corporation of California (EPA, 2005c) requiring the transport and disposal of the excavated soil at the ECI Property to a permitted hazardous waste landfill, and the backfilling and covering of the open excavations. The piles of soil were transported in January 2006 by the respondents. Backfilling of the excavations remains to be completed under the UAO.

2.2.3.6 Summary of Existing Data

This section provides a summary of characterization data for soils at the ECI Property, collected in 2005. A more detailed evaluation of data from soil borings collected at the ECI Property is presented in Appendix A of this QAPP (DQOs, Step 6). Results and conclusions from those analyses served as the basis for the field investigation described below.

Available Soil Quality Data

In June 2005, EPA learned that an Environmental Site Assessment and sampling had been performed at the ECI Property in preparation for its sale for residential development (EPA, 2005b). Between February and June 2005, over 200 soil samples were collected at the ECI Property, with sampled depths ranging from just below the ground surface to approximately 15 feet bgs. Some or all samples were analyzed for pesticides, PCBs, total

petroleum hydrocarbons (TPH)-gasoline (TPH-g), TPH-diesel (TPH-d), TPH-oil (TPH-o), volatile organic compounds (VOCs), and metals. Elevated concentrations of several hazardous substances were detected during soil sampling activities including pesticides, PCBs, TPH-g, TPH-d, and TPH-o.

The locations of the soil borings are shown in Figure 2-6.

The soil sampling and analyses included the following:

- Collection of soil and soil gas samples from 15 locations across the property (February 7 and 8, 2005)
- Collection of soil samples using a 150-foot by 150-foot grid (March 23, 2005)
- Collection and analysis of an additional 24 soil borings along the eastern portion of the ECI Property where pesticides and PCBs had been detected. Many of these soil samples were grab samples taken from the walls of the open excavations. The excavation soil sample locations are shown in Figure 2-7. (April 12 and 13, 2005)

Excavation activities were conducted between March and June 2005 (March 17, 2005; May 17, 18, 26, and 27, 2005; and June 2, 3, 8, and 9, 2005) to remove soils with elevated chemical concentrations. *“ECI performed excavations and stockpile activities and Haley & Aldrich provided oversight of the excavation and conducted confirmation soil sampling activities”* (EPA, 2005b).

Table 2-1 presents a summary of the results of pesticide analyses for soil samples collected in 2005 from the ECI Property. Tables 2-2 and 2-3 provide soil sample results for metals and VOC analyses, respectively. Total DDT concentrations represent the sum of the concentrations of DDT, DDE, and DDD. Soil concentrations are in milligrams per kilogram (mg/kg), which are equivalent units to ppm.

Elevated concentrations of several chemicals were identified as a result of soil sampling and analysis conducted of the ECI Property in spring 2005. Soil contaminant concentrations exceeding federal and/or state regulatory limits or the regional background include:

- Total DDT – Detected at a maximum reported concentration of 325 ppm total DDT. Samples containing elevated total DDT concentrations were collected from the eastern area of the ECI Property. Approximately 35 samples had soil concentrations of total DDT above 10 ppm (the upper end of the regional background range [EPA, 2001]).
- Chlordane – Detected at a maximum reported concentration of 3.5 ppm from soil collected along the easternmost portion of the property.
- PCBs – Detected at a maximum concentration of 23.1 ppm (sum of Aroclors 1254 and 1260) from soil collected along the southeastern and easternmost area of the property.

Other chemical constituents also have been detected in soil samples from the ECI Property, including benzene hexachloride (BHC), a pesticide manufactured at the former Montrose plant (maximum concentration of 0.019 ppm as beta-BHC), and petroleum hydrocarbons (maximum concentration of 21,000 ppm as TPH-oil). The soils surrounding these sampling locations were excavated; the excavated soils were stored on-property until transported to a permitted hazardous waste landfill in January 2006, (see Section 2.2.2).

Available Groundwater Quality Data

Recent groundwater quality data are available for the area, but are not related to this investigation of the Historical Stormwater Pathway – South. EPA has separately conducted groundwater RI/FS activities, and is currently conducting Remedial Design activities for groundwater related to the Montrose Chemical and Del Amo Superfund Sites. Data from the period of Azko Coatings ownership of 20846 Normandie Avenue are also available, but again are not related to this investigation of the historical stormwater pathway.

Available Surface Water and Sediment Quality Data

Recent surface water and sediment quality data are available for the current stormwater pathway (e.g., within the LACFCD Project 685 stormwater drainage system, and other segments of that man-made conveyance), but are not related to this investigation of the historical stormwater pathway. EPA is separately conducting RI/FS activities for the Current Stormwater Pathway, as part of the Montrose Chemical Superfund Site.

2.2.4 Data Needs and Uses

Using available information, EPA concluded that additional soil data from the surface and subsurface soils within the historical stormwater pathway are needed to characterize the lateral and vertical extent of Montrose-related contamination within the Study Area. Additional findings of total DDT contamination in soil from this area are considered likely based on the total DDT concentrations found within the historical stormwater pathway north of Torrance Boulevard (during EPA's Kenwood Avenue Removal Action described in the following section) and at the ECI Property.

Data needs and uses for the project are identified through the DQO process presented in Appendix A (*Guidance for Data Quality Objectives Process*. EPA QA/G-4, EPA/600/R-96/055; EPA 2000 and 1994).

In accordance with the DQO process, for each media and/or task, the specific problems/principal study questions have been identified and evaluated individually through the DQO steps.

The data needs and uses resulting from the DQO process are summarized in Tables 2-4a and 2-4b. Tables 2-4a and 2-4b list the analytes of concern and present regulatory criteria/action level requirements for the analytes. The tables present a listing of regulatory limits and action levels, and identify the most protective (e.g., lowest) regulatory criteria where there are multiple regulatory criteria/action levels for a given analyte. These regulatory limits were taken into consideration in selecting appropriate methods and laboratory reporting levels as described in Sections 2.4.2 and 3.4.

Table 2-5 lists the analytical methods and laboratory reporting limits selected to meet these criteria. Some of the selected methods/analytes have higher reporting limits than regulatory criteria, due to practicable method limitations. The analytes with regulatory limits lower than laboratory reporting levels can be seen in Table 2-5. These comparisons are carried out for EPA Contract Laboratory Program (CLP) standard limits. Lower detection limits will be requested through the CLP special services program as further described in Section 2.4.2. The final sample detection levels may also be higher than initial reporting limits because of sample matrix effects. Detection levels for the individual

samples will be reported in the final data. Laboratory-specific method detection limits (MDLs) are significantly below reporting levels. Where reporting limits are higher than regulatory limits, the project team will use MDLs as needed for project decisions. Project decisions are not expected to be significantly affected by the higher detection levels. The selected methods are state-of-the-art and practicable.

2.3 Project Description and Schedule

2.3.1 Description of Work to be Performed

The purpose of the soil sampling is to obtain additional information on the extent of Montrose-related contamination within the Study Area, to assess potential human health risks, and determine if further action is needed. Soil sampling will consist of collecting surface and subsurface soil samples from the following areas:

- ECI Property within the historical stormwater pathway
- Residential properties east of ECI Property within the historical stormwater pathway
- ECI Property west of the historical stormwater pathway

Surface and subsurface soil samples will be collected using direct push (Geoprobe) technologies, where possible. A continuous core will be collected from each boring. Soil samples will be collected from specified depth intervals, composited, and analyzed at an offsite laboratory to provide an average contaminant concentration for each interval. Samples will be analyzed for Montrose-related contaminants (pesticides/PCBs). Selected soil samples will be analyzed for geotechnical parameters.

2.3.2 Schedule of Activities

Field reconnaissance activities are expected to take place from approximately June through August 2006. Mobilization and field activities will commence during June 2006 and continue through completion. The Soil Sampling Report is anticipated approximately 6 to 8 months following completion of field activities.

2.4 Data Quality Objectives

2.4.1 Project Quality Objectives

Project objectives and associated data needs were evaluated through the DQO process (EPA, 2000), which is described in Appendix A. The DQO process provides for the optimization of collected data and subsequent decisions.

2.4.2 Measurement Performance Criteria

The QA objective of this plan is to develop implementation procedures that will provide data of known and appropriate quality for the needs identified in previous sections. Data quality is assessed by representativeness, comparability, accuracy, precision, and completeness. These terms, the applicable procedures, and level of effort are described below.

The applicable QC procedures, quantitative target limits, and level of effort for assessing data quality are dictated by the intended use of the data and the nature of the analytical methods. Analytical parameters and applicable detection levels, analytical precision, accuracy, and completeness will be in alignment with needs identified in Section 2.2.4 are presented in Table 2-5.

Reporting detection levels/target detection limits listed in Table 2-5 are per method reporting limits, equivalent to contract-required detection levels (CRDLs). *Target* implies that final sample detection levels may be higher because of sample matrix effects. Detection levels for the individual samples will be reported in the final data. Also, some of the reporting levels in Table 2-5 are higher than regulatory limits identified in Tables 2-4a and 2-4b. These comparisons are carried out for EPA CLP standard detection limits (i.e., detection limits that are for routine procedures rather than low detection procedures; the low detection limits are laboratory specific). Lower detection will be requested through the CLP special services program as further described in Section 3.4. Detection levels for the individual samples will be reported in the final data. Laboratory-specific MDLs are significantly below reporting levels. Where reporting limits are higher than regulatory limits, the project team will use MDLs as needed for project decisions. Project decisions are not expected to be significantly affected by the higher detection levels. The selected methods are state-of-the-art and practicable.

Representativeness is a measure of how closely the results reflect the actual concentration or distribution of the chemical compounds in the matrix samples. Sampling plan design, sampling techniques, and sample-handling protocols (e.g., for storage, preservation, and transportation) have been developed, and are discussed in subsequent sections of this document. The proposed documentation will establish that protocols have been followed and sample identification and integrity ensured.

Comparability expresses the confidence with which one data set can be compared to another. Data comparability will be maintained using defined procedures and the use of consistent methods and consistent units. Actual detection limits will depend on the sample matrix and will be reported as defined for the specific samples.

Accuracy is an assessment of the closeness of the measured value to the true value. For samples, accuracy of chemical test results is assessed by spiking samples with known standards and establishing the average recovery. For a matrix spike, known amounts of a standard compound identical to the compounds being measured are added to the sample. A quantitative definition of average recovery accuracy is given in Section 5.3. The level of effort for accuracy measurements will be a minimum frequency of 1 in 20 samples analyzed.

Precision is a measure of the data spread when more than one measurement has been collected from the same sample. Precision can be expressed as the relative percent difference; a quantitative definition is given in Section 5.3. The level of effort for precision measurements will be a minimum of 1 in 20 samples analyzed.

Completeness is a measure of the amount of valid data obtained from the analytical measurement system and the complete implementation of defined field procedures. The quantitative definition of completeness is given in Section 5.3. The target completeness

objective will be 90 percent; the actual completeness may vary depending on the intrinsic nature of the samples. The completeness of the data will be assessed during QC reviews.

2.5 Special Training Requirements/Certification

All project staff working on the project will be health and safety trained, and will follow requirements specified in the HSP for this project (EPA, 2006). The HSP describes the specialized training required for personnel on this project and the documentation and tracking of this training.

2.6 Documentation and Records

Field documentation and records will be as described in Section 3.0 and the FSP. Laboratory documentation will be per: (1) methods and QA protocols listed in Section 3.0, and (2) EPA Regional Laboratory-specific standard operating procedures. Overall project documentation will be per EPA's Region 9 RAC Program Plan.

3.0 Measurement Data Acquisition

This section presents sampling process design and requirements for sampling methods, sample handling and custody, analytical methods, QC, and instrumentation for the sampling activities that will be conducted. Data acquisition requirements and data management for these sampling events are also addressed in this section.

3.1 Sampling Process Design

3.1.1 Background

Background is discussed in Section 2.2.

3.1.2 Schedule of Analyses

Field reconnaissance activities will take place prior to June 2006. Mobilization and field activities will commence during June 2006 and continue through about August 2006. The Soil Sampling Report is due for completion by early 2007.

3.1.3 Rationale for Sampling Design

The rationale for sampling design is described in DQO Step 7 in Appendix A.

3.2 Sampling Methods Requirements

Sampling method requirements are detailed in Section 5.0 of the companion FSP (EPA, 2006).

3.3 Sample Handling and Custody Requirements

A sample is physical evidence collected from a hazardous waste site, from the immediate environment, or from another source. Because of the potential evidentiary nature of samples, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence. In addition to field notebooks, there are a number of documents for tracking sample custody.

Field documents, including sample custody seals, COC records, and packing lists, will be obtained from the Regional Sample Control Center (RSCC) in EPA's Quality Assurance Office. COC procedures will be used to maintain and document sample collection and possession. After sample packaging, one or more of the following COC paperwork forms will be completed, as necessary, for the appropriate samples:

- Organic traffic report and COC record
- Inorganic traffic report and COC record
- EPA Region 9 COC Record

- Overnight shipping courier air bill

Copies of the above forms will be filled out and distributed per instructions for sample shipping; documentation in FSP LITE II electronic forms also will be used as applicable. If requested, completed field QA/QC summary forms will be sent to the RSCC at EPA's Region 9 Quality Assurance Office at the conclusion of each sampling event.

3.3.1 Chain-of-Custody

Because samples collected during any investigation could be used as evidence, their possession must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. COC procedures are followed to document sample possession.

3.3.1.1 Definition of Custody

A sample is under custody if one or more of the following criteria are met:

- It is in your possession.
- It is in your view, after being in your possession.
- It was in your possession and then you locked it up to prevent tampering.
- It is in a designated secure area.

3.3.1.2 Field Custody

In collecting samples for evidence, only enough to provide a good representation of the media being sampled will be collected. To the extent possible, the quantity and types of samples and sample locations are determined before the actual fieldwork. As few people as possible should handle samples.

The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly.

The SM, in coordination with EPA, determines whether proper custody procedures were followed during the fieldwork, and decides if additional samples are required.

3.3.1.3 Transfer of Custody and Shipment

Samples are accompanied by a COC record. When transferring samples, the individuals relinquishing and receiving the samples must sign, date, and note the time on the record. This record documents custody transfer from the sampler, often through another person, to the analyst at the laboratory.

Samples are packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate COC record accompanying each shipping container (one for each field laboratory, and one for samples driven to the laboratory). Shipping containers will be sealed with custody seals for shipment to the laboratory. Courier names, and other pertinent information, are entered in the "Received by" section of the COC record.

Whenever samples are split with a facility owner or agency, it is noted in the remarks section of the COC record. The note indicates with whom the samples are being split, and is signed by both the sampler and recipient. If the split is refused, this will be noted and signed by both parties. If a representative is unavailable or refuses to sign, this is noted in

the remarks section of the COC record. When appropriate, as in the case where the representative is unavailable, the COC record should contain a statement that the samples were delivered to the designated location at the designated time.

All shipments are accompanied by the COC record identifying its contents. The original record and yellow copy accompany the shipment to the laboratory; the pink copy is sent to be retained by the SM.

If sent by mail, the package is registered with return receipt requested. If sent by common carrier, a bill of lading is used. Freight bills, postal service receipts, and bills of lading are retained as part of the permanent documentation.

3.3.1.4 Laboratory Custody Procedures

A designated sample custodian accepts custody of the shipped samples, and verifies that the packing list sample numbers match those on the COC records. Pertinent information as to shipment, pickup, and courier is entered in the "Remarks" section. The custodian then enters the sample numbers into a bound notebook, which is arranged by project code and station number.

The laboratory custodian uses the sample identification number or assigns a unique laboratory number to each sample, and is responsible for seeing that all samples are transferred to the proper analyst or stored in the appropriate secure area.

The custodian distributes samples to the appropriate analysts. Laboratory personnel are responsible for the care and custody of samples from the time they are received, until the sample is exhausted or returned to the custodian. The data from sample analyses are recorded on the laboratory report form.

When sample analyses and necessary QA checks have been completed in the laboratory, the unused portion of the sample will be disposed of properly. All identifying stickers, data sheets, and laboratory records are retained as part of the documentation. Sample containers and remaining samples are disposed of in compliance with all federal, state, and local regulatory requirements.

3.3.2 Custody Seals

When samples are shipped to the laboratory, they must be placed in containers sealed with custody seals. One or more custody seals must be placed on each side of the shipping container (cooler).

3.3.3 Field Notebooks

Typical field information to be entered in the field notebook is included in the companion FSP (Section 6.8) (EPA, 2006). In addition to COC records, a bound field notebook must be maintained by each STL to provide a daily record of significant events, observations, and measurements during field investigations. All entries should be signed and dated. It should be kept as a permanent record.

These notebooks are intended to provide sufficient data and observations to enable participants to reconstruct events that occurred during the project, and to refresh the memory of the field personnel if called upon to give testimony during legal proceedings. In

a legal proceeding, notes, if referred to, are subject to cross-examination and are admissible as evidence.

3.3.4 Corrections to Documentation

All original data recorded in field notebooks, sample identification tags, COC records, and receipts-for-sample forms will be written with waterproof ink, unless prohibited by weather conditions. None of these accountable serialized documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on an accountable document assigned to one team, the team leader may make corrections simply by drawing a single line through the error and entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

3.4 Analytical Methods Requirements

Project analytes, methods and detection limits have been listed in Table 2-5. Soil samples will be analyzed for pesticides and PCBs by the CLP. These analyses will follow the applicable EPA statement of work (SOW). The SOW specifies methodology, QA/QC, and documentation. EPA CLP methodology and QC for low concentration analyses will be implemented as needed. Table 2-5 shows the project-required detection levels as well as the CLP contract- required detection levels. As described in Section 2.2.4 and as shown in Table 2-5, some regulatory or risk limits are lower than the standard CLP limits. For these cases, the analyses will be carried out in accordance with special services provisions currently available under the CLP. A low-level organic SOW, selective ion monitoring (SIM) methodology, or larger sample volumes may be used to attain lower-level organic detection limits than the listed limits for routine CLP procedures; the lower limits are laboratory specific, thus have not been listed. Where the lowest regulatory limit is lower than the analytical reporting limit (Table 2-5), the laboratory-specific detection levels are expected to be significantly below the listed reporting limit. The selected methods will be state-of-the-art and practicable such as SIM analyses.

IDW analyses will similarly be analyzed by the CLP for volatiles, metals, and pesticides/PCBs. Other parameters will be analyzed by the regional laboratory per the specifications presented in Appendix B.

The distribution of analyses may change at the time of analysis, depending on implementation of additional procedures at the regional laboratory, as well as capacity.

3.5 Quality Control Requirements

QC requirements are detailed in the subsections below.

3.5.1 Field QC Procedures

QC requirements related to the sample collection process (i.e., design, methods, handling, and custody) have been discussed in the previous sections of this document.

Field QC samples include field duplicates, field blanks, and laboratory QC samples (for example, matrix spike and matrix spike duplicates [MS/MSDs]). QC samples will be collected immediately following collection of target samples, and using the same procedures as the collection of the target sample. These procedures are presented in the companion FSP.

3.5.2 Laboratory Procedures

Laboratory QC procedures will be conducted according to the following specifications:

- Analytical methodology according to the specific methods listed in Table 2-5 and Appendix B
- Instrument calibrations and standards as defined in specific methods listed in Appendix B and the CLP SOW
- Laboratory blank measurements at a minimum of 5 percent or 1-per-batch frequency
- Accuracy and precision measurements at a minimum of 1 in 20, 1 per set
- Data reduction and reporting according to the specific methods listed in Table 2-5
- Laboratory documentation equivalent to the CLP SOW or the specifications in Appendix B

3.6 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

Instrument maintenance logbooks are to be maintained in laboratories at all times. The logbooks, in general, shall contain a schedule of maintenance, as well as a complete history of past maintenance, both routine and nonroutine.

Preventive maintenance is to be performed according to the procedures described in the manufacturer's instrument manuals, including lubrication, source cleaning, detector cleaning, and the frequency of such maintenance. Chromatographic carrier gas-purification traps, injector liners, and injector septa are cleaned or replaced on a regular basis. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance will be performed when an instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decrease in sensitivity, or failure to meet one or another of the QC criteria.

Instrument downtime shall be minimized by keeping adequate supplies of all expendable items, where expendable means an expected lifetime of less than 1 year. These items include gas tanks, gasoline filters, syringes, septa, gas chromatography (GC) columns and packing, ferrules, printer paper and ribbons, pump oil, jet separators, open-split interfaces, and mass spectroscopy filaments.

Preventive maintenance for field equipment (e.g., pH meter) will be carried out in accordance with procedures and schedules outlined in the operation and maintenance handbook for the particular model.

3.7 Instrument Calibration and Frequency

The following subsections review instrument calibration and frequency information.

3.7.1 Field Calibration Procedures

For water analyses, if any, field equipment requiring calibration includes pH, conductivity, temperature, dissolved oxygen, and oxidation/reduction potential meters. These meters will be calibrated before the start of work and at the end of the sampling day. Any instrument “drift” from prior calibration should be recorded in a field notebook.

Calibration will be in accordance with procedures and schedules outlined in the operation and maintenance manual for the particular instrument.

Calibrated equipment will be uniquely identified either by using the manufacturer’s serial number or by other means. A label with the identification number and the date when the next calibration is due will be physically attached to the equipment. If this is not possible, records traceable to the equipment will be readily available for reference. In addition, the results of calibrations and records of repairs will be recorded in a logbook.

Scheduled periodic calibration of testing equipment does not relieve field personnel of the responsibility of employing properly functioning equipment. If an individual suspects an equipment malfunction, the device must be removed from service, it must be tagged so that it is not inadvertently used, and the appropriate personnel must be notified so that a recalibration can be performed or a substitute piece of equipment can be obtained.

Equipment that fails calibration or becomes inoperable during use will be removed from service and either segregated to prevent inadvertent use, or tagged to indicate it is out of calibration. Such equipment will be repaired and satisfactorily recalibrated. Equipment that cannot be repaired will be replaced.

Results of activities performed using equipment that has failed recalibration will be evaluated. If the activity results are adversely affected, the results of the evaluation will be documented and the task manager and QA/QC reviewer will be notified.

3.7.2 Laboratory Calibration Procedures

Laboratory calibration procedures are specified in the referenced methods in Appendix B for all parameters listed in Table 2-5. All calibrations, at a minimum, shall be at the following level of effort:

- Initial calibration for all methods will include, at a minimum, three-point calibration before a run.
- Continuing calibration for all methods will include a mid-range calibration standard after every 10th sample or every 12 hours, whichever is more frequent.

3.8 Data Acquisition Requirements (Nondirect Measurements)

Previously collected data and other information will be used to assist decisionmaking regarding activities during the soil investigation. The data have been tabulated and are shown in Section 2.0 above; the past data will be added to the electronic database as needed.

3.9 Data Management

Data for all measured parameters will undergo two levels of review and validation: (1) at the laboratory, and (2) outside the laboratory as described in Section 5.0. For this project, it is anticipated that samples will be submitted to the Region 9 laboratory and/or designated CLP laboratories and contract laboratories, and that validated data will be provided to CH2M HILL. Following receipt, validated data will be input into the database to facilitate database inquiries and report preparation. The data will be stored in the databases with all laboratory qualifiers included. Established data queries and formats developed during the previous work assignments will be adapted for incorporation of laboratory data from files, provided by EPA's QAO, to files compatible with the project database. The database will be available to EPA, or provided to others at EPA's request. Major components for complete data management will be as follows:

- **Data Conversion/Manipulation/Review.** Reports of sample-quality data from sampling are received from the QAO in hardcopy or electronic format. These data must be converted, input, reviewed, and QC checked.

In addition, available data from other sources may be incorporated into the database. These data will need to be manually input, output, reviewed, QC checked, then uploaded into the database.

- **Preparation of Tables.** Data tables will be prepared following receipt of validated data from the QAO following each sample event of the work assignment. Queries will be created for the database to generate updated tables. These tables will be used by the project team to assess the nature and extent of Montrose-related soil contamination within the Historic Stormwater Pathway – South.
- **Database Documentation.** An update of the database and complete documentation will be performed as needed. The commands, filenames, and general operating procedures for all the data queries will be documented as directed by the EPA WAM. This documentation will be provided to EPA and transferred to others at EPA's request.

4.0 Assessment/Oversight

4.1 Assessment and Response Actions

The review team and the SM will monitor the performance of the QA procedures. If problems arise and the WAM directs the SM, the review team will conduct field audits, which currently are not scheduled or included in the SOW. Audits may be scheduled to evaluate (1) the execution of sample identification, COC procedures, field notebooks, sampling procedures, and field measurements; (2) whether trained personnel staffed the sample event; (3) whether equipment was in proper working order (i.e., calibration); (4) the availability of proper sampling equipment; (5) whether appropriate sample containers, sample preservatives, and techniques were used; (6) whether sample packaging and shipment were appropriate; and (7) whether QC samples were properly collected. At a minimum, one unannounced assessment of Issues 5, 6, or 7 will be implemented once per year.

The analyses are expected to be performed by the EPA CLP, EPA regional laboratory and contract laboratories as described in Section 3.4. The distribution of analyses may change at the time of analyses depending on implementation of additional procedures at the regional laboratory as well as capacity. The QA of the regional laboratory is managed by the EPA QAO. Laboratories subcontracted to CH2M HILL, if any, will be selected based on prior performance on regional Superfund projects. Additionally, onsite audits or performance evaluation samples will be administered by the project QAO, as necessary and authorized by the EPA WAM.

Audits will be followed up with an audit report prepared by the reviewer. The auditor will also debrief the laboratory or the field team at the end of the audit and request that the laboratory or field team comply with the corrective action request, if applicable.

4.2 Reporting and Resolution of Issues

If QC audits result in detection of unacceptable conditions or data, the SM will be responsible for coordinating with the EPA WAM to develop and initiate corrective action. The WAM will be notified if nonconformance is of program significance or requires special expertise not normally available to the project team. In such cases, the WAM will decide whether any corrective action should be pursued. Corrective action may include the following:

- Reanalyzing samples if holding time criteria permit
- Resampling and analyzing
- Evaluating and amending sampling and analytical procedures
- Accepting data acknowledging a level of uncertainty

4.3 Reports to Management

The SM or WAM may request that a QA report be made to the WAM on the performance of sample collection and data quality. The report will include the following:

- Assessment of measurement data accuracy, precision, and completeness
- Results of performance audits
- Results of systems audits
- Significant QA problems and recommended solutions

Monthly progress reports will summarize overall project activities and any problems encountered. QA reports generated on sample collection and data quality will focus on specific problems encountered and solutions implemented. Alternatively, in lieu of a separate QA report, sampling and field measurement data quality information may be summarized and included in the final reports summarizing field activities. The objectives, activities performed, overall results, sampling, and field measurement data quality information of the project will be summarized and included in the final field activities reports along with any QA reports.

5.0 Data Validation and Usability

5.1 Data Review, Validation, and Verification Requirements

All data for all parameters will undergo two levels of review and validation: (1) at the laboratory, and (2) outside the laboratory by the EPA Quality Assurance Management Section (QAMS) or their designee. Data will be reviewed outside the laboratory at the following level of effort:

- Ninety percent of the sample analytical batches will be subject to a Tier 2 review for all the analytical parameters, detections, and nondetections, per the regional EPA QAO guidance. (For CLP analyses, this corresponds to Level 1B.) Also, 10 percent of the analytical batches will be subject to a Tier 3 review for all parameters, detections, and nondetections. The analytical batches selected for Tier 3 review will be selected at random, unless a new laboratory is performing the analyses. In this instance, the first analytical batch should undergo the Tier 3 review as a proactive measure.
- Tier 2 review has been selected to provide for review of all the QA/QC summary forms in accordance with EPA CLP National Functional Guidelines for Inorganic/Organic Data review. This is to include all calibrations and internal standards and flagging of the individual results, as opposed to review of a subset of the QC data as is the case for Tier 1A review. This Tier 2 corresponds to the CLP Tier 1B review level. Tier 2 (CLP Tier 1B) economizes the laboratory data review compared to Tier 3 by limiting the review to QC summary data as opposed to raw data checks. Review of QC summary data that includes all QC parameters provides for the needed comprehensive coverage for this remedial investigation. The review will compare QC summary data to acceptable limits and will qualify the individual associated data points per guidelines. The review will also compare detects in blanks to associated samples and qualify/modify sample concentrations per guidelines.

The level of effort for data validation described in this section is based on the objectives of this project and deal with quantitative evaluation of samples at trace levels for all analytes. The full database needs consistent flags/qualifiers for comparable and reproducible data. This level of effort should accomplish that. These levels of effort are appropriate because data are compared to regulatory limits used for risk assessments and quantitative comparisons to establish trends at trace levels. Quantitative use at trace levels applies to all analytes, not just a subset of the target analytes. All analytes are contaminants of concern, even though, for example, arsenic may be detected more often than the other analytes. Establishing the validity of nondetect results is as important as the detected results for the RI, thus both detection and nondetection results will be reviewed.

5.2 Validation and Verification Methods

Initial data reduction, validation, and reporting at the laboratory will be performed as described in the laboratory standard operating procedures.

Independent data validation by EPA or their designee will follow EPA *Contract Laboratory Program National Functional Guidelines for Inorganic/Organic Data Review* (EPA, 1994, 1999, 2001b, and 2004) and the regional guidance as described above.

5.3 Reconciliation with Data Quality Objectives

Results obtained from the project will be reconciled with the requirements specified in Tables 2-4a and 2-4b. Assessment of data for precision, accuracy, and completeness will be per the following quantitative definitions.

5.3.1 Precision

If calculated from duplicate measurements:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2) / 2}$$

RPD = relative percent difference
 C_1 = larger of the two observed values
 C_2 = smaller of the two observed values

If calculated from three or more replicates, use relative standard deviation (RSD) rather than relative percent difference (RPD):

$$RSD = (s / \bar{y}) \times 100\%$$

RSD = relative standard deviation
 s = standard deviation
 \bar{y} = mean of replicate analyses

Standard deviation, s , is defined as follows:

$$s = \sqrt{\frac{\sum_{i=1}^n (y_i / \bar{y})^2}{n - 1}}$$

s = standard deviation
 y_i = measured value of the i^{th} replicate
 \bar{y} = mean of replicate analyses
 n = number of replicates

5.3.2 Accuracy

For measurements where matrix spikes are used:

$$\%R = 100\% \times \left[\frac{S - U}{C_{sa}} \right]$$

- %R = percent recovery
 S = measured concentration in spiked aliquot
 U = measured concentration in unspiked aliquot
 C_{sa} = actual concentration of spike added

For situations where a standard reference material (SRM) is used instead of or in addition to matrix spikes:

$$\%R = 100\% \times \left[\frac{C_m}{C_{sm}} \right]$$

- %R = percent recovery
 C_m = measured concentration of SRM
 C_{sm} = actual concentration of SRM

5.3.3 Completeness (Statistical)

Defined as follows for all measurements: _____

$$\%C = 100\% \times \left[\frac{V}{T} \right]$$

- %C = percent completeness
 V = number of measurements judged valid
 T = total number of measurements

5.3.4 Representativeness

Representativeness is a measure of how closely the results reflect the actual concentration or distribution of the chemical compounds in the matrix samples. Sampling plan design, sampling techniques, and sample-handling protocols (for example, for storage, preservation, and transportation) have been developed, and are discussed in previous sections of this document. The proposed documentation will be reviewed to establish that protocols have been followed, that the number and location of samples are per plans, and that sample identification and integrity have been ensured.

6.0 References

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Tables

Table 2-1. Summary of Detected Pesticides/PCBs and TPH in Soil Samples
 ECI Property, 20846 Normandie Avenue, Torrance, CA

SOIL INVESTIGATION FOR HISTORICAL STORMWATER PATHWAY - SOUTH
 MONTROSE CHEMICAL SUPERFUND SITE, LOS ANGELES COUNTY, CALIFORNIA

Boring ID	Sample Number	Sample Date	Depth (feet)	Total DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Chlordane	cis-Chlordane	gamma-Chlordane	Dieldrin	Alpha-BHC	Beta-BHC	Delta-BHC	Gamma-BHC	Aroclor 1254	Aroclor 1260	TPH-Gas	TPH-Diesel	TPH-Oil
				mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
EX-SB05-SSM-05	EX-SB05-SSM-05	05/26/05	5	9.5	1.1	0.63	7.8	1 U	0.01 U	0.01 U	0.01 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.029			
EX-SB05-SSQ-05	EX-SB05-SSQ-05	05/26/05	5	0.9	0.43	0.11	0.31	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U		
EX-SB05-SSU-05	EX-SB05-SSU-05	06/02/05	5	0.8	0.25	0.15	0.36	0.1 U	0.01 U	0.01 U	0.01 U	0.02 U	0.02 U	0.02 U	0.02 U	0.051	0.017			
EX-SB05-SW-05	EX-SB05-SW-05	03/17/05	5	3.3	1.3	0.34	1.7	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.038	1 U	170	480
EX-SB05-SW-10	EX-SB05-SW-10	03/17/05	10	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1 U	97	230
EX-SB05-SWEE-05	EX-SB05-SWEE-05	06/03/05	5	53.9	6.1	1.8	46	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.005 U	0.005 U			
EX-SB05-SWFF-05	EX-SB05-SWFF-05	06/08/05	5	0.5	0.2	0.13	0.15	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.037	0.018		
EX-SB05-SWH-05	EX-SB05-SWH-05	05/18/05	5	1.7	0.56	0.26	0.91	0.005 U	0.0005 U	0.0005 U	0.002J	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.038	0.02			
EX-SB05-SWP-05	EX-SB05-SWP-05	05/26/05	5	1.4	0.86	0.11	0.43	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB05-SWT-05	EX-SB05-SWT-05	06/02/05	5	4.0	0.26	0.49	3.2	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.026			
EX-SB09-BE-035	EX-SB09-BE-035	03/17/05	3.5	10.8	0.82	0.73	9.2	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB09-BTE-05	EX-SB09-BTE-05	05/18/05	5	0.3	0.077	0.047	0.14	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB09-BTF-05	EX-SB09-BTF-05	05/18/05	5	2.4	0.45	0.34	1.6	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.01 J			
EX-SB09-BTI-05	EX-SB09-BTI-05	05/26/05	5	0.0	0.0026	0.0025	0.011	0.0091	0.001 J	0.0016	0.002 J	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB09-BTO-05	EX-SB09-BTO-05	06/02/05	5	0.1	0.099	0.006	0.015	0.016	0.0028	0.003	0.0033	0.001 U	0.001 U	0.001 U	0.001 U	0.005 U	0.007 J			
EX-SB09-BTT-06	EX-SB09-BTT-06	06/09/05	6	3.1	1	0.19	1.9	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U			
EX-SB09-BW-035	EX-SB09-BW-035	03/17/05	3.5	0.9	0.07	0.04	0.74	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB09-SE-02	EX-SB09-SE-02	03/17/05	2	2.3	0.39	0.61	1.3	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.076	0.034			
EX-SB09-SEC-03	EX-SB09-SEC-03	05/18/05	3	2.4	0.5	0.73	1.2	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.081	0.046			
EX-SB09-SED-03	EX-SB09-SED-03	05/18/05	3	18.1	2.6	2.5	13	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.073	0.042			
EX-SB09-SEG-03	EX-SB09-SEG-03	05/26/05	3	6.9	1	0.63	5.3	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.005 U	0.005 U			
EX-SB09-SEH-03	EX-SB09-SEH-03	05/26/05	2	12.3	1.9	1.3	9.1	1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.055			
EX-SB09-SEL-03	EX-SB09-SEL-03	06/02/05	3	2.4	0.94	0.75	0.69	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.045	0.024			
EX-SB09-SEP-03	EX-SB09-SEP-03	06/03/05	3	5.7	2.4	2.3	1	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.095	0.054			
EX-SB09-SER-03	EX-SB09-SER-03	06/09/05	3	2.7	0.64	0.28	1.8	0.005 U	0.0027	0.0005 U	0.0024	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.02	0.022			
EX-SB09-SN-02	EX-SB09-SN-02	03/17/05	2	5.9	0.91	1.2	3.8	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.08	0.042			
EX-SB09-SNA-03	EX-SB09-SNA-03	05/18/05	3	0.7	0.075	0.22	0.43	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.008 J			
EX-SB09-SNB-03	EX-SB09-SNB-03	05/18/05	3	2.9	0.66	0.73	1.5	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.019			
EX-SB09-SNJ-03	EX-SB09-SNJ-03	05/26/05	3	8.2	1.4	1.1	5.7	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.075	0.026			
EX-SB09-SNQ-03	EX-SB09-SNQ-03	06/09/05	3	1.4	0.66	0.44	0.31	0.05 U	0.006 J	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.042	0.025			
EX-SB09-SS-02	EX-SB09-SS-02	03/17/05	2	0.2	0.055	0.065	0.067	0.096	0.009	0.018	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB09-SSK-03	EX-SB09-SSK-03	05/26/05	3	8.4	1.8	0.78	5.8	1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.005 U			
EX-SB09-SSM-03	EX-SB09-SSM-03	06/02/05	3	2.3	0.63	0.27	1.4	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.027	0.017			
EX-SB09-SSS-03	EX-SB09-SSS-03	06/09/05	3	2.0	0.65	0.32	1	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.031	0.023			
EX-SB09-SW-02	EX-SB09-SW-02	03/17/05	2	0.0	0.001 J	0.0006 J	0.0027	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB09-SWN-03	EX-SB09-SWN-03	06/02/05	3	1.2	0.62	0.2 J	0.37	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.023	0.019			
EX-SB20-BE-09	EX-SB20-BE-09	05/17/05	9	4.2	2.7	0.8	0.68	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U			
EX-SB20-BTCC-11	EX-SB20-BTCC-11	06/08/05	11	17.9	12	1.5	4.4	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.001 J	0.018	0.0041	0.0005 U	0.005 U	0.005 U			
EX-SB20-BTCC-12	EX-SB20-BTCC-12	06/09/05	12	21.4	8.5	3.2	9.7	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.019	0.0025 U	0.0062	0.005 U	0.005 U			
EX-SB20-BTGG-11	EX-SB20-BTGG-11	06/09/05	11	21.8	16	1	4.8	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.085	0.056			
EX-SB20-BTI-11	EX-SB20-BTI-11	05/26/05	11	2.4	1.3	0.65	0.4	0.25 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.005 U	0.005 U			
EX-SB20-BTJ-11	EX-SB20-BTJ-11	05/26/05	11	4.8	2.6	1	1.2	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.005 U	0.005 U			
EX-SB20-BTU-11	EX-SB20-BTU-11	06/03/05	11	0.1	0.067	0.015	0.057	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.007 J			
EX-SB20-SE-01	EX-SB20-SE-01	05/18/05	1	-	--	--	--	--	--	--	--	--	--	--	--	0.033	0.025			
EX-SB20-SE-03	EX-SB20-SE-03	05/17/05	3	7.2	0.53	2.1	4.6	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SE-07	EX-SB20-SE-07	05/17/05	7	32.4	4.1	4.3	24	0.005 U	0.0005 U	0.0005 U	0.015	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SEAA-03	EX-SB20-SEAA-03	06/09/05	3	4.5	0.98	0.88	2.6	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	6.1	1.8			
EX-SB20-SEB-07	EX-SB20-SEB-07	05/26/05	7	0.0	0.0021	0.0079	0.028	0.061	0.015	0.017	0.0032	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SEQ-03	EX-SB20-SEQ-03	06/02/05	3	0.1	0.015	0.028	0.058	0.12	0.029	0.028	0.004	0.001 U	0.001 U	0.001 U	0.001 U	0.005 U	0.005 U			
EX-SB20-SES-07	EX-SB20-SES-07	06/03/05	7	1.3	0.2 J	0.26	0.88	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.005 U	0.007 J			
EX-SB20-SN-01	EX-SB20-SN-01	05/18/05	1	-	--	--	--	--	--	--	--	--	--	--	--	0.005 U	0.025			
EX-SB20-SN-03	EX-SB20-SN-03	05/17/05	3	1.4	0.11	0.15	1.1	0.1 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.005 U	0.005 U			
EX-SB20-SN-07	EX-SB20-SN-07	05/17/05	7	3.4	0.26	0.099	3	0.35	0.031	0.05	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U			
EX-SB20-SNA-07	EX-SB20-SNA-07	05/26/05	7	24.7	1.3	2.4	21	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SNBB-07	EX-SB20-SNBB-07	06/08/05	7	0.0	0.001 J	0.0058	0.013	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SNDD-03	EX-SB20-SNDD-03	06/09/05	3	0.4	0.1	0.087	0.17	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.017	0.005 U			

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 ECI Property, 20846 Normandie Avenue, Torrance, CA

SOIL INVESTIGATION FOR HISTORICAL STORMWATER PATHWAY - SOUTH
 MONTROSE CHEMICAL SUPERFUND SITE, LOS ANGELES COUNTY, CALIFORNIA

Boring ID	Sample Number	Sample Date	Depth (feet)	Total DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Chlordane	cis-Chlordane	gamma-Chlordane	Dieldrin	Alpha-BHC	Beta-BHC	Delta-BHC	Gamma-BHC	Aroclor 1254	Aroclor 1260	TPH-Gas	TPH-Diesel	TPH-Oil
				mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
EX-SB20-SNR-03	EX-SB20-SNR-03	06/02/05	3	7.1	1.1	1.2	4.8	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.021			
EX-SB20-SNR-07	EX-SB20-SNR-07	06/03/05	7	1.1	0.2 J	0.23	0.65	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.044	0.02			
EX-SB20-SS-01	EX-SB20-SS-01	05/18/05	1	-	--	--	--	--	--	--	--	--	--	--	--	0.025	0.032			
EX-SB20-SS-03	EX-SB20-SS-03	05/17/05	3	21.3	3.4	1.9	16	0.005 U	0.0005 U	0.0005 U	0.0096	0.0005 U	0.0025	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SS-07	EX-SB20-SS-07	05/17/05	7	23.7	8.4	2.3	13	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SSC-03	EX-SB20-SSC-03	05/26/05	3	11.7	1.1	3.3	7.3	1.2 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.005 U	0.005 U			
EX-SB20-SSD-07	EX-SB20-SSD-07	05/26/05	7	2.6	0.21	0.2	2.2	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SSE-03	EX-SB20-SSE-03	05/26/05	3	0.8	0.2	0.12	0.49	0.12 U	0.012 U	0.012 U	0.012 U	0.012 U	0.012 U	0.012 U	0.012 U	0.043	0.023			
EX-SB20-SSF-07	EX-SB20-SSF-07	05/25/05	7	1.5	0.39	0.16	0.95	0.25 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025	0.005 U			
EX-SB20-SSFF-03	EX-SB20-SSFF-03	06/09/05	3	1.0	0.53	0.14	0.34	0.05 U	0.005 J	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.055	0.035			
EX-SB20-SSM-03	EX-SB20-SSM-03	06/02/05	3	0.1	0.02	0.012	0.057	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.001 U	0.001 U	0.001 U	0.001 U	0.005 U	0.009 J			
EX-SB20-SSO-03	EX-SB20-SSO-03	06/02/05	3	12.9	1.2	1.9	9.8	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.005 U			
EX-SB20-SW-01	EX-SB20-SW-01	05/18/05	1	-	--	--	--	--	--	--	--	--	--	--	--	0.005 U	0.028			
EX-SB20-SW-03	EX-SB20-SW-03	05/17/05	3	7.0	0.87	1.4	4.7	0.005 U	0.0005 U	0.0005 U	0.0009J	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SW-07	EX-SB20-SW-07	05/17/05	7	0.3	0.096	0.044	0.13	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SWAA-07	EX-SB20-SWAA-07	06/08/05	7	27.7	2.8	0.88	24	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0069	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB20-SWBB-03	EX-SB20-SWBB-03	06/09/05	3	12.3	4	1.8	6.5	0.025 U	0.0025 U	0.0025 U	0.006 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U			
EX-SB20-SWEE-03	EX-SB20-SWEE-03	06/09/05	3	1.7	0.31	0.32	1.1	0.096	0.011	0.021	0.008 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U			
EX-SB20-SWG-03	EX-SB20-SWG-03	05/26/05	3	36.6	6.2	2.4	28 E	2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.005 U	0.005 U			
EX-SB20-SWH-07	EX-SB20-SWH-07	05/26/05	7	0.4	0.11	0.021	0.25	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.01 J			
EX-SB20-SWL-03	EX-SB20-SWL-03	06/02/05	3	6.4	0.92	1.4	4.1	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.024 U			
EX-SB20-SWN-03	EX-SB20-SWN-03	06/02/05	3	10.4	0.96	1.7	7.7	0.5 U	0.05 U	0.05 U	0.05 U	0.1 U	0.1 U	0.1 U	0.1 U	0.005 U	0.005 U			
EX-SB20-SWP-07	EX-SB20-SWP-07	06/03/05	7	10.6	2.4	1.3	6.9	0.5 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.005 U	0.026			
EX-SB20-SWT-03	EX-SB20-SWT-03	06/02/05	3	7.1	1.1	1.2	4.8													
EX-SB32-BT-12	EX-SB32-BT-12	05/18/05	12	0.5	0.041	0.072	0.37	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.026			
EX-SB32-BTC-10	EX-SB32-BTC-10	06/02/05	10	7.1	0.24	0.49	6.4	0.11	0.022	0.028	0.014	0.001 U	0.001 U	0.001 U	0.001 U	0.045	0.079			
EX-SB32-BTH-12	EX-SB32-BTH-12	06/09/05	12	10.3	0.51	0.84	8.9	0.025 U	0.0025 U	0.0025 U	0.004 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.087	0.038			
EX-SB32-NA-09	EX-SB32-NA-09	06/02/05	9	3.6	0.2	0.87	2.5	0.2	0.04	0.044	0.0025 U	0.005 U	0.005 U	0.005 U	0.005 U	0.031	0.054			
EX-SB32-SE-09	EX-SB32-SE-09	05/18/05	9	4.8	0.46	1.8	2.5	3.5	0.49	0.46	0.08J	0.025 U	0.025 U	0.025 U	0.025 U	0.005 U	0.005 U			
EX-SB32-SEB-09	EX-SB32-SEB-09	06/02/05	9	3.2	0.17	2.5	0.57	0.43	0.053	0.048	0.18	0.005 U	0.005 U	0.005 U	0.005 U	0.052	0.005 U			
EX-SB32-SEF-09	EX-SB32-SEF-09	06/09/05	9	0.8	0.036	0.31	0.5	0.2	0.038	0.043	0.024	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.068	0.046			
EX-SB32-SN-09	EX-SB32-SN-09	05/18/05	9	0.8	0.087	0.47	0.26	0.17	0.02	0.021	0.02 J	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.062			
EX-SB32-SNE-09	EX-SB32-SNE-09	06/09/05	9	1.1	0.062	0.69	0.33	0.2	0.036	0.037	0.1	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.029	0.06			
EX-SB32-SS-09	EX-SB32-SS-09	05/18/05	9	0.1	0.01	0.1	0.037	0.03 J	0.003 J	0.0086	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.018			
EX-SB32-SSD-09	EX-SB32-SSD-09	06/02/05	9	0.6	0.01 J	0.42	0.13	0.05 U	0.005 U	0.005 U	0.065	0.01 U	0.01 U	0.01 U	0.01 U	0.005 U	0.018			
EX-SB32-SSG-09	EX-SB32-SSG-09	06/09/05	9	0.6	0.023	0.52	0.078	0.011	0.0025	0.0024	0.026	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.021			
EX-SB32-SW-09	EX-SB32-SW-09	05/18/05	9	0.2	0.025	0.13	0.046	0.025 U	0.0025 U	0.0025 U	0.004 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.009 J			
EX-SB35-BTI-09	EX-SB35-BTI-09	05/17/05	9	1.7	0.15	0.21	1.3	0.1 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.005 U	0.005 U			
EX-SB35-BTM-09	EX-SB35-BTM-09	05/26/05	9	2.9	1.8	0.37	0.72	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-BTO-09	EX-SB35-BTO-09	06/03/05	9	7.5	1.8	1.5	4.2	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.11	0.067			
EX-SB35-BTV-12	EX-SB35-BTV-12	06/09/05	12	11.4	8.9	0.58	1.9	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.059	0.096			
EX-SB35-SEG-03	EX-SB35-SEG-03	05/17/05	3	0.7	0.034	0.11	0.52	0.043	0.0099	0.01	0.0029	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SEH-07	EX-SB35-SEH-07	05/17/05	7	0.6	0.032	0.093	0.5	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U			
EX-SB35-SEL-07	EX-SB35-SEL-07	05/26/05	7	4.0	0.2	0.5	3.3	0.005 U	0.0005 U	0.0005 U	0.02	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SEQ-07	EX-SB35-SEQ-07	06/03/05	7	0.4	0.17	0.044	0.16	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.01 J	0.01 J			
EX-SB35-SNE-03	EX-SB35-SNE-03	05/17/05	3	2.3	0.19	0.56	1.5	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SNF-07	EX-SB35-SNF-07	05/17/05	7	0.7	0.033	0.095	0.62	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SNN-07	EX-SB35-SNN-07	06/03/05	7	4.1	1.6	0.16	2.3	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.034	0.024			
EX-SB35-SNS-07	EX-SB35-SNS-07	06/09/05	7	0.2	0.13	0.014	0.066	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.007 J			
EX-SB35-SSC-03	EX-SB35-SSC-03	05/17/05	3	2.5	0.19	0.44	1.9	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SSD-07	EX-SB35-SSD-07	05/17/05	7	9.2	0.83	0.79	7.6	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SSK-07	EX-SB35-SSK-07	05/26/05	7	7.8	0.57	0.9	6.3	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			
EX-SB35-SSP-07	EX-SB35-SSP-07	06/03/05	7	2.5	1.4	0.17	0.92	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.02	0.02 J			
EX-SB35-SSU-07	EX-SB35-SSU-07	06/09/05	7	2.0	1.2	0.32	0.47	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.022			
EX-SB35-SWA-03	EX-SB35-SWA-03	05/17/05	3	7.8	0.63	1.2	6	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U			
EX-SB35-SWB-07	EX-SB35-SWB-07	05/17/05	7	5.6	1	1.2	3.4	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U			

Table 2-1. Summary of Detected Pesticides/PCBs and TPH in Soil Samples
 ECI Property, 20846 Normandie Avenue, Torrance, CA

SOIL INVESTIGATION FOR HISTORICAL STORMWATER PATHWAY - SOUTH
 MONTROSE CHEMICAL SUPERFUND SITE, LOS ANGELES COUNTY, CALIFORNIA

Boring ID	Sample Number	Sample Date	Depth (feet)	Total DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Chlordane	cis-Chlordane	gamma-Chlordane	Dieldrin	Alpha-BHC	Beta-BHC	Delta-BHC	Gamma-BHC	Aroclor 1254	Aroclor 1260	TPH-Gas	TPH-Diesel	TPH-Oil
				mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
EX-SB35-SWJ-07	EX-SB35-SWJ-07	05/26/05	7	5.6	0.73	1	3.9	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.081	0.039			
EX-SB35-SWR-07	EX-SB35-SWR-07	06/03/05	7	9.2	1	1.7	6.5	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.16	0.092			
EX-SB35-SWT-07	EX-SB35-SWT-07	06/09/05	7	0.9	0.28	0.11	0.48	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U			
SB-01	SB-01-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	300	1000
SB-01	SB-01-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	5 J	7 J
SB-02	SB-02-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	6 J	4 J
SB-02	SB-02-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	10 J	4 J
SB-03	SB-03-020805-01	02/08/05	0-1	0.0	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	2.4	0.31	--	--	--
SB-03	SB-03-020805-03	02/08/05	2-3	0.0	0.002 J	0.0005 U	0.001 J	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-03	SB-03-020805-05	02/08/05	4-5	0.0	0.0022	0.0007 J	0.0028	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	--	--	0.37 U	300	980
SB-03	SB-03-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	6 J
SB-03	SB03A-041205-01	04/12/05	0-1	0.1	0.004	0.019	0.044	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.021	0.01 J	--	--	--
SB-03	SB03A-041205-03	04/12/05	2-3	0.1	0.021	0.022	0.055	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.022	0.01 J	--	--	--
SB-03	SB03B-041205-01	04/12/05	0-1	0.1	0.02 U	0.02 U	0.02 U	0.2 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.01 U	0.01 U	--	--	--
SB-03	SB03B-041205-03	04/12/05	2-3	0.0	0.0005 U	0.0005 U	0.002 J	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-04	SB-04-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	170	380
SB-04	SB-04-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	64	150
SB-05	DUP-02-020805	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	970	3600
SB-05	SB-05-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	11	17
SB-05	SB-05-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	7900	21000
SB-05	SB-05-020805-15	02/08/05	14-15	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	31	79
SB-06	SB-06-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	5 J	6 J
SB-06	SB-06-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	8 J	4 J
SB-07	SB-07-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	7 J
SB-07	SB-07-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	5 J
SB-08	SB-08-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	11	16
SB-08	SB-08-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	7 J	5 J
SB-09	SB-09-020805-01	02/08/05	0-1	0.0	0.0005 U	0.032	0.0031	0.005 U	0.011	0.015	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.029	0.005 U	--	--	--
SB-09	SB-09-020805-03	02/08/05	2-3	1.3	0.39	0.49	0.42	0.045	0.0039	0.0036	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-09	SB-09-020805-05	02/08/05	4-5	0.0	0.0005 U	0.0005 U	0.0005 J	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	--	--	0.37 U	12	28
SB-09	DUP-01-020805	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	10
SB-09	SB-09-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	14
SB-09	SB09A-041205-03	04/12/05	2-3	0.5	0.29	0.13	0.084	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.018	--	--	--
SB-09	SB09A-041205-05	04/12/05	4-5	0.0	0.001 J	0.0071	0.0032	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.008 J	--	--	--
SB-09	SB09B-041205-03	04/12/05	2-3	0.0	0.002 J	0.0062	0.0031	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-09	SB09B-041205-05	04/12/05	4-5	1.9	0.42	0.32	1.2	0.026	0.005 U	0.005 U	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-09	SB09C-041205-03	04/12/05	2-3	10.1	2	1.1	7	0.005 U	0.005 U	0.005 U	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-09	SB09C-041205-05	04/12/05	4-5	0.4	0.18	0.057	0.18	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U	--	--	--
SB-10	SB-10-020805-01	02/08/05	0-1	0.0	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-10	SB-10-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	6 J	6 J
SB-10	SB-10-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	5 J	7 J
SB-11	SB-11-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	550	1500
SB-11	SB-11-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	4 J
SB-12	SB-12-020805-01	02/08/05	0-1	0.0	0.0005 U	0.001 J	0.0005 U	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.029	0.009 J	--	--	--
SB-12	SB-12-020805-03	02/08/05	2-3	0.0	0.0044	0.039	0.0034	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-12	SB-12-020805-05	02/08/05	4-5	0.0	0.01	0.0055	0.002 J	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0008 J	--	--	0.37 U	30	36
SB-12	SB-12-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	9 J	4 J
SB-13	SB-13-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	140	310
SB-13	SB-13-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	40	96
SB-14	SB-14-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	120	280
SB-14	DUP-03-020805	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	34	46
SB-14	SB-14-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	190	420
SB-15	SB-15-020805-05	02/08/05	4-5	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	7 J	4 J
SB-15	SB-15-020805-10	02/08/05	9-10	-	--	--	--	--	--	--	--	--	--	--	--	--	--	0.37 U	6 J	4 J
SB-16	SB-16-032305-01	03/23/05	0-1	0.0	0.002 J	0.03	0.014	0.01	0.0005 U	0.0016	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.024	0.01 J	--	--	--
SB-16	SB-16-032305-03	03/23/05	2-3	0.1	0.027	0.025	0.016	0.005 U	0.0005 U	0.0005 U	0.0005 U	-	-	-	-	0.018	0.018	--	--	--

Table 2-1. Summary of Detected Pesticides/PCBs and TPH in Soil Samples
 ECI Property, 20846 Normandie Avenue, Torrance, CA

SOIL INVESTIGATION FOR HISTORICAL STORMWATER PATHWAY - SOUTH
 MONTROSE CHEMICAL SUPERFUND SITE, LOS ANGELES COUNTY, CALIFORNIA

Boring ID	Sample Number	Sample Date	Depth (feet)	Total DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Chlordane	cis-Chlordane	gamma-Chlordane	Dieldrin	Alpha-BHC	Beta-BHC	Delta-BHC	Gamma-BHC	Aroclor 1254	Aroclor 1260	TPH-Gas	TPH-Diesel	TPH-Oil
				mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
SB-28	SB28A-041305-03	04/13/05	2-3	0.2	0.033	0.04	0.08	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.026	--	--	--
SB-28	SB28A-041305-05	04/13/05	4-5	0.0	0.0009 J	0.009	0.002 J	0.005 U	0.0005 U	0.0012	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--
SB-28	DUP04-041305-00	04/13/05	2-3	1.1	0.062	0.22	0.78	0.04 J	0.0082	0.0072	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.021	--	--	--
SB-28	SB28B-041305-03	04/13/05	2-3	0.2	0.012	0.028	0.15	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.01 J	--	--	--
SB-28	SB28B-041305-05	04/13/05	4-5	0.6	0.067	0.2	0.33	0.04 J	0.004 J	0.0079	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.016	--	--	--
SB-28	SB28B-041305-07	04/13/05	6-7	0.4	0.1	0.22	0.066	0.005 U	0.0005 U	0.0005 U	0.0044	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.022	--	--	--
SB-28	SB28B-041305-10	04/13/05	9-10	0.6	0.051	0.48	0.037	0.005 U	0.0005 U	0.0005 U	0.011	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.022	0.028	--	--	--
SB-28	DUP02-041305-00	04/13/05	2-3	0.1	0.023	0.037	0.079	0.025 U	0.0025 U	0.0064	0.009 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.021	--	--	--
SB-28	SB28C-041305-03	04/13/05	2-3	0.7	0.043	0.21	0.43	0.04 J	0.004 J	0.0051	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.018	--	--	--
SB-28	SB28C-041305-05	04/13/05	4-5	2.9	0.84	0.95	1.1	0.05 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.005 U	0.022	--	--	--
SB-28	SB28C-041305-07	04/13/05	6-7	0.9	0.4	0.34	0.13	0.12 U	0.012 U	0.012 U	0.012 U	0.012 U	0.012 U	0.012 U	0.012 U	0.005 U	0.01 J	--	--	--
SB-28	SB28C-041305-10	04/13/05	9-10	2.5	1.1	0.86	0.5	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-29	SB-29-041305-03	04/13/05	2-3	0.3	0.038	0.057	0.16	0.03 J	0.0058	0.004 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.024	--	--	--
SB-29	SB-29-041305-07	04/13/05	6-7	0.3	0.093	0.07	0.11	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.008 J	--	--	--
SB-29	SB-29-041305-10	04/13/05	9-10	0.3	0.15	0.1	0.063	0.025 U	0.0025 U	0.0051	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.006 J	--	--	--
SB-30	SB-30-041305-03	04/13/05	2-3	0.0	0.0006 J	0.0008 J	0.002 J	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-30	DUP01-041305-00	04/13/05	4-5	0.1	0.024	0.049	0.014	0.044	0.004 J	0.0081	0.006 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.01 J	--	--	--
SB-30	SB-30-041305-07	04/13/05	4-5	0.1	0.035	0.019	0.009 J	0.025 U	0.0025 U	0.003 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.01 J	--	--	--
SB-30	SB-30-041305-10	04/13/05	9-10	0.1	0.13	0.011	0.0025 U	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U	--	--	--
SB-31	SB-31-041305-03	04/13/05	2-3	3.6	0.14	0.71	2.7	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U	--	--	--
SB-31	SB-31-041305-05	04/13/05	4-5	0.4	0.059	0.12	0.19	0.025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.01 J	--	--	--
SB-31	SB-31-041305-07	04/13/05	6-7	0.0	0.0005 U	0.004	0.0073	0.006 J	0.0011	0.0013	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.007 J	--	--	--
SB-32	SB-32-041205-03	04/12/05	2-3	1.2	0.27	0.38	0.54	0.1 U	0.01 J	0.022	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	--	--	--
SB-32	SB-32-041205-07	04/12/05	6-7	2.1	0.25	0.45 E	1.4 E	0.025 U	0.0025 U	0.0025 U	0.01	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U	--	--	--
SB-32	SB-32-041205-10	04/12/05	9-10	9.1	1.3	1.2	6.6	0.025 U	0.003 J	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.0025 U	0.005 U	0.005 U	--	--	--
SB-33	SB-33-041205-03	04/12/05	2-3	0.1	0.004	0.042	0.019	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-33	SB-33-041205-07	04/12/05	6-7	0.0	0.0062	0.0078	0.012	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 J	--	--	--
SB-34	SB-34-041205-03	04/12/05	2-3	0.0	0.0035	0.0041	0.022	0.005 U	0.0007 J	0.0025	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.01 J	--	--	--
SB-34	SB-34-041205-07	04/12/05	6-7	0.0	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--
SB-35	SB-35-041205-03	04/12/05	2-3	12.6	0.62	2.3	9.7	1.2 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.005 U	0.005 U	--	--	--
SB-35	SB-35-041205-07	04/12/05	6-7	7.9	5.8	0.71	1.4	0.25 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	0.025 U	22	1.1	--	--	--
SB-35	SB-35-041205-10	04/12/05	9-10	0.3	0.170	0.037	0.057	0.005 U	0.001 U	0.001 U	0.001 U	0.0005 U	0.0005 U	0.0005 U	0.0005 U	0.005 U	0.005 U	--	--	--

Notes:
 mg/kg = milligrams per kilogram
 Only results with total DDT > 1.0 mg/kg were tabulated
 NA = not available
 Only 4,4' isomers were analyzed for DDE, DDT and DDD.
 -- = not analyzed
 Data Source: ECI, 2005.
 Hard copy of data not included in CD received from EPA.
 J = Concentration is estimated because it falls between the method detection limit and the laboratory reporting limit.
 U = Concentration is non-detect at the laboratory reporting limit.
 E = Concentration exceeds the upper level of the calibration range.
 TPH = Total petroleum hydrocarbons
 BHC = benzene hexachloride
 DDE = 4,4'-dichlorodiphenyldichloroethene
 DDT = 4,4'-dichlorodiphenyltrichloroethane
 DDD = 4,4'-dichlorodiphenyldichloroethane

**Table 2-2
Metals In Soil Samples
ECI Property
20846 Normandie, Torrance, CA**

Boring ID Sample ID Depth (ft) Sample Date	reporting limit	SB-03	SB-09	SB-10	SB-12	SB-16	SB-27	EPA PRGs		CA specific EPA PRGs		DTSC Soil Screening \	
		SB-03-020805-01 0 to 1 2/8/2005	SB-09-020805-01 0 to 1 2/8/2005	SB-10-020805-01 0 to 1 2/8/2005	SB-12-020805-01 0 to 1 2/8/2005	SB-16-032305-01 0 to 1 3/23/2005	SB-27-032305-01 0 to 1 3/23/2005	Industrial	Residential	Industrial	Residential	Industrial	Residential
Antimony	1.2	ND	ND	ND	ND	ND	ND	410	31	NA	NA	380	30
Arsenic	0.48	ND	ND	ND	ND	1.3	ND	1.6	0.39	0.25	0.062	0.24	0.07
Barium		120	63	57	190	140	190	67000	5400	NA	NA	63000	5200
Beryllium	0.45	ND	ND	ND	ND	ND	ND	1900	150	NA	NA	1700	150
Cadmium	0.52	ND	ND	ND	ND	ND	ND	450	37	NA	NA	7.5	1.7
Chromium		18	9.4	14	18	19	24	100000	100000	NA	NA	NA	NA
Cobalt		6.9	4.7	8.8	17	8.1	12	1900	900	NA	NA	3200	660
Copper		22	8.3	25	13	19	22	41000	3100	NA	NA	38000	3000
Lead		23	8.7	3.7	6.5	11	6.3	800	400	NA	NA	3500	150
Mercury	0.12	0.12	0.08 J	0.02 J	0.04 J	0.03 J	0.01 J	310	23	NA	NA	180	18
Molybdenum	0.46	1.6	1.8	1.1	0.9 J	ND	ND	5100	390	NA	NA	4800	380
Nickel		13	11	9.3	13	14	17	NA	NA	NA	NA	16000	1600
Selenium	0.82	ND	ND	ND	ND	1.5	1.9	5100	390	NA	NA	4800	380
Silver	0.45	ND	ND	ND	ND	ND	ND	5100	390	NA	NA	4800	380
Thallium	0.92	ND	ND	ND	ND	ND	ND	67	5.2	NA	NA	63	5
Vanadium		31	22	27	58	34	51	100	78	NA	NA	6700	530
Zinc		69	26	29	34	51	61	100000	23000	NA	NA	100000	23000

Notes

All concentrations are in mg/kg

ND = Not detected

Bold values indicate exceeds the PRG

**Table 2-3
VOCs in Soil Samples
ECI Property
20846 Normandie, CA**

Boring ID		SB-01	SB-02	SB-03	SB-04	SB-05	SB-06	SB-07	SB-08	SB-09	SB-10
Sample ID		SB-01-020805-10	SB-02-020805-10	SB-03-020805-10	SB-04-020805-10	SB-05-020805-10	SB-06-020805-10	SB-07-020805-10	SB-08-020805-10	DUP-01-020805	SB-10-020805-10
Depth (ft)	Detection	9 to 10	9 to 10	9 to 10							
Sample Date	Limit	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005
Ethylbenzene	1.8	ND	82	ND	ND	ND	ND	ND	ND	ND	ND
Methylbenzene	2.1	ND	5 J	ND	ND	ND	ND	ND	ND	ND	ND
O-Xylene	2.1	ND	97	ND	ND	ND	ND	ND	ND	ND	ND
P/M -Xylene	4.9	ND	160	ND	ND	ND	ND	ND	ND	ND	ND
Xylenes		NR	NR	NR							
Tetrachloroethene	1.7	ND	ND	ND							

Note
All concentrations are in mg/kg
Only detected VOCs are shown
ND = Not detected
NR = Not reported
Bold values indicate exceeds the PRG

**Table 2-3
VOCs in Soil Samples
ECI Property
20846 Normandie, CA**

Boring ID		SB-11	SB-11	SB-12	SB-13	SB-14	SB-15	EPA Region 9 PRGs		DTSC Soil Screening Values	
Sample ID	Detection	SB-11-020805-10	SB-11-020805-15	SB-12-020805-10	SB-13-020805-10	SB-14-020805-10	SB-15-020805-10	Industrial	Residential	Industrial	Residential
Depth (ft)		9 to 10	14 to 15	9 to 10	9 to 10	9 to 10	9 to 10				
Sample Date	Limit	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005	2/8/2005				
Ethylbenzene	1.8	ND	ND	ND	ND	ND	ND	400	400	NA	NA
Methylbenzene	2.1	ND	ND	ND	ND	ND	ND	520	520	NA	NA
O-Xylene	2.1	ND	ND	ND	ND	ND	ND	NA	NA	NA	NA
P/M -Xylene	4.9	ND	ND	ND	ND	ND	ND	NA	NA	NA	NA
Xylenes		NR	NR	NR	NR	NR	NR	420	270	NA	NA
Tetrachloroethene	1.7	5.5	ND	ND	ND	ND	ND	1.3	0.48	NA	NA

Note
All concentrations are in mg/kg
Only detected VOCs are shown
ND = Not detected
NR = Not reported
Bold values indicate exceeds the PRG

TABLE 2-4a
Data Needs and Uses – Soil Investigation

Parameters/ Compounds	Data Use	Data Users	Rationale	Regulatory Limits/Action Level				
				California DTSC Soil Screening Numbers (mg/kg)		EPA Region 9 Preliminary Remediation Goals (mg/kg)		Lowest Limit (mg/kg)
				Residential	Industrial	Residential Soil	Industrial Soil	
Soil Investigation Parameters								
TCL Pesticides/PCBs (A subset of the TCL list is shown below)	Nature and Extent Regulatory Comparison Risk Assessment Fate and Transport	Hydrogeologists Regulatory Specialists Risk Assessors						
Aroclor 1016			Other Aroclors detected on ECI Property	0.089	0.3	0.22	0.74	0.089
Aroclor 1248			Other Aroclors detected on ECI Property					
Aroclor 1254			Detected on- property					
Aroclor 1260			Detected on- property					
Aroclor 1262			Other Aroclors detected on ECI Property					
Aroclor 1268			Other Aroclors detected on ECI Property					
BHC, alpha-			Montrose-related, Detected on- property	NA	NA	0.09	0.36	0.09

TABLE 2-4a
Data Needs and Uses – Soil Investigation

Parameters/ Compounds	Data Use	Data Users	Rationale	Regulatory Limits/Action Level				
				California DTSC Soil Screening Numbers (mg/kg)		EPA Region 9 Preliminary Remediation Goals (mg/kg)		Lowest Limit (mg/kg)
				Residential	Industrial	Residential Soil	Industrial Soil	
BHC, beta-			Montrose-related, Detected on- property	NA	NA	0.32	1.3	0.32
BHC, gamma-			Montrose-related, Detected on- property	NA	NA	0.44	1.7	0.44
BHC, delta-			Montrose-related, Detected on- property	NA	NA	NA	NA	NA
Camphechlor			Detected on- property	0.46	1.8	0.44	1.6	0.44
Chlordane			Detected on- property	0.43	1.7	1.6	6.5	0.43
Chlordane, Cis-			Detected on- property	NA	NA	NA	NA	NA
Chlordane, Gamma-			Detected on- property	NA	NA	NA	NA	NA
DDT			Montrose-related, Detected on- property	1.6	6.3	1.7	7.0	1.6
DDE			Montrose-related, Detected on- property	1.6	6.3	1.7	7.0	1.6

TABLE 2-4a
Data Needs and Uses – Soil Investigation

Parameters/ Compounds	Data Use	Data Users	Rationale	Regulatory Limits/Action Level				
				California DTSC Soil Screening Numbers (mg/kg)		EPA Region 9 Preliminary Remediation Goals (mg/kg)		Lowest Limit (mg/kg)
				Residential	Industrial	Residential Soil	Industrial Soil	
DDD			Montrose-related, Detected on- property	2.3	9.0	2.4	10	2.3
Dieldrin			Detected on- property	0.035	0.13	0.03	0.11	0.03
Endrin				21	230	18	180	18
Endrin Aldehyde			Detected on- property	NA	NA	NA	NA	NA
Endrin Ketone			Detected on- property	NA	NA	NA	NA	NA
Ethylbenzene			Detected on- property	NA	NA	400	400	400
Heptachlor			Detected on- property	0.13	0.52	0.11	0.38	0.11
Heptachlor Epoxide			Detected on- property	NA	NA	0.053	0.19	0.053
Geotechnical Parameters for Treatment Options	To evaluate treatment feasibility	Treatment Technologists						
Atterberg Limits								N/A
Gradations w/ Hydrometer								N/A

TABLE 2-4a
Data Needs and Uses – Soil Investigation

Parameters/ Compounds	Data Use	Data Users	Rationale	Regulatory Limits/Action Level				Lowest Limit (mg/kg)
				California DTSC Soil Screening Numbers (mg/kg)		EPA Region 9 Preliminary Remediation Goals (mg/kg)		
				Residential	Industrial	Residential Soil	Industrial Soil	
Moisture content								N/A
Density								N/A
Direct shear								N/A

Notes:

mg/kg – milligrams per kilogram

NA – not available

N/A – not applicable

DTSC – Department of Toxic Substances Control

TCL – Target compound list, EPA CLP list.

Rationale – Organic compounds detected in ECI soil samples (ECI, 2005)

EPA Region 9 Preliminary Remediation Goals (mg/kg), October 2004

California Human Health Screening Levels (mg/kg), January 2005

TABLE 2-4b
Data Needs and Uses – Investigation-Derived Waste

Parameters ⁽¹⁾ /Compounds	Data Use	Data Users	Regulatory Limits/Action Level			Lowest Limit ⁽⁵⁾
			EPA Toxicity Characteristic TCLP ⁽²⁾ (mg/L)	California Toxicity Characteristic –TTL ⁽³⁾ (mg/kg)	California Toxicity Characteristic –STLC ⁽⁴⁾ (mg/L)	
Investigation-Derived Waste Soils						
Inorganics:	Waste Disposal Decisions	Field Team				
Antimony				500	15	
Arsenic			5.0	500	5.0	
Asbestos				1.0 ⁽⁴⁾		
Barium			100	10,000	100	
Beryllium				75	0.75	
Cadmium			1.0	100	1.0	
Chromium (total)			5.0	2,500	5	
Chromium (VI)				500	5	
Cobalt				8,000	80	
Copper				2,500	25	
Fluoride salts				18,000	180	
Lead			5.0	350	5.0	
Mercury			0.2	20	0.2	
Molybdenum				3,500	350	

TABLE 2-4b
Data Needs and Uses – Investigation-Derived Waste

Parameters ⁽¹⁾ /Compounds	Data Use	Data Users	Regulatory Limits/Action Level			Lowest Limit ⁽⁵⁾
			EPA Toxicity Characteristic TCLP ⁽²⁾ (mg/L)	California Toxicity Characteristic –TTLc ⁽³⁾ (mg/kg)	California Toxicity Characteristic –STLC ⁽⁴⁾ (mg/L)	
Nickel				2,000	20	
Selenium			1.0	100	1.0	
Silver			5.0	500	5	
Thallium				700	7.0	
Vanadium				2,400	24	
Zinc				5,000	250	
Total Petroleum Hydrocarbons: ⁽⁸⁾	Waste Disposal Decisions	Field Team				
Gasoline Fraction				1,000		
Diesel Fraction				10,000		
Volatile Organic Compounds:	Waste Disposal Decisions	Field Team				
Benzene			0.5			
Carbon tetrachloride			0.5			
Chlorobenzene			100			
Chloroform			6.0			
1,4-Dichlorobenzene			7.5			
1,2-Dichloroethane			0.5			
1,1-Dichloroethylene			0.7			

TABLE 2-4b
Data Needs and Uses – Investigation-Derived Waste

Parameters ⁽¹⁾ /Compounds	Data Use	Data Users	Regulatory Limits/Action Level			Lowest Limit ⁽⁵⁾
			EPA Toxicity Characteristic TCLP ⁽²⁾ (mg/L)	California Toxicity Characteristic –TTLc ⁽³⁾ (mg/kg)	California Toxicity Characteristic –STLC ⁽⁴⁾ (mg/L)	
Methyl ethyl ketone (2-Butanone)			200			
Tetrachloroethylene (PCE)			0.7			
Trichloroethylene (TCE)			0.5	2,040	204	
Vinyl chloride			0.2			
Pesticides:	Waste Disposal Decisions	Field Team				
Aldrin				1.4	0.14	
Chlordane			0.03	2.5	0.25	
DDT, DDE, DDD				1.0	0.1	
DDT						0.087 ⁽⁷⁾
Dieldrin				8.0	0.8	
Endrin			0.02	0.2	0.02	
Heptachlor (and its epoxide)			0.008	4.7	0.47	
Lindane			0.4	4.0	0.4	
Methoxychlor			10.0	100	10	
Toxaphene			0.5	5	0.5	
Polychlorinated biphenyls (PCBs):	Waste Disposal Decisions	Field Team				
PCBs				50	5.0	

TABLE 2-4b
Data Needs and Uses – Investigation-Derived Waste

Parameters ⁽¹⁾ /Compounds	Data Use	Data Users	Regulatory Limits/Action Level			Lowest Limit ⁽⁵⁾
			EPA Toxicity Characteristic TCLP ⁽²⁾ (mg/L)	California Toxicity Characteristic –TTL ⁽³⁾ (mg/kg)	California Toxicity Characteristic –STLC ⁽⁴⁾ (mg/L)	
Investigation-Derived Wastewater						
TCL Volatiles	Waste Disposal Decisions	Field Team	(9)	(9)*	(9)	(9)
TCL Pesticides/PCBs						
TAL Inorganics						

Notes:

mg/kg – milligrams per kilogram

mg/L – milligrams per liter

µg/L – micrograms per liter

TCLP – toxicity characteristic leaching procedure

TTL – total threshold limit concentration

STLC – soluble threshold limit concentration

(1) The analyte list for waste disposal may be modified depending on specific disposal facility requirements. The list is expected to be a subset of this list.

(2) EPA hazardous waste criteria: For determination of whether a solid waste (e.g., soil cuttings) may exhibit the characteristic of toxicity, to arrive at a soil concentration expressed in mg/kg, apply rule-of-thumb of 20 x TCLP standard (milligrams per liter [mg/L]) listed in this table. Maximum Concentration of Contaminants for the Toxicity Characteristic, 40 Code of Federal Regulations (CFR) Section 261.24, Toxicity Characteristic. The standards listed in this table are generally compared against concentrations obtained using the TCLP.

(3) California hazardous waste criteria: 22 California Code of Regulations (CCR) Section 66261.24, TTL Values for Inorganic Persistent and Bioaccumulative Toxic Substances.

(4) California hazardous waste criteria: 22 CCR Section 66261.24, STLC Values for Organic Persistent and Bioaccumulative Toxic Substances.

(5) Since the waste analysis includes liquids and solid-phase measurements, selecting the lowest limit does not apply. The selected methodology in Table 2-5 will meet the required detection limits.

(6) TTL for asbestos as percentage.

(7) Resource Conservation and Recovery Act (RCRA) universal treatment standard (UTS) for DDT, which is 0.087 mg/kg in soil (40 CFR 268.48 and 268.49)

(8) See Table 2-1 in the Leaking Underground Fuel Tank Field Manual (RWQCB, 1996).

(9) The same criteria as for soils, the TCLP and STLC criteria apply.

TABLE 2-5
Measurement Performance Criteria

Parameter/Compound	Method	Lowest Project Criteria (µg/L)	Reporting Limit/Target Detection Limit ⁽¹⁾ (µg/L)	Analytical Accuracy (% Recovery)	Analytical Precision (Relative % Deviation)	Overall Completeness (%)
Soil Investigation Parameters						
TCL Pesticides/PCBs:						
alpha-BHC	CLP ⁽¹⁾	0.09	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
beta-BHC	CLP ⁽¹⁾	0.32	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
delta-BHC	CLP ⁽¹⁾	0.44	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
gamma-BHC (Lindane)	CLP ⁽¹⁾	N/A	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Heptachlor	CLP ⁽¹⁾	0.11	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aldrin	CLP ⁽¹⁾		1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Heptachlor epoxide	CLP ⁽¹⁾	0.053	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Endosulfan I	CLP ⁽¹⁾		1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Dieldrin	CLP ⁽¹⁾	0.03	3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
4,4'-DDE	CLP ⁽¹⁾		3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Endrin	CLP ⁽¹⁾	18	3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Endosulfan II	CLP ⁽¹⁾		3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
4,4'-DDD	CLP ⁽¹⁾		3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Endosulfan sulfate	CLP ⁽¹⁾		3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
4,4'-DDT	CLP ⁽¹⁾		3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Methoxychlor	CLP ⁽¹⁾		17	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Endrin ketone	CLP ⁽¹⁾	N/A	3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Endrin aldehyde	CLP ⁽¹⁾	N/A	3.3	CLP ⁽¹⁾	CLP ⁽¹⁾	90%

TABLE 2-5
Measurement Performance Criteria

Parameter/Compound	Method	Lowest Project Criteria (µg/L)	Reporting Limit/Target Detection Limit ⁽¹⁾ (µg/L)	Analytical Accuracy (% Recovery)	Analytical Precision (Relative % Deviation)	Overall Completeness (%)
alpha-Chlordane	CLP ⁽¹⁾		1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
gamma-Chlordane	CLP ⁽¹⁾	N/A	1.7	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Toxaphene	CLP ⁽¹⁾		170	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1016	CLP ⁽¹⁾	0.089	33	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1221	CLP ⁽¹⁾	0.089	67	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1232	CLP ⁽¹⁾	0.089	33	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1242	CLP ⁽¹⁾	0.089	33	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1248	CLP ⁽¹⁾	0.089	33	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1254	CLP ⁽¹⁾	0.089	33	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Aroclor-1260	CLP ⁽¹⁾	0.089	33	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
Geotechnical Parameters for Treatment Options						
Atterberg limits	ASTM D-4318	N/A	N/A	N/A	N/A	90%
Gradations w/ hydrometer	ASTM D-422	N/A	N/A	N/A	N/A	90%
Moisture content /density	ASTM D-2937	N/A	N/A	N/A	N/A	90%
Investigation-Derived Waste Soils						
TCLP VOC:	EPA 1311 ⁽²⁾ /CLP plus CA Title 22 ⁽²⁾ /CLP	(2)	CLP ⁽¹⁾	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
TCLP Pesticides/PCBs	EPA 1311 ⁽²⁾ /CLP plus CA Title 22 ⁽²⁾ /CLP	(2)	CLP ⁽¹⁾	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
TCLP Metals:	EPA 1311 ⁽²⁾ /CLP plus CA Title 22 ⁽²⁾ /CLP	(2)	CLP ⁽¹⁾	CLP ⁽¹⁾	CLP ⁽¹⁾	90%

TABLE 2-5
Measurement Performance Criteria

Parameter/Compound	Method	Lowest Project Criteria (µg/L)	Reporting Limit/Target Detection Limit ⁽¹⁾ (µg/L)	Analytical Accuracy (% Recovery)	Analytical Precision (Relative % Deviation)	Overall Completeness (%)
TPH (Gasoline)	EPA 8015	(2)		75-125	±25	90%
TPH (Diesel)	EPA 8015	(2)		75-125	±25	90%

Investigation-Derived Wastewater

TCL Volatiles:	CLP ⁽¹⁾		CLP ⁽¹⁾	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
TCL Pesticides/PCBs	CLP ⁽¹⁾		CLP ⁽¹⁾	CLP ⁽¹⁾	CLP ⁽¹⁾	90%
TAL Inorganics:	CLP ⁽¹⁾		CLP ⁽¹⁾	CLP ⁽¹⁾	CLP ⁽¹⁾	90%

Notes:

(1) CLP – EPA CLP methodology and QA/QC as defined in most current EPA CLP Statement of Work (SOW). To meet the project criteria, low-level procedures will be requested through the CLP special services. The listed target limits are for routine procedures. For low-level procedures such as selective ion monitoring (SIM), limits are laboratory-specific and will be identified prior to analyses.

(2) Subsequent to EPA 1311 or California Title 22 leaching procedures, leachates will be analyzed per CLP methodology through the CLP special services.

N/A – not applicable.

Figures

Appendix A Data Quality Objectives

Appendix A

Data Quality Objectives

This document is an appendix to the Quality Assurance Project Plan (QAPP) for the *Soil Investigation, Historical Stormwater Pathway – South, Montrose Chemical Superfund Site, Los Angeles County, California* (United States Environmental Protection Agency [EPA], March 2006). This appendix details the data quality objectives (DQOs) for the investigation. The DQOs have been broadly described in Section 2.0 of the QAPP. This appendix documents the rationale and conclusions from completing the seven steps in the DQO process; the seven steps are as follows:

- Step 1 State the Problem
- Step 2 Identify the Decision
- Step 3 Identify Inputs to the Decision
- Step 4 Define the Boundaries for the Study
- Step 5 Develop a Decision Rule
- Step 6 Specify Tolerable Limits on Decision Errors
- Step 7 Optimize the Design

The DQO process derives from detailed evaluation and interpretation of available site information, which is included as a subsection in Step 6 below. The final product of the DQOs specifies four design objectives, as discussed in Step 7 and as summarized in Tables A-4 through A-7 at the end of this appendix.

Step 1. State the Problem

(1) Identify Members of the Planning Team

The members of the planning team are the EPA Work Assignment Manager (WAM) and Remedial Project Manager (RPM), CH2M HILL Site Manager (SM), CH2M HILL hydrogeologists, CH2M HILL risk assessor, CH2M HILL chemist, CH2M HILL statistician, and CH2M HILL Quality Assurance Officer (QAO).

(2) Identify the Primary Decisionmaker

EPA may conduct or oversee work conducted by others (e.g., representatives of the Montrose Chemical Corporation of California or the property owner), and has final approval authority for the work. Work conducted by others may be conducted under voluntary status and/or under an administrative order.

(3) Develop a Concise Description of the Problem

An industrial property located at 20846 Normandie Avenue, Torrance, California (near the intersection of Normandie Avenue and Torrance Boulevard) is occupied by Ecology Control Industries, Inc. (ECI). In the spring of 2005, the owner of 20846 Normandie Avenue was conducting soil sampling for due diligence activities related to the potential sale of the property. Several chemicals were detected in sampled soils from the southern and eastern edges of the

ECI Property including 4,4'-dichlorodiphenyltrichloroethane (DDT), 4,4'-dichlorodiphenyl-dichloroethene (DDE), and 4,4'-dichlorodiphenyldichloroethane (DDD), total petroleum hydrocarbons (TPH), polychlorinated biphenyls (PCBs), and benzene hexachloride (BHC). To address these findings, the owner removed (excavated) soil surrounding sampling locations having contaminated soil and stockpiled soil on-property.

Upon learning that soils containing elevated concentrations of pesticides had been excavated, EPA required excavated soil to be secured (e.g., covered with plastic sheeting and anchored down with sand bags) to prevent the possible release and/or human exposure (e.g., via fugitive dust) until soil could be properly disposed. Sampling and analysis information, including sample locations and analytical results, showed that total DDT (DDT, DDE, and DDD) had been detected in some subsurface soil samples from the eastern and southern areas of the ECI Property at concentrations exceeding the regional background range (1 to 3 milligrams per kilogram [mg/kg], and up to 10 mg/kg) (EPA, 2001c), with a maximum detected concentration of 325 mg/kg.

DDT and BHC were pesticides manufactured at the former Montrose Chemical Corporation (Montrose) Plant property, located at 20201 Normandie Avenue. Historically, releases from the Montrose Plant property entered the stormwater pathway starting at the Montrose Plant property, crossed Normandie Avenue, and continued down the west side of Kenwood Avenue in a ditch (known as the Kenwood Ditch). The path of the historical ditch continued south of Torrance Boulevard through the eastern portion of the ECI Property, across the adjacent Royal Boulevard Landfill property and beyond (see Figure 2-3 in Section 2.0 of the QAPP).

In the late 1960s, an underground storm drain was installed to replace the stormwater ditch; this is referred to as Project 685. Project 685 and its easement enter the northeastern edge of the ECI Property along Torrance Boulevard; parallel the eastern ECI Property boundary; and, at the southeastern property boundary, exit into the adjacent fenced grassy property known as the Royal Boulevard Landfill (see Figure 2-3 in Section 2.0 of the QAPP).

The segment of the historical stormwater drain north of Torrance Boulevard was addressed by EPA in 2001, at which time soils along Kenwood Avenue, from Del Amo Alley to Torrance Boulevard, were tested for DDT. Sampling and excavation on Kenwood Avenue revealed soil with elevated DDT concentrations at about 4.5 feet below the current street grade. Soils containing elevated DDT concentrations that presented a human health risk were removed and replaced with clean fill.

This QAPP and the companion Field Sampling Plan (FSP) are being prepared to plan additional sampling to characterize the segment of the historical stormwater pathway within the ECI Property and the back yards of residential lots adjacent to (directly east of) the ECI Property located on Raymond Avenue and West 209th Street, south of Torrance Boulevard.

The extent of the historical stormwater pathway in this area was determined by evaluating historical aerial photographs from 1935 to 1965, identifying the extent of ponded water and potential wetland and riparian vegetation along the path of the historical drainage (EPA, 2005a). The result of this evaluation – i.e., the extent of the historical stormwater pathway in this area – is shown in Figure 2-4 of the QAPP; for areas within the ECI Property, it is indicated by green cross-hatching in Figure 2-6 of the QAPP, and Figures A-2 through A-5 of Appendix A.

As part of the Montrose Chemical Superfund Site, the Study Area requires additional sampling to characterize the nature and extent of Montrose-related contamination, as required by the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA). EPA does not believe that the sampling already performed at the ECI Property on behalf of the property owner adequately meets this objective. Additional surface soil and subsurface soil samples from the Study Area portion of the historical stormwater pathway need to be collected and analyzed for Montrose-related contamination to provide a more detailed characterization of the nature and extent in this portion of the Historical Stormwater Pathway – South.

Sampling the ECI Property and the back yards of the adjacent residential lots is intended to provide information on the extent of Montrose-related chemicals in soil, including pesticides (e.g., DDT/DDE/DDD, and BHC) and PCBs. EPA will oversee the soil sampling; and sample analyses will be performed by an EPA-approved laboratory. Sampling may be conducted by EPA or others and/or their representatives. EPA will determine if additional sampling or excavation is needed within the ECI Property and/or the adjacent residential properties. After validation of the chemical analyses is complete, sampling results will be made available to the owners of the sampled properties, and summarized in reports.

Problem Statement(s):

Based on the above, there is the need to:

- (a) Characterize the nature and extent of contamination in the historical stormwater pathway by sampling soil from 20846 Normandie Avenue and the back yards of adjacent residential properties, which may have been impacted by releases from the Montrose Chemical Corporation property at 20201 Normandie Avenue.
- (b) Support a human health risk assessment (HHRA).
- (c) Support a removal action, if necessary.
- (d) Support a feasibility study (FS), if necessary.
- (e) Support characterization of investigation-derived waste (IDW).

(4) Specify Available Resources and Relevant Deadlines for the Study

The soil sampling effort at the ECI Property and adjacent residential properties is expected to begin during the spring/summer of 2006.

Step 2. Identify the Decision

(1) Identify the Principal Study Question

- (a) Characterize nature and extent of soil contamination: Are soils within (or impacted by) the historical stormwater pathway at the ECI Property and adjacent residential properties contaminated by Montrose-related chemicals at levels of concern, and if so, what are the horizontal and vertical extents of that soil contamination?
- (b) Support an HHRA: What are the human health risks due to Montrose-related chemicals in soils within (or impacted by) the historical stormwater pathway at the ECI Property and adjacent residential properties, either individually or in combination?

- (c) Support a removal action, if necessary: What is the extent of Montrose-related soil contamination requiring a removal action, if any?
- (d) Support an FS: What are the alternatives for remediation of Montrose-related contamination in soil within (or impacted by) the historical stormwater pathway at the ECI Property and adjacent residential properties, if needed?
- (e) Support characterization of IDW: Do IDW soil concentrations meet the waste acceptance criteria for disposal at an offsite treatment, storage, or disposal facility (TSDF) for either nonhazardous waste or hazardous waste? Do IDW water constituent concentrations meet the acceptance criteria for disposal at a hazardous or nonhazardous offsite TSDF?

(2) Define Alternate Actions that Could Result from Resolution of the Principal Study Question

- (a) Characterize nature and extent of soil contamination.
 - i) No further characterization of soil at the ECI Property or residential properties would be necessary to define the extent of Montrose-related soil contamination, and data are adequate to carry out an HHRA.
 - ii) Additional characterization is necessary to define the extent of Montrose-related contamination, or to support the completion of a risk assessment or removal action.
- (b) Support an HHRA.
 - i) Propose no action based on calculated human health risks for soil within (or impacted by) the historical stormwater pathway at the ECI Property and adjacent residential properties individually and in combination.
 - ii) Propose action (removal or remediation) based on calculated human health risks for soil within (or impacted by) the historical stormwater pathway at the ECI Property and/or adjacent residential properties.
- (c) Support a removal action, if necessary.
 - i) No removal action is necessary.
 - ii) Conduct a removal action in one or more areas, as needed based on calculated risks and soil criteria.
- (d) Support an FS.
 - i) Do not conduct an FS to evaluate remedial needs and alternatives.
 - ii) Conduct an FS to evaluate remedial needs and alternatives, if current (or post-removal) human health risks exceed risk (or other soil) criteria.

- (e) Support characterization of IDW.
 - i) IDW soil and liquid can be disposed of at an offsite TSDF for nonhazardous substances.
 - ii) IDW soil and liquid must be disposed of at an offsite TSDF as either hazardous soil or liquid (Resource Conservation and Recovery Act [RCRA] waste and/or Comprehensive Environmental Response, Compensation, and Liability Act of 1980 [CERCLA] waste).

(3) Combine the Principal Study Question and the Alternative Actions into a Decision Statement

- (a) Characterize nature and extent of soil contamination: If data from soil sampling indicate the presence and extent of Montrose-related chemicals at the ECI Property and adjacent residential properties at concentrations exceeding EPA Region 9 Preliminary Remediation Goals (PRGs), California Environmental Protection Agency (Cal-EPA) Department of Toxic Substances Control (DTSC) soil screening numbers, and background DDT concentrations (EPA, 2001c), then these compounds will be identified as compounds of concern and additional sampling for the specific phase/area may be required.
- (b) Support an HHRA: If the calculated human health risks are acceptable and require no action for the ECI Property or residential properties, then a decision of No Further Action (NFA) will be proposed. If, however, the risks indicate that an action is required for the ECI Property or a residential property, then EPA will determine if a removal or remedial action is appropriate for the specific area/phase.
- (c) Support a removal action, if necessary: If the reported concentrations and calculated risks indicate unacceptable and/or short-term risks, a removal action will be warranted. If, however, concentrations and calculated risks do not indicate unacceptable or short-term risks, then evaluations will proceed for other alternatives as described below.
- (d) Support an FS: If current (or post-removal action) concentrations indicate unacceptable long-term human health risks at the ECI Property or any of the residential properties, then an FS will be conducted. If, however, current (or post-removal action) concentrations indicate long-term human health risks are at acceptable levels for the ECI Property and the residential properties, then an NFA determination can be proposed.
- (e) Support characterization of IDW: If data indicate IDW soil is nonhazardous, then it will be disposed at an offsite nonhazardous TSDF. If, however, data indicate that the IDW soil is hazardous, then it will be disposed at a TSDF as hazardous waste. If IDW water meets the TSDF acceptance criteria of nonhazardous waste, then it will be disposed at a TSDF as nonhazardous waste. If however, the IDW water exceeds the criteria of hazardous waste, then it will be disposed at the TSDF as hazardous waste.

(4) Organize Multiple Decisions

Based on the answer to the principal study questions, decisions about additional sampling and analysis or laboratory corrective action will be made by the planning team.

- (a) Characterize nature and extent of soil contamination: The assessment of the nature and extent of soil contamination may indicate that the extent of soil contamination within or impacted by the historical stormwater pathway is greater than originally anticipated, thus triggering the need for additional soil sampling at the ECI Property or in any of the adjacent residential properties.
- (b) Support an HHRA: The HHRA may indicate that health risks due to Montrose-related soil contamination require that additional data will be needed to further refine or support the conclusions of the HHRA.
- (c) Support a removal action, if necessary: If soil sampling results or the HHRA indicate that a removal action is needed, then additional soil sampling and chemical and geotechnical analyses may be needed to refine the extent of remedial action that is needed.
- (d) Support an FS: Additional soil chemical analyses and geotechnical analyses may be needed to fully support an FS in order to develop and evaluate the remedial alternatives according to CERCLA FS guidance (EPA, 1988).
- (e) Support characterization of IDW: If IDW exceeds hazardous waste criteria, and the TSDF would not be able to accept the waste because of land disposal restrictions (LDRs), then evaluations of appropriate treatment/disposal options will be completed; this evaluation may follow the above FS evaluation.

Step 3. Identify Inputs to the Decision

The purpose of this step is to identify the information and measurements needed to support the decision statement.

(1) Identify the Information that will be Required to Resolve the Decision Statement

Chemicals of concern for proposed sampling are listed in Section 2.0 of the QAPP in Tables 2-4a and 2-4b. Detected chemicals include organochlorine pesticides (DDT, DDE, DDD, chlordane, cis-chlordane, gamma-chlordane, dieldrin, alpha-BHC, beta-BHC, delta-BHC, gamma-BHC), PCBs (Aroclor 1254, Aroclor 1260), volatile organic compounds (VOCs), and total petroleum hydrocarbons (TPH) as gasoline (TPH-g), as diesel (TPH-d), and TPH-oil. However, evaluation of past data indicates that only pesticides/PCBs are of concern for this evaluation of soils within (or impacted by) the historical stormwater pathway.

- (a) Characterize the nature and extent of soil contamination: To resolve the decision statement, soil concentration data will be needed for pesticides and PCBs (as summarized in Table 2-4a, Data Needs and Uses). Soil concentrations will be evaluated against applicable regulatory criteria (EPA Region 9 PRGs for the residential and industrial scenarios, Cal-EPA DTSC soil screening values, and regional background soil concentrations of total DDT (EPA, 2001c). The PRGs and DTSC soil screening values are provided in Table 2-4a.

- (b) Support an HHRA: To resolve the decision statement, soil concentration data for pesticides and PCBs will be needed to determine whether human health risks are acceptable, pose a long-term risk, or represent a short-term human health risk.
- (c) Support a removal action, if necessary: To resolve the decision statement, soil concentration data for pesticides and PCBs will be needed to assess whether a short-term human health risk is present. If the reported concentrations and/or associated screening risk assessment indicate possible exposures resulting in the potential for short-term toxicity, or other unacceptable risks, then EPA will consider the need for a removal action. If a removal action is indicated, geotechnical parameters may be needed to evaluate soil removal options (e.g., excavation and disposal, capping). Geotechnical parameters for excavation could include Atterberg Limits (American Society for Testing and Materials [ASTM] D-4318), Gradations w/ Hydrometer (ASTM D-422), Moisture Content/Density (ASTM D-2937), and Direct Shear (ASTM D-3080).
- (d) Support an FS: To resolve the decision statement, data are needed to characterize the depth, lateral extent, and volume of soil within (or impacted by) the historical stormwater pathway from the Montrose plant that exceeds criteria including the EPA PRGs, DTSC soil screening values, and calculated human health risks. Chemical analyses of pesticides/PCBs are needed with limits of detection to meet state and federal applicable or relevant and appropriate requirements (ARARs). In addition, the following geotechnical soil tests are needed to evaluate the remedial alternatives for excavation and disposal: Atterberg Limits (ASTM D-4318), Gradations w/Hydrometer (ASTM D-422), Moisture Content/Density (ASTM D-2937), and Direct Shear (ASTM D-3080). The geotechnical parameters listed are needed primarily for evaluation of alternatives involving excavation.
- (e) Support characterization of IDW: To resolve the decision statement for IDW soil, soil concentrations will be needed for pesticides/PCBs, VOCs, California Code of Regulations (CCR) Title 22 metals, TPH-g, and TPH-d. For IDW water, analytical results of pesticides/PCBs, VOCs, metals, and TPH, will be necessary to meet the waste acceptance criteria for offsite TSDFs. The analytical parameter list for IDW samples may be modified depending on specific waste facility requirements.

(2) Determine the Sources for Each Item of Information Identified

The following sources of the needed data will be supplied through the sampling and analysis of both field soil and any clean fill to be used with a removal action.

- (a) Characterize nature and extent of soil contamination: Boring logs, visual inspection of existing open excavations at ECI, surveyed coordinates and elevations of soil borings, and the analysis of surface and subsurface soil samples from new borings.
- (b) Support an HHRA: Laboratory analysis results of soil samples; and, to evaluate exposure points and pathways, visual inspection of ECI Property open soil excavations, adjacent residential properties, and where exposed, the underground Project 685 drain.

- (c) Support a removal action, if necessary: Laboratory analysis results of soil samples, geotechnical analysis of soil samples to support evaluation of alternatives, and visual inspection of the ECI Property and adjacent residential properties.
- (d) Support an FS: Laboratory analysis of soil samples, geotechnical analyses of soil samples to support evaluation of alternatives, and visual inspection of ECI Property and adjacent residential properties.
- (e) Support characterization of IDW: Laboratory analysis results of IDW soil and water.

(3) Identify the Information that is Needed to Establish the Action Level

Tables 2-4a and 2-4b in Section 2.0 of the QAPP list the appropriate criteria and/or regulatory limits for constituents in soils.

- (a) Characterize nature and extent of soil contamination: Action levels for soils will utilize the EPA Region 9 PRGs (industrial PRGs for current use and residential PRGs for current or potential future residential use) and the DTSC soil and screening level concentrations (Tables 2-4a and 2-4b).
- (b) Support an HHRA: Information needed includes the EPA and Cal-EPA toxicity criteria (e.g., cancer slope factors and reference doses) for estimating cancer risks and hazards during the HHRA process. Additionally, action levels for the HHRA will utilize EPA's acceptable risk ranges for noncarcinogens (Hazard Index of 1) and for carcinogens (excess lifetime cancer risks of 10^{-6} to 10^{-4}), and Region 9 PRGs (industrial and residential) to support a baseline or screening level HHRA (Tables 2-4a and 2-4b).
- (c) Support a removal action, if necessary: For removal action consideration, action levels of 10^{-5} human health excess lifetime residential cancer risk or a chronic Hazard Index of 10 will be used.
- (d) Support an FS: An FS will be initiated based on the findings of those efforts listed in (a), (b), and (c) above.
- (e) Support characterization of IDW:

The following summarizes the regulatory criteria with regard to waste disposal. The final list of parameters will depend on the input from the disposal facilities, and thus, the full list below will be pared down before the start of the work.

For IDW Soil:

(1) The IDW soil waste must be disposed based on characteristic and listed waste criteria. IDW soils generated during sampling may be considered listed hazardous wastes (e.g., U061 or U129), and thus would be regulated under state and federal hazardous waste laws (40 Code of Federal Regulations [CFR] 261.33 [a], [b], and [c]). EPA has previously determined that the DDT-contaminated soil excavated from the Montrose Superfund Site historical stormwater drainage pathway north of Torrance Boulevard (Kenwood Removal Action) was a federally listed hazardous waste pursuant to RCRA.

(2) DDT has been detected in soil within the historical stormwater pathway at the ECI Property at concentrations exceeding the RCRA universal treatment

standard (UTS), which is 0.087 mg/kg in soil (40 CFR 268.48 and 268.49). Comparison to the UTS standard is needed to determine whether the IDW soil would need to be disposed under the RCRA LDRs.

(3) EPA has determined that offsite disposal of the excavated soil must comply with the CERCLA Offsite Rule (42 United States Code [USC] Section 9621[d][3]), which governs the offsite transportation and disposal of hazardous waste. Because DDT and BHC present in soil at the ECI Property are believed to have been released from the Montrose Plant property, soil from the historically low-lying areas of the property must be disposed at a facility that meets the requirements of the Offsite Rule.

(4) Excavated soils may also be subject to regulation under (1) federal and California hazardous waste laws for RCRA characteristic waste, (2) 40 CFR Section 261.24, 22 (toxicity characteristic), and (3) CCR Section 66261.24 (characteristics of toxicity which include total threshold limit concentration [TTLC] and soluble threshold limit concentration [STLC] standards).

For IDW Water:

(1) Waste acceptance criteria for offsite TSD are required, including pesticides/PCBs, VOCs, TPH-g, TPH-d, and Title 22 metals.

(4) Confirm that Appropriate Measurement Methods Exist to Provide the Necessary Data

Standard EPA methods are available for the target analytes. The analytical methods are provided in Table 2-5 in Section 2.0 of the QAPP.

Step 4. Define the Boundaries for the Study

(1) Specify the Characteristics that Define the Population of Interest

For each of the four DQOs defined above as (a), (b), (c), and (d), the populations of interest include surficial and deeper soil collected from the extent of the historical stormwater pathway (including soil impacted by it) on the ECI Property and adjacent residential properties. The population of interest relevant to DQO (e) includes IDW soil containerized in drums, roll-off bins, and other storage containers; and IDW water containerized in drums and other storage tanks.

(2) Define the Spatial Boundary of the Decision Statement

(a) Define the geographical area to which the decision statement applies. For DQOs (a), (b), (c), and (d), the geographical boundaries are the areas that have been historically impacted by the stormwater pathway, such as within the ECI Property and adjacent residential properties, as shown in Figure 2-4 in Section 2.0 of the QAPP. The initial soil investigation is limited to the ECI Property and adjacent residential properties. If necessary, subsequent phases of work may address properties and/or parcels further along the historical stormwater pathway (e.g., Royal Boulevard Landfill and adjacent parcels). For DQO (e), the “geographical areas” are IDW soil generated during the drilling and sampling of soil borings, and

contained in drums or roll-off bins; and IDW water generated from decontamination of drilling and sampling equipment.

- (b) Divide the population into strata that have relatively homogeneous characteristics. Analysis of existing data, discussed in Step 6, establishes the homogeneous strata.

(3) Define the Temporal Boundary of the Decision Statement

- (a) Determine the timeframe to which the decision statement applies – The decisions will apply until removal or remedial actions are planned and started, or determined by EPA to not be necessary.
- (b) Determine when to collect data – The soil sampling results are not dependent on the time of year and can be taken any time. The investigations are expected to start in the spring/summer of 2006 and continue at the discretion of EPA.

(4) Define the Scale of Decisionmaking

The scale of decisionmaking will be the areas of the historical stormwater pathway plus any soil that is impacted by the historical stormwater pathway, initially within the ECI Property and adjacent residential properties and expanding to additional areas, if needed.

(5) Identify Practical Constraints on Data Collection

The sampling locations and schedule will depend on access and physical obstructions (e.g., trees or structures on residential properties). For example, drilling equipment will not be able to approach the edge of the open excavations at the ECI Property due to the potential for sidewall collapse, unless they are reinforced by shoring. Additionally, the capacity of sampling and analysis teams, as well as weather constraints, may limit the pace of work.

Step 5. Develop a Decision Rule

(1) Specify the Statistical Parameter that Characterizes the Population of Interest

- (a) Characterize nature and extent of soil contamination: Statistical parameters to be used in decisionmaking will include the mean, the upper 95 percent confidence on the mean, the upper 90th percentile, and individual maximum concentrations per analyte. Data subsets will include property-specific areas, individual depth layers within the sampling strata defined by the evaluation of historical data, and the conceptual site model of contaminant deposition through the drainage pathway.
- (b) Support an HHRA: A screening level or baseline risk assessment will follow EPA guidance.
- (c) Support a removal action, if necessary: Soil concentrations or the results of an HHRA will determine the need for a removal action based on short-term toxicity. Chemical and geotechnical data will be utilized based on professional judgment to perform preliminary evaluations of the need for a potential removal action.
- (d) Support an FS: Site chemical and geotechnical data will be utilized based on professional judgment to perform preliminary feasibility evaluations and to assess further data collection and technical evaluations.

- (e) Support characterization of IDW: Comparison to applicable criteria will be made on a point-by-point basis (e.g., IDW soil concentrations compared against background concentrations, STLC and TTLC values, and RCRA UTS values).

(2) Specify the Action Level for the Study

See Step 3, Item (3) for the action levels for each DQO. The action levels are also listed in Tables 2-4a and 2-4b in Section 2.0 of the QAPP.

(3) Develop a Decision Rule (an “if...then...” statement)

- (a) Characterize nature and extent of soil contamination: All available chemical information will be tabulated, plotted, and/or statistically evaluated as described in Step 5, Subsection 1(a) above to assess the nature and extent of contamination. If soil analyses indicate the presence of any Montrose-related chemicals at the ECI Property and/or adjacent residential properties that exceed the action levels defined in Step 3 (EPA Region 9 PRGs [residential or industrial], the California DTSC soil screening numbers, DDT background concentrations, or the RCRA UTC values), then it will be identified as a chemical of concern for future consideration. If results exceed the action levels for either the phase/area under consideration, or the full study area, then additional sampling and analysis may be required.
- (b) Support an HHRA: If the data from the samples are less than the action levels described in Tables 2-4a and 2-4b (EPA Region 9 PRGs or DTSC soil screening numbers), then additional sampling to fill gaps or no action for the specific area/phase may be decided. If the data results exceed the action levels, then additional sampling and/or analysis may be needed, such as a baseline risk assessment per EPA guidance.
- (c) Support a removal action, if necessary: If the reported soil concentrations and/or their associated risks indicate the potential for short-term toxicity, or other unacceptable risks, a removal action may be warranted. EPA had previously (June 2001c) determined that a removal action was necessary (Kenwood Removal Action) where residential exposure scenarios were both complete pathways and the 95 percent upper confidence limit (UCL) on the mean of the data for the pathway corresponded to a one-in-one-hundred thousand (10^{-5}) cancer risk (i.e., 17 parts per million [ppm] total DDT), or any single soil sample exceeded the chronic hazard index of 10 (i.e., 350 ppm total DDT).
- (d) Support an FS: If current (or post-removal action) concentrations indicate unacceptable long-term human health risks (e.g., greater than a 10^{-5} cancer risk) at either the ECI Property or the residences, then an FS will be conducted per EPA guidance (EPA, 1988). Preliminary considerations will be based on professional evaluation of the chemical and geotechnical data obtained in this sampling.
- (e) Support characterization of IDW:

Soil IDW:

The following is a comprehensive decision rule that takes into account all the regulations. Subsequent to input from the disposal facility, this decision rule may be modified.

- (1) If IDW soil contains DDT or other Montrose-related constituents, EPA may determine that it must be disposed as a federal RCRA listed hazardous waste.
- (2) If EPA determines that the soil is a federal RCRA-listed waste, IDW soil concentrations exceeding 10 times the corresponding federal RCRA UTS, which for DDT is 0.87 mg/kg, would require treatment to achieve concentrations below that value (10 times the UTS) prior to land disposal under the RCRA LDRs. (If the sampling results for soil IDW show that hazardous substances, including DDT and BHC, are not present at or above 10 times those RCRA UTS values, then EPA may determine that the soil no longer contains a RCRA-listed waste, with the result that the soil would no longer be considered a RCRA-listed waste.) If EPA makes the determination that the soil is not considered a federal RCRA-listed waste, then offsite treatment prior to land disposal at an appropriate offsite facility would not be required, and the soil could be transported as nonfederal RCRA waste to be land-disposed offsite at a non-RCRA facility without prior treatment.
- (3) If Montrose-related waste constituents are detected in soil or IDW waste, then soils could be considered a CERCLA hazardous waste and would have to be handled according to the Offsite Rule.
- (4) If the analytical data indicate that soil IDW exceeds toxicity characteristic leaching procedure (TCLP) criteria, then it would be considered a federal RCRA hazardous waste and would have to be disposed at a RCRA hazardous waste disposal facility. If the soil IDW exceeds the STLC or TTLC criteria, then it will be considered a California hazardous waste and California state treatment standards and disposal limitations would apply.

Step 6. Specify Tolerable Limits on Decision Errors

In accordance with EPA guidance, tolerable limits on decision errors, which are used to establish performance goals for the data collection design, are specified in this step. The following discussion is limited to problem statements (a) and (b) regarding the nature and extent of contamination and risk assessment. The other problems (treatment and waste disposal) will be addressed through a judgmental design, as detailed in the FSP.

For the nature and extent of contamination and risk assessment, performance specifications and design optimization (Step 7) have been developed based on review of historical data. Statistical evaluation of previously available analytical data focused on samples taken from 35 soil borings on the ECI Property between February and June 2005 by Haley & Aldrich, consultants to ECI, as previously discussed in Section 2.2.3.9 of the QAPP.

Results from those evaluations are presented in the following subsections. Following a description of available data (Step 6, Subsection 1) and data management steps taken prior to statistical evaluations (Step 6, Subsection 2), results are summarized including a description of statistical distributions of analytes quantified (Step 6, Subsection 3). Then relationships among pesticides and PCBs quantified in the individual samples are examined (Step 6, Subsection 4). The description closes with discussion of total DDT distribution (both lateral and vertical) across the ECI Property (Step 6, Subsection 5). Evaluation of the historical data was used to determine existing data gaps and, thus, focused the design of the Phase 1 and 2 soil boring sampling proposed in the QAPP. These detailed results have driven the performance

specifications on grid spacing and sample sizes for Phase 1 and Phase 2, as currently designed and described in Step 7. Phase 1 results may modify details of Phase 2, which would be finalized in a Phase 2 QAPP addendum.

(1) Summary/Interpretation of Available Soil Boring Data

Between February and June 2005, Haley & Aldrich installed 35 soil borings on the ECI Property, as shown in Figure 2-6 in the QAPP. Nearest-neighbor boring distances between borings were as close as 25.6 feet (ft) (7.8 meters [m]) for borings SB-04 and SB-35, and as far as 132.2 ft (40.3 m) for borings SB-23 and SB-24. Based on results from the original 35 locations, excavations were conducted at and around 6 of the 35 locations, with excavation wall grab samples collected at depths up to 15 feet below ground surface (bgs). Excavation wall sample locations (shown in Figure 2-7) were substantively closer, ranging from 0.5 ft (0.15 m) up to 3.2 ft (2.8 m) between samples. Seventeen analytes quantified in soil samples included the organochlorine pesticides, PCBs, and TPH. These 17 analytes were not consistently measured in all samples, resulting in an imbalance of the number of analytes reported from individual locations, which limited statistical evaluations. A limited number of samples (16) were analyzed for VOCs (Table 2-3); an additional 6 samples were analyzed for metals (Table 2-2). The VOCs were rarely detected and, with one exception (tetrachloroethene in boring SB-11 at 9 to 10 ft bgs), did not exceed either EPA Region 9 residential and industrial PRGs or DTSC soil screening values. One metal, arsenic, exceeded both the EPA Region 9 residential PRG and DTSC soil screening values, in a soil sample from boring SB-16 at a depth of 0 to 1 ft bgs.

(2) Data Description/ Data Management Procedures

- Analytical results were manually transcribed from hardcopy datasheets and entered into an Excel spreadsheet (Table 2-1 in the QAPP). Data were provided to EPA by Haley & Aldrich (ECI, 2005) in response to EPA's 104(e) Request for Information to ECI.
- Total DDT was calculated as the sum of the concentrations of the three primary DDT isomers: 4,4'-DDT, 4,4'-DDD, and 4,4'-DDE (identified as DDT44, DDD44, and DDE44 in the statistical analysis findings such as Table A-1 and Figure A-1). For total DDT sums with one or more detected constituent isomers, a qualifier designated "D" for detect was indicated. In the 16 cases where none of the isomers in the total DDT calculation were detected, the qualifier of "U" for nondetected was used.
- Maps from Haley & Aldrich were used (digitized) to identify coordinates of sample boring locations.
- Soil boring sample results indicated that samples exceeding a total DDT concentration of 10 mg/kg (the upper end of the regional background range of DDT (EPA, 2001c) were localized to the area of the historical stormwater pathway, including the Project 685 subsurface drainage channel (also referred to as the Kenwood Drain), which is identified in Figure 2-6 as the Los Angeles County Flood Control District (LACFCD) Easement. A data partition was added to distinguish borings located within the area of the historical stormwater pathway, and the remaining area of the ECI Property.

- A second data partition was used to define categorical depth intervals based on sample depths (in feet bgs), documented in the available data:

Interval	Depth of Samples (ft bgs)
0_SRF	0 to 1
1_SHALLOW	1 \ 2 \ 2-3 \ 3 \ 3.5
2_MID	4 \ 4-5 \ 5 \ 6 \ 6-7 \ 7
3_DEEP	9 \ 9-10 \ 10 \ 11 \ 12 \ 14-15 \ 15

- Analytical and location data were matched into a single file using sample identification fields.

(3) Analyte Statistical Distributions

Table A-1 lists the 14 pesticide/PCB and 3 TPH analytes that were quantified in samples collected by Haley & Aldrich, and summarizes those results with conventional summary statistics, including: total counts of analyzed samples (267 samples analyzed for total DDT), counts of samples with detectable quantities of the analytes and the frequency of detection ratio (FD); ranges of reported nondetect results and reported positively detected results; mean, median, standard deviation, and coefficient of variations (ratio of standard deviation divided by mean); and comparison to a total DDT criterion of 10 ppm, the upper range of regional background total DDT values), with the counts for each of reported detects and nondetects exceeding that criterion. Concentrations reported as nondetects were used at the reporting limit in all calculations.

In Table A-1, the summary statistics are sorted by analytical groups (pesticide/PCB and TPH), then by decreasing frequency of detection of the analytical groups in soil samples collected from the ECI Property by Haley & Aldrich.

The statistical evaluation results presented in Table A-1 show the following:

- Pesticides, including DDT isomers, PCBs, chlordane isomers, dieldrin, and BHC isomers were quantified in well over 200 samples, with locations balanced across the ECI Property, allowing these analytes and relationships among the analytes to be examined statistically.
- TPH components were limited to a subset of approximately 45 samples and, in most cases, samples analyzed for TPH components were not also quantified for pesticides. Because of this imbalance in characterization, any relationships between the occurrence of pesticides and TPH cannot be quantified.
- Total DDT and its constituents were the most commonly occurring of the chemicals in this analysis, detected in 91 to 94 percent of samples collected. The minimum detected concentrations coincide with the minimum nondetect limit of detection; however, the frequency of detected values and the ranges of detections, which are orders of magnitude greater than maximum nondetects, clearly indicate the prevalence and elevated levels of DDT isomers in the samples. The criterion used to evaluate total DDT (10 mg/kg) was exceeded in 34 of the 267 samples analyzed, a 12.7 percent frequency of exceedance.

- Four pesticides (the three chlordane isomers [chlordane, cis-chlordane and gamma-chlordane] and dieldrin) and two PCBs (Aroclors 1254 and 1260) were less commonly detected, with FDs ranging between 15 and 20 percent.
- The BHC isomers (alpha-, beta-, gamma- and delta-) were infrequently detected. FDs range from less than 1 percent for delta-BHC up to 5 percent for beta-BHC. Further, when positively detected, the concentration range of the detected levels falls within the interval of reported limits of detection for nondetect results, indicating that the differentiation of detect and nondetect is not strong.

(4) Relationships Among Analytes

Potential relationships among analytes quantified in the same samples were evaluated in two ways: (1) by the use of correlation matrices, and (2) by the use of total DDT exceedance of the 10 ppm criterion as a data partition.

- Correlation matrices - Correlation matrices were calculated and graphically displayed as paired variables. This is summarized briefly here and further described below (Step 6, Subsection 4.1). That evaluation was limited to the examination of potential relationships among the 10 analytes detected in 15 or more percent of the samples analyzed (i.e., BHC isomers and TPH fractions were excluded). Analytes were limited to this subset because correlation calculations are sensitive to variables having many values at the same level (such as the limit of detection for nondetect samples). BHC pesticides, which were detected in 5 percent or less of the samples and at very low concentrations, were, consequently, excluded from the correlations. The effect of including analytes with more than 15 percent, but less than 100 percent, of samples analyzed is discussed further in Step 6, Subsection 4.1, below. TPH measures were also excluded from the correlation matrices because the number of samples in which both sets of analytes (pesticides/PCBs and TPH) were quantified was limited to six samples (correlations are evaluated only for the subset of observations with all variables quantified).
- Exceedance of the 10 ppm criterion - The second approach taken to examine relationships among different pesticides (Step 6, Subsection 4.2) used total DDT exceedance of the criterion as a data partition. Summary statistics for each analyte were calculated both for the subset of samples with total DDT exceeding the criterion (10 ppm) and for the subset of samples with total DDT results falling below the criterion. Comparing summary statistics for the analytes is a less sophisticated analytical method, but could be applied to all analytes detected, including the suite of BHC pesticides, which were detected infrequently.

(4.1) Correlation Matrix/Scatter Plot Matrix Evaluation

Evaluation of the more commonly detected analytes relies on a multivariate correlation matrix, supplemented with a graphical display of the same paired variables. Figure A-1 displays the correlation matrix for the 10 analytes having FDs greater than 15 percent (total DDT, DDD/DDE/DDT, straight chlordane, cis-chlordane and gamma-chlordane, dieldrin, and Aroclor 1254 and 1260 [ARO1254 and ARO1260]), supplemented with a graphical display of the same paired variables. The correlation matrix posted below the scatter plot correlation matrix gives the probability of statistically significant

relationships between the variable pairs. Only the lower triangle of the square matrix is provided, because, for example, the correlation of DDT against DDD is identical to the correlation of DDD against DDT. The diagonal values in the triangle (zero values) correspond to the probability that the positive correlation of the variable to itself is due to chance alone. That probability is, by definition, zero because the relationship of a variable to itself is 1-to-1 and the probability of that occurring due to chance alone is zero. The probabilities in the matrix are adjusted, based on both the number of paired observations (per variable pair) as well as the number of combined variables being compared. By convention, a probability of 0.05 is taken to mean that the analyte pairs are significantly related. The correlation can be either positive or negative.

The matrices of scatter plots are simply the graphical displays of the same paired observations for each set of paired analytes. Overall results from the correlation and scatter plot matrices are described, as follows:

- Total DDT (labeled DDTAT in Figure A-1) and its subcomponents (DDT44/DDE44/DDD44 in Figure A-1) correlate significantly. This is shown in both the matrix (emphasized by highlighting in turquoise that portion of the probability matrix), where the probabilities of the six paired variables are 0.000, and in the graphical display of Figure A-1, which show comparatively linear bundles of paired points. There is some noise (graphically) between the components, but the relationships are strong between each component and the total DDT.
- Chlordane and its related analytes [cis-chlordane, gamma-chlordane] and dieldrin correlate positively (with 95 percent confidence). In the matrix, this has been highlighted in the green block (correlation of 0.000) and the corresponding graphical plot. However, the frequencies of detection and the overlap of detected and nondetected concentrations suggest that these correlations may reflect analytical artifacts (e.g., for two analytes in one sample, the nondetected limits of detection should vary together) rather than a true statistically significant relationship.
- PCBs correlate only between each other (Aroclor 1254 and 1260) as shown in the matrix by a single yellow-highlighted correlation probability and comparatively sparse ellipse with two outliers.

There are comparatively minor correlations between some pairs of total DDT and chlordane. However, these could be the result of the large number of nondetects in the other analytes included in the correlation calculations. Subsequent evaluations, limiting correlations to total DDT, chlordane, dieldrin and Aroclor 1254 indicate no relationships with DDT and a single relationship between dieldrin and chlordane, which is, in all likelihood, an artifact of co-varying analyte reporting limits.

(4.2) DDT Criterion Exceedance as Data Partition

Table A-2 examines the potential relationships in a different way, using the concentrations of total DDT as a data partition, and includes summary statistics for each analyte partitioned by whether the sample concentration of total DDT did or did not exceed the 10 mg/kg criterion, which was previously determined to be an upper limit

of total DDT background concentrations in the vicinity of the Montrose property (EPA, 2001c).

It is reasonable to assume that if an analyte co-varies with total DDT, summary statistics would differ for the subset of results coming from samples with high and low levels of total DDT. The 10 mg/kg criterion is a convenient cut-point against which to aggregate different samples. The conclusions of this analysis are provided below:

- DDT isomers exhibit substantive differences in summary statistics from subsets defined by the exceedance of the total DDT criterion. FDs and maximum, mean, and median concentrations are all higher in the subset corresponding to the exceedance of the total DDT criterion. The pattern is most marked in 4,4-DDT, less apparent in 4,4-DDD, and least obvious in 4,4-DDE.
- Chlordanes tend to exhibit a negative relationship to the total DDT subset of samples exceeding the criterion, in that FDs are consistently zero with the limited number of detections occurring in the subset of samples that did not exceed the total DDT criterion.
- BHCs are consistently not detected in the subset of samples that are below the DDT criterion. However, while BHC detections occur in samples with total DDT exceedances, the frequencies of detection are low. Similarly, maximum, mean, and median values are higher in the subset of samples with DDT exceedances, but the ranges of concentrations approach the reporting limits. These results suggest that there may be a weak relationship between exceedance of DDT and BHC concentrations, but that the BHC contamination levels are so attenuated that the signal is weak to indiscernible at the levels of detection reported by Haley & Aldrich.
- Similar to the chlordane, the two Aroclors have FDs higher in the subset of samples with no DDT exceedance. However, maximum and mean concentrations are slightly elevated in the same subsets. Dieldrin appears to exhibit little to no change with respect to the exceedance/nonexceedance of the total DDT criterion.
- Finally, TPH quantification in samples quantified for DDT are so rare (six samples total) that any relationships observed could not support definitive conclusions.

(5) Lateral and Vertical Differences in Total DDT Concentrations

The spatial distribution of total DDT was examined through graphical plots including plan view maps designating concentration ranges for the four depth categories defined, and boxplots comparing the distributions of observations across areas and depths.

(5.1) Plan View Maps

Figures A-2 through A-5 present total DDT concentrations in soil for the four depth ranges (0 to 1 ft bgs, 1 to 3.5 ft bgs, 4 to 7 ft bgs, and 9 to 15 ft bgs), respectively. These figures show the soil boring locations on the ECI Property as a solid dot. The relative size of the dots is proportional to the total DDT concentration at that location. Total DDT concentrations less than 10 mg/kg (the upper regional background level) are represented as a black dot, and concentrations exceeding 10 mg/kg are shown in red. At locations

with multiple samples within the depth category (e.g., areas of excavation and additional sampling within the excavations), the sizing has been based on the maximum total DDT concentration at that location. A review of Figures A-2 through A-5 show that samples exceeding 10 ppm total DDT occurred only along the Project 685 underground easement, and at subsurface depths.

Figure 2-7 in Section 2.0 of the QAPP shows the locations of the excavations and the excavation wall samples, as interpreted from information provided by Haley & Aldrich. Excavations 20 and 35 were joined into one open excavation by additional excavation activity after soil samples were collected. Table A-3 summarizes total DDT concentrations for the site overall, for areas without excavations, and for each of the six original excavations. As shown in Table A-3, the maximum total DDT concentrations (Dmax) exceeded 10 ppm in five of the six excavations. Excavation 3, which did not have elevated total DDT concentrations, was excavated to address elevated PCBs detected in soil samples. Excavations 5 and 20 had discrete subsurface soil samples exceeding 50 ppm total DDT, and the 95 percent UCL values were also greater than the 10 ppm total DDT criterion. The soil sample having the maximum total DDT concentration (325 ppm) was collected at 5 feet bgs from Excavation 5.

(5.2) Boxplots

Boxplots graphically display the relative distribution of different data subsets. Figure A-6 schematically depicts the components and initial interpretation of a generic boxplot. Figure A-7 includes two panels that compare total DDT concentrations measured to areas of the ECI Property, and to the different depths of samples collected from the ECI Property. These two boxplot panels, and the coding constraints and interpretation of the two boxplots are discussed below:

(5.2.1) ECI Areas Panel

The upper panel of Figure A-7 presents boxplots identified as “IN,” “OUT_N,” and “OUT_S”; these codes aggregate soil samples from borings located within the historical stormwater pathway area, outside that area on the northern portion of the ECI Property, and outside that area on the southern portion of the ECI Property, respectively. The broken line of the plot at 10 mg/kg indicates the upper end of the regional background range for total DDT.

- The two boxplots of samples from borings outside the historical stormwater pathway area have broad overlap with each other and fall well below the criterion, indicating that the areas outside the channel are similar and contain concentrations of total DDT at levels that do not exceed the range of regional background concentrations.
- The boxplot for soil boring samples from within the historical stormwater pathway area exhibits little overlap with those boxplots for areas outside the historic stormwater pathway area. Additionally, the upper quartile whisker (line extending from the box) extends beyond the 10 mg/kg criterion, meaning that soil borings within the historic stormwater drainage area account for all 34 values exceeding the 10 ppm criterion.

(5.2.2) Sample Depth Panel

Boxplots comparing sample depths, displayed in the lower panel of Figure A-7, were generated to see if there were clear depth intervals within which DDT criterion exceedances did not occur. Sample depth subsets in the plot have been coded according to the sample interval depth partition codes, described in Step 6, Subsection 2. Interpretation of the boxplot panel is not straightforward, because sampling depths were not consistent within borings.

The box plots show that surface samples do not exceed the criterion for total DDT and that a portion of subsurface samples from each of the depth intervals also do not exceed the total DDT criterion. However, all borings were not sampled at the same depths; therefore, interpretation of the box plots comparing depth class intervals is not so straightforward.

Available data are not sufficient to refine sampling depths for future sampling events. For future sampling, decisions as to the appropriate sample depths and depth intervals should rely on other criteria, such as risk-based scenarios for anticipated land use. Consistency in the vertical depths for sample collection in the proposed sampling will prevent similar uncertainties in interpretation, for evaluation of the vertical boundary of DDT contamination at the ECI Property and adjacent residential properties.

Step 7. Optimize the Design

The following applies to Problem Statements (a) and (b) (see Step 1) regarding the nature and extent of contamination and risk assessment. The other problem statements (treatment and waste disposal) will be addressed through judgmental design, as detailed in the FSP.

This section, Step 7, describes the considerations for design of the proposed soil sampling investigation to be conducted at the ECI Property and adjacent residential properties. Three elements are summarized below and described further in the following subsections.

- Step 7, Subsection 1 identifies data gaps to be filled in order to determine the extent to which any contamination from the historical stormwater pathway is present at the ECI Property and/or adjacent residential properties, and to determine if such contamination constitutes a risk to human health.
- Step 7, Subsection 2 specifies four design objectives that are based on the data gaps identified in Subsection 1, as well as discussions with EPA regarding project objectives. Subsection 2 identifies data required to address the problem statements and describes proposed sampling corresponding to each problem statement. Step 7, Subsection 3 describes performance specifications used to determine the number of samples and the grid spacings for each of the three areas where sampling has been proposed. DQOs 4 through 7 document the design objectives for the four DQO decision points, presenting decision inputs, study boundaries, decision rules, acceptable limits on decision errors, and the optimization steps taken to finalize the proposed sampling design for the four problem statements developed.

(1) Data Gaps

The following bullets identify gaps in currently available information that are needed to determine the nature and extent of contamination associated with the historical stormwater pathway, any removal or remediation potentially required for the ECI Property and/or adjacent residential properties, and any necessary restrictions that may be required on future uses. The proposed soil boring locations are presented in Figure A-8.

- Soil borings collected in the LACFCD easement and buffer of the historical stormwater drainage pathway on the ECI Property (identified as part of the Phase 1 area in Figure A-8) demonstrated the presence of total DDT in exceedance of the range of regional background concentrations, and several samples exceeded applicable criteria. Neither lateral extent nor vertical depths of soil contamination have yet been characterized sufficiently to determine the extent of contamination associated with the central area of the historical stormwater drainage pathway.
- Analysis of historical aerial photographs identifies the eastern boundary of the historical stormwater pathway as extending slightly eastward beyond the ECI Property boundary into what are currently residential properties. Sampling data are not available for these properties. Supplementary sampling in the backyards of these residential properties is needed because of the proximity to the total DDT found in the subsurface soil along the historical stormwater drainage pathway on the ECI Property, and to determine the eastward extent of the historical stormwater drainage pathway. The residential sampling locations are also part of Phase 1.
- Soil borings collected in the historical stormwater drainage pathway on the ECI Property west of the LACFCD easement and buffer (identified as part of the Phase 2 area in Figure A-8) demonstrated the presence of total DDT in exceedance of the range of regional background concentrations. Neither lateral extent nor vertical depths of soil contamination have yet been characterized sufficiently to determine the extent of contamination associated with this western area of the historical stormwater drainage pathway.
- Soil borings on the ECI Property outside the area of the historical stormwater pathway indicate that total DDT is not present in soil (at any location or depth) at concentrations exceeding 10 mg/kg, the upper end of the range of regional background concentrations for total DDT. However, spatial coverage along the boundary of the historical stormwater pathway requires supplementary sampling to provide sufficient confidence to define the western extent of the historical stormwater drainage pathway. The area just outside (west) the drainage pathway (identified as the western portion of the Phase 2 area in Figure A-8) will be sampled to define the extent of the historical drainage pathway and the DDT sources found in the drainage pathway area. The remaining area of the ECI Property, beyond the Phase 2 area, is not proposed for further characterization at this time. However, if results from the proposed sampling suggest that the conceptual site model is incorrect (e.g., the extent of DDT contamination and/or the historical stormwater pathway is beyond that identified in Figure A-8), potential supplemental sampling may be necessary.

- Finally, the relationships among the various contaminants, including the organochlorine pesticides and PCBs (e.g., trends in concentration, location, and frequency of detection) found within the historical stormwater pathway (the ECI Property and/or adjacent residential properties) can be used to support a determination of the source of the DDT and/or other contaminants detected. Sampling is intended to provide additional information regarding the extent to which the DDT, and other contaminants found in soil, may be attributable to the historical stormwater drainage pathway.

(2) Design Objectives

The data gaps listed in Step 7, Subsection 1 above, resulted in the development of four objectives. Three of these objectives are specific to further characterization of soil in the historical stormwater pathway for the distribution of organochlorine pesticides and PCBs. The fourth objective addresses the issue of the potential source of contaminants and will utilize data from the characterization sampling identified above.

Although the four data gaps are related and the proposed sampling cumulatively addresses all the information required to make conclusions as to nature and extent of contamination within the historical drainage pathway and the potential risks associated with that contamination, EPA has developed a phased approach to implement the sampling design. The phased approach is intended to optimize implementation of the proposed sampling design. Figure A-8 shows the proposed sampling phases and boring locations.

- Phase 1 consists of 96 soil borings and focuses on the area immediately adjacent to the LACFCD easement (36 borings) and the adjacent residential properties (60 borings). Phase 1 is intended to ensure that the areas of greatest concern are addressed in as timely a manner as possible.
- Phase 2 consists of an additional 22 soil borings within the historical stormwater drainage pathway on the ECI Property (west of the Phase 1 sampling area) and up to 25 soil borings beyond the pathway. Phase 2 sampling locations will allow for a better focus of the extent to which supplemental sampling is required, based on Phase 1 results. Figure A-8 shows the Phase 2 sampling locations, which represent an extension of the Phase 1 design and also include a preliminary design for the area west of the historical stormwater drainage pathway. The Phase 2 design may be refined based on Phase 1 results.

Each of these phases, and the corresponding areas to be sampled, are further described below.

(2.1) Residential Properties East of ECI Property within Historical Stormwater Pathway (Phase 1)

Design Objective:

To what extent does the soil contamination relate to the historical stormwater pathway and, therefore, extend laterally and vertically within the residential properties east of the ECI Property?

Proposed Sampling

Lateral Extent: The residential properties east of ECI Property have not been sampled to characterize the historical stormwater pathway and evaluate the presence of related contaminants. Based on an analysis of historical aerial photographs for this segment of the stormwater pathway, it is possible that the historical stormwater drainage pathway extended into what are now the residential properties, before they were brought to their current elevation and developed. Subsurface soil beneath the western portion of the residential lots may have been part of the banks of the historical stormwater drainage pathway, and thus could potentially have been impacted by DDT contamination. Therefore, Phase 1 of the proposed sampling will include back yards of the indicated seven contiguous residential properties that have a portion of the historical stormwater drainage on the property.

Both the characterization of nature and extent of potential contamination associated with the historical stormwater pathway, and the assessment of potential risk to human health associated with DDT contamination from the historical stormwater pathway (if any) require characterization of the greater area as well as each property individually. To conduct property-specific assessment of human health risks resulting from potential residential exposures, an increased density in sampling and a reduction in grid spacing are required. Therefore, proposed spacing is a square grid with nodes on 20-foot centers, resulting in 56 primary nodes throughout this residential portion of the historical stormwater pathway, with between 7 and 12 sample locations per residential property. The rationale for grid spacing is provided below in Step 7, Subsection 3.1 – Spatial Coverage.

This primary grid will be supplemented with four additional samples used to support evaluation of the sufficiency of the grid spacing in these residential properties. These four additional samples will be collocated around grid sampling nodes. Collocated samples will be collected from four of the properties to be sampled, at locations that fall within the historical stormwater drainage pathway, as shown in Figure A-8.

Should DDT concentrations fall below risk-based standards, no additional sampling in the residential properties will be necessary. If results indicate that DDT concentrations exceeding the criteria extend beyond the area proposed for sampling in residential properties, then additional areas of the seven residential properties may be sampled. No additional sampling within the residential properties has been identified at this time.

Vertical Extent: Sampling depths at the residential properties will extend from the surface to 14 ft bgs. A total depth of 14 feet is based on the estimated depth to the contact of the predevelopment drainage swale and the overlapping fill. Sample intervals have been established to include the following increments: 0 to 6 inches, 6 to 24 inches, 2 to 5 ft, 5 to 8 ft, 8 to 11 ft, and 11 to 14 ft, resulting in six sample depths per soil boring location.

Analytes Measured: All samples collected will be analyzed for pesticides/PCBs. Boring logs will be prepared from continuous cores collected in the field.

Design objectives: Objectives defining the problem decision points, inputs, boundaries, decision rules, acceptable limits on decisions, and optimization of sampling are presented in Table A-4 (Design Objective 1).

(2.2) ECI Property within Historical Stormwater Pathway (Both Phase 1 and Phase 2 Sampling)

Design Objective

To what extent does the soil contamination related to the historical stormwater pathway extend laterally and vertically within soils found on the ECI Property?

Proposed Sampling

Lateral Extent: Currently available sample results from the ECI Property are limited to those available from the sampling performed by Haley & Aldrich in 2005 (Figure 2-6, Tables 2-1 through 2-3 in Section 2.0 of the QAPP). Statistical evaluation of this data shows a comparatively higher frequency of total DDT values exceeding regional background levels in soil borings located within the portion of the ECI Property where the historical stormwater pathway occurred, as compared to those collected from borings located on the ECI Property outside (west of) the historical drainage pathway area. However, the existing soil sampling is not sufficient to characterize the full extent of the historical stormwater pathway within the ECI Property. The low density of existing samples may not have captured the extent of subsurface DDT-contaminated materials. Additionally, the inconsistent findings of DDT concentrations in soil samples indicate that denser supplementary sampling is necessary.

Sampling within the historical stormwater pathway on the ECI Property will be conducted in two phases.

- Phase 1 sampling within the ECI Property focuses on the area of the LACFCD easement with an approximate 15-foot buffer on each side, extending to the eastern ECI Property boundary. Phase 1 sampling includes the 30 soil borings.
- Phase 2 sampling includes the remaining area within the drainage pathway west of the Phase 1 sampling (22 borings).

The primary sampling grid for the ECI Property within the historical stormwater pathway is a square grid with nodes on 30-foot centers (Figure A-8), resulting in 30 nodes for Phase 1 and 22 nodes for Phase 2. The rationale for grid spacing is provided in Step 7, Section (3.1) – Spatial Coverage. The Phase 1 grid will be supplemented with six collocated samples, and the Phase 2 grid will be supplemented with four to seven collocated samples.

Vertical Extent: The LACFCD Project 685 underground storm drain “as-built” drawings show that the historical ditch was located at a depth corresponding to approximately 20 feet below current ground level. The conceptual model for the historical stormwater pathway assumes that the drainage was sloped downward toward the centerline of the historical stormwater pathway. Thus, the Phase 2 area of the historical stormwater pathway (corresponding to the outer edges of the drainage area) is presumed to have had a higher bottom elevation (less than 20 ft bgs).

Phase 1: Sample depths in the Phase 1 area on the ECI Property will extend from the surface to 20 ft bgs. Sample intervals will include the following increments: 0 to 6 inches, 6 to 24 inches, 2 to 5 ft, 5 to 8 ft, 8 to 11 ft, 11 to 14 ft, 14 to 17 ft, and 17 to 20 ft, for a total of eight depth intervals.

Phase 2: Sample depths for the Phase 2 area on the ECI Property will extend from the surface to 14 feet bgs. A total depth of 14 feet is based on the estimated depth to the contact of the predevelopment drainage swale and the overlapping fill. The Phase 2 samples within the historical stormwater pathway will be collected at intervals of 0 to 6 inches, 6 to 24 inches, 2 to 5 ft, 5 to 8 ft, 8 to 11 ft, and 11 to 14 ft.

Analytes Measured: All samples collected will be analyzed for pesticides/PCBs. Boring logs will be prepared from continuous cores collected in the field.

DQO Statements: Statements defining the problem decision points, inputs, boundaries, decision rules, acceptable limits on decisions, and optimization of sampling are presented in Table A-5 (Design Objective 2).

(2.3) ECI Property, West of the Historical Stormwater Pathway (Phase 2)

Design Objective

To what extent does soil contamination related to the historical stormwater pathway extend laterally and vertically within soils found on the western portion of the ECI Property (west of the historical stormwater pathway area)?

Proposed Sampling

Lateral Extent: As stated above, currently available sample results from the ECI Property, including west of the historical stormwater pathway, are limited to that available from the sampling performed by Haley & Aldrich in 2005 (Figure 2-6, Tables 2-1 through 2-3 in Section 2.0 of the QAPP). Statistical evaluation of this data for the ECI Property showed that none of the soil samples from the western portion of the ECI Property (west of the historical drainage pathway area) exceeded 10 ppm total DDT.

The proposed Phase 2 sampling area west of the historical stormwater drainage pathway is intended to confirm the conceptual model of the extent of the historical stormwater drainage pathway. The findings of the existing data for this area suggest that supplemental sampling that is less dense in this area (as compared to the area within the historical stormwater drainage) will provide sufficient characterization. Additionally, the conceptual model for the ECI Property and historical stormwater pathway assumes that this area was not part of the banks of the historical stormwater pathway. Therefore, the proposed sampling depth for these borings is approximately 10 feet bgs. This depth will cover potential exposures from typical construction activities.

The proposed spacing for the area west of the historical stormwater pathway is a square grid with approximately 25 nodes that lie on approximately 60-foot centers and generally extend between 1 and 2 node distances from the boundary of the historical stormwater pathway, as shown in Figure A-8. The rationale for grid spacing is provided in Step 7, Subsection 3.1 – Spatial Coverage. Sampling beyond the ECI drainage

pathway, with up to 25 soil borings, is scheduled as part of the Phase 2 activities and will be refined based on Phase 1 results. If, in contrast to currently available data, exceedances of the action levels occur in the supplemental samples outside the historical stormwater pathway, additional sampling could be required, based on the frequency and extent of exceedances observed. However, a reasonable number of samples systematically distributed throughout the area that result in no exceedances should suffice to demonstrate that the distribution of DDT contamination is related to the historical stormwater pathway and not a propertywide condition.

Vertical Extent: Sample depths will extend from ground surface to 11 ft bgs (slightly beyond 10 feet to capture a full sample interval). Sample depth intervals have been established to include the following increments: 0 to 6 inches, 6 to 24 inches, 2 to 5 ft, 5 to 8 ft, and 8 to 11 ft bgs, for a total of five depths at each soil boring location. The primary sampling grid for the ECI Property west of the historical stormwater pathway has 25 nodes for Phase 2 and will be supplemented with four to seven collocated samples.

Analytes Measured: All samples collected will be analyzed for organochlorine pesticides/PCBs. Boring logs will be prepared from continuous cores collected in the field.

DQO Statements: Statements defining the problem decision points, inputs, boundaries, decision rules, acceptable limits on decisions, and optimization of sampling are presented in Table A-6 (Design Objective 3).

(2.4) DDT Relationships to Other Organochlorine Pesticides

Design Objective

To what extent do soil contaminants related to the historical stormwater pathway correlate with total DDT and/or DDT isomers in the areas, and at the depths sampled both on and off the ECI Property inside and outside the historical stormwater pathway area?

Proposed Sampling

Statistical evaluations will rely on all results from the three areas identified in Step 7, Subsections 2.1 through 2.3, above.

Statements defining the problem decision points, inputs, boundaries, decision rules, acceptable limits on decisions, and optimization of sampling are presented in Table A-7 (Design Objective 4).

(3) Grid Spacing and Sample Size Performance Specifications

Performance specifications for the proposed sampling can be described both in terms of the spatial coverage corresponding to the grid spacing (Step 7, Subsection 3.1) and statistical coverage corresponding to the number of primary and secondary nodes (Step 7, Subsection 3.2) below.

(3.1) Spatial Coverage

Spatial coverage corresponding to the grid spacing is commonly described in terms of the largest radius of a homogeneously contaminated area that could be expected to be undetected (assuming no analytical false negative errors) and the confidence associated with that radius. Previously existing information demonstrates substantive heterogeneity in contaminant concentration levels in soils on the ECI site. This performance specification is a lower bound on the size of the area which may be left undetected with the specified grid spacing. Results from collocated samples supplementing results from the primary nodes will be used to estimate small-scale spatial heterogeneity (variability) on the scale less than half the grid spacing, both within the residential area soils, and within the historical stormwater pathway areas inside and outside the LAFCDC easement. Using methods developed by Gilbert (1987), the following table defines the radii of a homogeneously contaminated area that could be undetected at several levels of confidence: 85, 90, and 95 percent for the 20-, 30-, and 60-foot node spacings to be used in the various sampling areas (residential area and areas within and up to approximately 200 feet outside of the historical stormwater drainage pathway, respectively):

Primary Grid Spacing	Confidence	"Hot Spot" Radius [ft]		
		95%	90%	85%
20 ft		11.8	11.0	10.6
30 ft		17.7	16.5	15.9
60 ft		35.4	33.0	31.8

The primary grid spacings could potentially leave a radius of undetected homogeneously contaminated material in the residential area as large as between 10.6 and 11.8 feet, with between 85 and 95 percent confidence; in the historical stormwater drainage pathway area within the ECI Property as large as between approximately 16 and 18 feet, with the same levels of confidence; and on the ECI Property immediately outside the stormwater drainage as large as between approximately 32 and 35 feet.

The secondary collocated samples will provide additional coverage in a subset of locations within the residential and ECI drainage areas. Those results will be used to define the effectiveness of the primary grid spacing and to provide smaller-scale variability.

(3.2) Statistical Coverage

Statistical performance specifications corresponding to the counts of samples can be evaluated using the binomial distribution of confidence and coverage. For the proposed sampling, each of the areas to be sampled (at each of the sampling depths) will have sample counts from both the primary grid plus the secondary samples in the collocated borings. Sample location counts for the two Phase 1 areas are 36 and 60, including collocated borings, by DQO objective. Sample location counts for the primary grid of

the two Phase 2 areas are 22 and 25. The number of collocated borings for each of the two Phase 2 areas will range from 4 to 7.

The binomial distribution may be used to estimate the confidence and coverage associated with prespecified sample sizes. For example, sample sizes greater than 60 are sufficient to ensure covering the upper 95th percentile of a population with 95 percent confidence. Samples of 21 results are sufficient to provide an estimate of the upper 87th percentile of the distribution with 95 percent confidence. Counts of soil borings within the primary areas of concern (the historical stormwater drainage on and off the ECI Property) meet or exceed that performance specification.

Figures

Tables

TABLE A-1
 ECI Haley-Aldrich Sampling Summary Sitewide Results

	Count Detects	Count Results	Frequency of Detection	Minimum Nondetect	Maximum Nondetect	Minimum Detect	Maximum Detect	Arithmetic Mean	Median	Standard Deviation	Coefficient of Variation	RB Level = Risk-Based Action Level	Count Detects > RB Level	Percent Exceedance	Count Nondetects > RB Level
DDT total	251	267	0.94	0.0015	0.3	0.0015	325.1	5.42	0.664	21.55	3.98	10	34	12.7	--
DDT44	244	267	0.91	0.0005	0.1	0.0005	310	3.96	0.33	19.98	5.05				
DDD44	242	267	0.91	0.0005	0.1	0.0006	19	0.96	0.11	2.40	2.49				
DDE44	242	267	0.91	0.0005	0.1	0.0006	8.7	0.50	0.13	0.86	1.75				
ARO1260	138	267	0.52	0.005	0.05	0.005	1.8	0.03	0.008	0.15	4.58				
ARO1254	80	267	0.30	0.0005	0.05	0.006	22	0.13	0.005	1.40	10.65				
CDNEg	54	266	0.20	0.0005	0.2	0.0006	0.46	0.01	0.0025	0.04	2.72				
CDNEc	50	266	0.19	0.0005	0.2	0.0006	0.49	0.01	0.0025	0.04	2.87				
DLDRN	50	266	0.19	0.0005	0.2	0.0005	0.18	0.01	0.0025	0.03	2.15				
CDNE	39	266	0.15	0.005	2	0.006	3.5	0.12	0.0205	0.32	2.58				
BHCb	12	265	0.05	0.0005	0.2	0.0005	0.019	0.01	0.001	0.03	2.29				
BHCa	2	265	0.01	0.0005	0.2	0.001	0.0011	0.01	0.0005	0.03	2.34				
BHCg	2	265	0.01	0.0005	0.2	0.0008	0.0062	0.01	0.0005	0.03	2.33				
BHCd	1	265	0.00	0.0005	0.2	0.0041	0.0041	0.01	0.0005	0.03	2.33				
TPHg		46	0.00	0.37	1			0.53	0.37	0.28	0.52				
TPHo	45	45	1.00			4	21000	724.73	37	3148.12	4.34				
TPHd	45	46	0.98	10	10	3.8	7900	259.95	24	1163.85	4.48				

Notes:
 Data were provided to EPA by Haley & Aldrich (ECI, 2005)

DDT total	Total DDT Isomers	BHCb	beta-benzene hexachloride
DDT44	4,4'-dichlorodiphenyltrichloroethane	BHCa	alpha-benzene hexachloride
DDD44	4,4'-dichlorodiphenyldichloroethane	BHCg	gamma-benzene hexachloride
DDE44	4,4'-dichlorodiphenyldichloroethene	BHCd	delta-benzene hexachloride
ARO1260	Aroclor1260		
ARO1254	Aroclor1254	TPHg	Total Petroleum Hydrocarbons-gasoline
CDNEg	GammaChlordane	TPHo	Total Petroleum Hydrocarbons-oil
CDNEc	cis-Chlordane	TPHd	Total Petroleum Hydrocarbons-diesel
DLDRN	Dieldrin		
CDNE	Chlordane		

TABLE A-2
ECI Haley-Aldrich Sampling Summary Partitioned by DDT Exceedance

DDT Isomers	Analyte	Count Results	Frequency of Detection	Minimum Nondetect	Maximum Nondetect	Minimum Detect	Maximum Detect	Arithmetic Mean	Median
No DDT Exceedance DDT Exceedance	DDD44 DDD44	232 35	0.892241359 1	0.0005	0.1	0.0006 0.51	5.8 19	0.359064655 4.972285714	0.0605 3.3
No DDT Exceedance DDT Exceedance	DDE44 DDE44	232 35	0.892241359 1	0.0005	0.1	0.0006 0.58	2.5 8.7	0.273648707 1.965714286	0.079 1.7
No DDT Exceedance DDT Exceedance	DDT44 DDT44	232 35	0.900862098 1	0.0005	0.1	0.0005 1.9	7.8 310	1.00007069 23.56857143	0.155 9.5
No DDT Exceedance DDT Exceedance	DDTsig DDTsig	232 35	0.931034505 1	0.0015	0.3	0.0015 10	9.53 325.1	1.632784052 30.50657143	0.3715 17.7
CHLORDANES									
No DDT Exceedance DDT Exceedance	CDNE CDNE	231 35	0.16883117 0	0.005 0.005	1 2	0.006	3.5	0.101385714 0.276571429	0.016 0.025
No DDT Exceedance DDT Exceedance	CDNEc CDNEc	231 35	0.216450214 0	0.0005 0.0005	0.1 0.2	0.0006	0.49	0.010990476 0.027785714	0.0025 0.0025
No DDT Exceedance DDT Exceedance	CDNEg CDNEg	231 35	0.233766228 0	0.0005 0.0005	0.1 0.2	0.0006	0.46	0.011273593 0.027785714	0.0025 0.0025
BHCs									
No DDT Exceedance DDT Exceedance	BHCa BHCa	230 35	0 0.057142857	0.0005 0.0005	0.1 0.2			0.009643478 0.030545714	0.0005 0.0025
No DDT Exceedance DDT Exceedance	BHCb BHCb	230 35	0.013043478 0.257142872	0.0005 0.0005	0.1 0.2	0.0005 0.0009	0.0025 0.019	0.00966 0.032145714	0.0005 0.0025
No DDT Exceedance DDT Exceedance	BHCd BHCd	230 35	0 0.028571429	0.0005 0.0005	0.1 0.2	0.0041	0.0041	0.009643478 0.030617143	0.0005 0.0025
No DDT Exceedance DDT Exceedance	BHCg BHCg	230 35	0.004347826 0.028571429	0.0005 0.0005	0.1 0.2	0.0008 0.0062	0.0008 0.0062	0.009644783 0.03062	0.0005 0.0025
PCB-DIELDRIN									
No DDT Exceedance DDT Exceedance	ARO1254 ARO1254	228 35	0.315789461 0.171428576	0.0005 0.005	0.016 0.05	0.006 0.045	22 0.11	0.151046053 0.018542857	0.005 0.005
No DDT Exceedance DDT Exceedance	ARO1260 ARO1260	228 35	0.548245609 0.257142872	0.005 0.005	0.024 0.05	0.005 0.026	1.8 0.096	0.035364035 0.018742857	0.01 0.005
DIELDRIN									
No DDT Exceedance DDT Exceedance	DLDRN DLDRN	231 35	0.186147183 0.200000003	0.0005 0.0005	0.1 0.2	0.0005 0.003	0.18 0.015	0.009971861 0.02908	0.0025 0.0032
TPH									
No DDT Exceedance DDT Exceedance	TPH-d TPH-d	5 1	1 1			12 18	300 18	138.4 18	170 18
No DDT Exceedance DDT Exceedance	TPH-g TPH-g	5 1	0 0	0.37 1	1 1			0.622 1	0.37 1
No DDT Exceedance DDT Exceedance	TPH-oil TPH-oil	5 1	1 1			28 37	980 37	426.8 37	480 37

Notes:

DDT total	Total DDT Isomers	BHCg	gamma-benzene hexachloride
DDT44	4,4'-dichlorodiphenyltrichloroethane	BHCd	delta-benzene hexachloride
DDD44	4,4'-dichlorodiphenyldichloroethane		
DDE44	4,4'-dichlorodiphenyldichloroethene	TPHg	Total Petroleum Hydrocarbons-gasoline
ARO1260	Aroclor1260	TPHo	Total Petroleum Hydrocarbons-oil
ARO1254	Aroclor1254	TPHd	Total Petroleum Hydrocarbons-diesel
CDNEg	GammaChlordane		
CDNEc	cis-Chlordane		
DLDRN	Dieldrin		
CDNE	Chlordane		
BHCb	beta-benzene hexachloride		
BHCa	alpha-benzene hexachloride		

TABLE A-3
 ECI Total DDT Distribution: By Site / Excavation Unit

	Site	EXC03	EXC05	EXC09	EXC20	EXC32	EXC35	noEXC
Count Detects (D)	251	25	37	35	52	15	25	61
Count Results (N)	267	28	37	35	52	15	25	74
Frequency of Detection: D/N	0.94	0.89	1.00	1.00	1.00	1.00	1.00	0.82
Minimum Nondetect	0.0015	0.0015						0.0015
Maximum Nondetect	0.3	0.06						0.3
Minimum Detect	0.0015	0.0018	0.0204	0.0015	0.0031	0.147	0.21	0.002
Maximum Detect	325.1	0.314	325.1	18.1	52.7	10.25	12.62	5.68
Arithmetic Mean	5.4	0.0	18.7	3.4	8.7	3.1	4.4	0.39
Median	0.67	0.0	4.0	2.0	3.8	1.2	2.9	0.06
Standard Deviation	21.6	0.1	54.2	4.3	11.5	3.3	3.8	0.9
Coefficient Of Variation: SD/MEAN	4.0	1.5	2.9	1.3	1.3	1.1	0.9	2.4
95% UCL on Mean	8.0	0.1	36.7	4.8	11.9	4.9	6.0	0.6
Risk-Based Action Level	10	10	10	10	10	10	10	10
Count Detects > DDTcrit	34		10	4	17	1	2	
Percent Exceedance: 100*(D>CRIT/N)	12.7	0.0	27.0	11.4	32.7	6.7	8.0	0.0
Count Nondetects > DDTcrit		0	0	0	0	0	0	0

Notes:

D/N = number of detections/number of samples

SD/MEAN = standard deviation/arithmetic mean

UCL = upper confidence limit

DDTcrit = total DDT criterion of 10 ppm which is the upper range of regional background total DDT values

100*(D>CRIT/N) = Percentage of detections greater than DDT criterion:

(D = number of detections, CRIT = DDT criterion, N = number of results)

TABLE A-4
Design Objective 1

<p><i>To what extent does the historical stormwater pathway and related soil contamination extend laterally and vertically within residential soils east of ECI Property?</i></p>
<p>Decision Point</p> <p>Seven residential properties east of the ECI Property and within the historical stormwater drainage.</p>
<p>Inputs to the Decision</p> <p>Approximately 56 soil boring locations on 20-foot grid spacing, sampled at 6 depth intervals to 14 feet bgs; 4 collocated soil boring locations, within the residential properties and the extent of the historical stormwater pathway, sampled to 14 ft bgs.</p>
<p>Study Boundaries</p> <p>Approximately 0.82 acre of residential property (excluding building footprints) for the 7 residences directly east from the ECI Property, within the historical stormwater drainage.</p>
<p>Decision Rules</p> <p>Applicable criterion will be analyte-specific risk-based concentrations appropriate for residential land use. Sample results from all residences will be evaluated on depth-specific intervals. <i>If</i> the upper confidence on the mean concentration exceeds the criterion <i>or if</i> the upper 90th percentile of the observations exceeds the criterion <i>or if</i> the maximum concentration exceeds twice the criterion, the layer will be considered contaminated. The specific area(s) potentially requiring removal or remediation will depend on spatial distribution of observed elevated concentrations.</p> <p>Spatial overlap of multiple sample depths failing the comparisons to criterion will be used to identify three-dimensional areas requiring further characterization, removal and/or remediation.</p> <p>Sample sizes in the residential areas are sufficient to allow for both residential areawide or residential property-specific risk calculations. Residential-specific comparisons to criteria will be performed, as needed, depending on the location and/or localization of elevated contaminant concentrations.</p>
<p>Acceptable Limits on Decision Errors</p> <p>Statistical distributions of contaminant concentrations will be evaluated for normality, lognormality using the Shapiro-Wilks goodness-of-fit test to determine the appropriate method of calculating best estimates of central tendency, and upper bounds on contaminant concentrations.</p> <p>95 percent UCL on the mean and the 90th percentile of the observations ensure that neither the population overall nor the upper bound of the observed distribution exceeds applicable criteria. The 2X criterion maximum comparison ensures that no localized area exceeds twice the criterion.</p> <p>The option for supplemental residence-specific comparisons ensures that no individual property will be less well protected.</p>
<p>Optimized Sampling Design</p> <p>The 20-foot grid spacing ensures that contaminated areas of radius greater than 12 ft will be detected within the historical stormwater drainage pathway area of the residential properties, with 95 percent confidence. The localized cluster samples provide a test of grid sufficiency and could be used to establish a geostatistical model to predict contaminant concentrations at unsampled locations at a spatial coverage less than that collected during this proposed sampling effort.</p>

TABLE A-5
Design Objective 2

<p><i>To what extent does the historical stormwater pathway and related soil contamination extend laterally and vertically within soils found on the ECI Property within the historical stormwater pathway?</i></p>
<p>Decision Point</p> <p>ECI Property within historical stormwater drainage pathway.</p>
<p>Inputs to the Decision</p> <p>Soil sample results from ECI soil borings performed by Haley & Aldrich (2005) (Table 2-1 in the QAPP).</p> <p>Thirty (30) Phase 1 borings (sampled at 8 depth intervals to 20 ft bgs) and potentially up to 22 Phase 2 borings (sampled at 6 depth intervals up to 14 ft bgs) will be collected on 30-foot grid spacing.</p> <p>Six Phase 1 collocated soil borings sampled to 20 ft bgs. Phase 2 collocated borings will be assigned to between 4 and 7 of the primary grid nodes, contingent upon results from the Phase 1 investigation.</p>
<p>Study Boundaries</p> <p>Approximately 1.48 acres lying within the historical stormwater water drainage. Bounded by ECI Property boundaries on north, south, and east, and extent of historical stormwater drainage on west.</p>
<p>Decision Rules</p> <p>Applicable criterion will be analyte-specific risk-based concentrations appropriate for both residential and industrial land use.</p> <p>Sample results will be evaluated on depth-specific intervals. <i>If</i> the upper confidence on the mean concentration exceeds the criterion <i>or if</i> the upper 90th percentile of the observations exceeds the criterion <i>or if</i> the maximum concentration exceeds twice the criterion, the layer will be considered contaminated for land use specific to the criterion applied. The specific area potentially requiring removal or remediation will depend on spatial distribution of observed concentrations.</p> <p>Spatial overlap of multiple sample depths failing the comparison to criterion will be used to identify three-dimensional areas requiring further characterization, removal, and/or remediation in order to satisfy conditions applicable to current and proposed land use.</p>
<p>Acceptable Limits on Decision Errors</p> <p>Statistical distributions of contaminant concentrations will be evaluated for normality and lognormality using the Shapiro-Wilks goodness-of-fit test to determine the appropriate method of calculating best estimates of central tendency and upper bounds on contaminant concentrations.</p> <p>95 percent UCL on the mean and the 90th percentile of the observations ensure that neither the population overall nor the upper bound of the observed distribution exceeds applicable criteria. The 2X criterion maximum comparison ensures that no localized area exceeds twice the criterion.</p>
<p>Optimized Sampling Design</p> <p>The 30-foot grid spacing ensures that contaminated areas with radius greater than ~18 ft will be detected within the historical stormwater drainage pathway, with 95 percent confidence. Collocated samples provide a test of grid sufficiency.</p>

TABLE A-6
Design Objective 3

<p><i>To what extent does the historical stormwater pathway related soil contamination extend laterally and vertically within soils on the ECI Property west of, but proximal to, the historical stormwater pathway area?</i></p>
<p>Decision Point</p> <p>ECI Property, outside of the historical stormwater pathway, extended up to 2 nodes beyond historical stormwater drainage pathway boundary and/or areas of documented excavation, on a 60-foot grid (every other node of the 30-foot grid).</p>
<p>Inputs to the Decision</p> <p>Previously available ECI soil sampling results collected by Haley & Aldrich (2005) (Table 2-1 in the QAPP).</p> <p>Approximately 25 Phase 2 soil boring locations on an approximately 60-foot grid spacing, sampled at 5 depth intervals to 11 ft bgs. Phase 2 collocated borings will be assigned to between 4 and 7 of the primary grid nodes, contingent upon results from the Phase 1 investigation.</p>
<p>Study Boundaries</p> <p>ECI Property lying immediately outside (west of) the historical stormwater drainage pathway area. Bounded on north, south, and west by parcel boundaries, and historical stormwater drainage pathway area on east.</p>
<p>Decision Rules</p> <p>Applicable criterion will be analyte-specific, risk-based concentrations appropriate for both proposed future residential and current industrial land uses.</p> <p>Sample results will be evaluated on depth-specific intervals. <i>If</i> the UCL on the mean concentration exceeds the criterion <i>or if</i> the upper 90th percentile of the observations exceeds the criterion <i>or if</i> the maximum concentration exceeds twice the criterion, the layer will be considered contaminated for land use specific to the criterion applied. The specific area potentially requiring remediation will depend on spatial distribution of observed concentrations.</p> <p>Spatial overlap of multiple sample depths failing the comparison to criterion will be used to identify three-dimensional areas requiring further characterization, removal, and/or remediation in order to satisfy conditions applicable to proposed land use.</p>
<p>Acceptable Limits on Decision Errors</p> <p>Statistical distributions of contaminant concentrations will be evaluated for normality, lognormality using the Shapiro-Wilks goodness-of-fit test to determine the appropriate method of calculating best estimates of central tendency, and upper bounds on contaminant concentrations.</p> <p>95 percent UCL on the mean and the 90th percentile of the observations ensure that neither the population overall nor the upper bound of the observed distribution exceeds applicable criteria. The 2X criterion maximum comparison ensures that no localized area exceeds twice the criterion.</p>
<p>Optimized Sampling Design</p> <p>The approximately 60-foot grid spacing ensures that contaminated areas with a radius greater than approximately 35 ft will be detected with 95 percent confidence within the area.</p> <p>Design optimization has been ensured with substitution of Haley & Aldrich sample results where proposed grid samples overlap.</p> <p>Analysis optimization has been ensured with collection of samples in soil borings to 10 ft bgs.</p>

TABLE A-7
Design Objective 4

<p><i>To what extent do historical stormwater pathway related soil contaminants correlate with total DDT and/or DDT isomers in the areas and at the depths sampled both on and off the ECI Property inside and outside the historical stormwater drainage pathway area?</i></p>
<p>Decision Point</p> <p>Three areas of proposed sampling, independently.</p>
<p>Inputs to the Decision</p> <p>Previously available Haley & Aldrich sampling results.</p> <p>All samples from each of the proposed 151 to 157 soil borings at all vertical intervals.</p>
<p>Study Boundaries</p> <p>ECI Property within the historical stormwater drainage.</p> <p>ECI Property outside the historical stormwater drainage.</p> <p>Residential properties within the historical stormwater drainage.</p> <p>Vertical sampling intervals within each lateral area.</p>
<p>Decision Rules</p> <p>Relationships between detected analytes will be evaluated using correlation coefficients and graphical displays. Relationships, for which the probability of the correlation coefficient between two variable pairs (adjusting for the number of pair combinations) is less than 0.05, will be considered an indication that the analytes co-vary.</p> <p>Multiple lines of evidence, interpretation of statistically significant correlation structures in either different areas sampled and/or different depths will be used to map the possible source and transport of materials detected in either the ECI parcels or adjacent residences.</p>
<p>Acceptable Limits on Decision Errors</p> <p>Application of the 0.05 alpha error to paired variables, adjusted for the number of variable pairs evaluated, ensures that co-varying concentrations could be due to chance alone only 5 times out of 100.</p>
<p>Optimized Sampling Design</p> <p>Sample sizes identified and sample clusters will ensure that area and depth-specific evaluations will allow for interpretation of potentially different contaminant source(s) across the project area.</p>

Appendix B

Analytical Technical Specifications

**USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA**

**STANDARD OPERATING PROCEDURE 380
PURGEABLE AROMATICS AND HYDROCARBONS BY GC PID/FID**

Revision 5
Effective Date: March 28, 2005

Reviewed by: _____
Richard Bauer Date
Chemistry Team Leader/Technical Director

Reviewed by: _____
K. W. Hendrix, Laboratory QA Officer Date

Approved by: _____
Brenda Bettencourt, Laboratory Director Date

Periodic Review:

Signature	Title	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____

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APPENDIX A. DEVIATIONS FROM THE REFERENCE METHOD

APPENDIX B. ANALYTES AND QUANTITATION LIMITS

APPENDIX C. QUALITY CONTROL MEASURES AND CRITERIA

APPENDIX D. RECOMMENDED INSTRUMENT PARAMETERS

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APPENDIX G. TYPICAL DATA PACKAGE FORMAT

APPENDIX H. SOP DISTRIBUTION AND ACKNOWLEDGEMENT LIST

1 PURPOSE AND APPLICABILITY

This method describes the procedures used to determine benzene, toluene, ethyl benzene, xylenes (BTEX), tert-butyl methyl ether (MTBE), and total petroleum hydrocarbons as gasoline (TPH-g) in water and solid matrices. The structural isomers meta and para xylene coelute and they are reported as an isomeric pair.

Samples may be screened for MTBE using this method. However, no confirmation is provided; therefore only absence of MTBE can be determined definitively. Hydrocarbon compounds, especially from weathered fuels, can co-elute with methyl tert-butyl ether and cannot be distinguished from it using this method. This method can be used for monitoring wells where the presence of MTBE has been confirmed during previous sampling events using either GC/MS analysis or a dual column GC procedure.

This SOP is based on methods 5030B, 5035, 8015B, and 8021B, from EPA SW-846 Revision 2, December 1996. Deviations from reference methods are described in Appendix A.

Analytes and quantitation limits are provided by matrix in Appendix B.

2 SUMMARY

An inert gas is bubbled through a portion of an aqueous sample (or methanol extract from solid samples). Volatile organic compounds are vaporized and swept through a sorbent column where they are adsorbed. The sorbent column is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column.

A temperature program is used in the gas chromatograph to separate the organic compounds followed by detection using a photo ionization detector (PID) and a flame ionization detector (FID) in series. The PID is used to quantitate the aromatic compounds and MTBE while TPH as gasoline is quantitated with the FID.

TPH as gasoline is quantitated by comparing its area sum response over the retention time range which it elutes to the area sum response of gasoline standards analyzed under the same conditions as the sample. If required, probable identification of gasoline in samples is done by comparing the chromatographic pattern generated by analysis of the sample to the chromatographic pattern of gasoline analyzed under the same conditions as the standard. The identification of TPH as gasoline may be complicated by environmental processes such as evaporation, biodegradation, or the presence of more than one fuel type.

A single response analyte is quantitated by comparing its area response in its expected retention time window to the area response of standards analyzed under the same conditions as the sample.

3 DEFINITIONS

Analytical Sample - Any sample in which anions are being determined, excluding standards, blanks, or QC reference samples.

Calibration Blank (CB) - A blank that is the same matrix as the calibration standards, but without the analytes.

Continuing Instrument Calibration Verification (CCV) – A standard containing the analytes of interest, which is used to verify the accuracy of the analysis and monitor instrument drift. It is analyzed periodically throughout the analysis sequence (after every ten samples and at the end of the analytical run). The CCV meets the requirement of the instrument performance check sample specified in the reference method.

FID - Flame Ionization Detector.

Initial Calibration Standards (ICAL) – Standards used to calibrate the instrument response with respect to analyte concentration.

Laboratory Control Sample (LCS) - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is analyzed like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LCS is also known as a laboratory fortified blank (LFB) or blank spike (BS).

LIMS - Laboratory Information Management System. The Element database.

Matrix Spike (MS) - An aliquot of an analytical sample to which known quantities of the method analytes are added. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations. The MS is also known as laboratory fortified matrix (LFM).

Matrix Spike Duplicate (MSD) – A duplicate aliquot of an analytical sample to which known quantities of the method analytes are added. The MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results and to determine laboratory precision. The MSD is also known as laboratory fortified matrix duplicate (LFMD).

Method Blank (MB) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample. The MB is used to detect sample contamination resulting from the procedures used to prepare and analyze the samples in the laboratory environment. The MB is also known as laboratory reagent blank (LRB).

Method Detection Limit (MDL) - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

PID – Photo Ionization Detector.

Quantitation Limit (QL) - The concentration at which confidence in the reported value requires no qualifying remarks. A standard is analyzed at the QL to verify the previously established calibration curve.

Quantitation Limit Standard (QLS) - A standard used to check the accuracy of the analysis at the quantitation limit. Equivalent to the lowest level calibration standard.

Sample Delivery Group (SDG) - A group of twenty samples or less from a project that is sent to the laboratory for analysis.

Second Source Calibration Verification (SCV) - A solution of method analytes of known concentrations that is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check the initial calibration. The SCV is also known as quality control sample (QCS).

Solid Sample - For the purpose of this method, a sample taken from matrices classified as soil, solid, sludge, or sediment.

Stock Standard Solution (SSS) - A concentrated standard containing the method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

Storage Blank (SB) – An aliquot of reagent water stored with samples in the sample storage refrigerator. The storage blank indicates whether contamination may have occurred during sample storage.

Surrogate Analyte (SA) - A pure analyte which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in a known amount before extraction or other processing, and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

TPH - Total Petroleum Hydrocarbon.

Water Sample - For the purpose of this method, a sample taken from matrices classified as drinking, surface, ground, or storm runoff water, or industrial or domestic wastewater.

4 HEALTH & SAFETY

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation must be performed in a well-vented laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN for additional information.

Safety precautions must be taken when handling solutions and samples. Protective clothing including laboratory coats, safety glasses, and gloves must always be worn. Contact lenses must not be worn. If solutions come into contact with your eyes, flush with water continuously for 15 minutes. If solutions come in contact with your skin, wash thoroughly with soap and water. ESAT personnel should contact the Group Leader or Health and Safety and Environmental Compliance Task Manager and EPA staff should see the Team Leader or the Laboratory Safety, Health and Environmental Compliance Manager to determine if additional treatment is required. Refer to the Material Safety Data Sheets located in the library and the LAN for additional information.

4.1.1 Methanol

Methanol is the primary solvent used for the preparation of standards and for soil sample extraction in these procedures. Methanol is harmful if inhaled and may be fatal or cause blindness if ingested. Symptoms of overexposure via inhalation are drowsiness and intoxication, headache, visual disturbances leading to blindness, coughing, and shortness of breath, collapse, and death at high concentrations. Skin contact may result in absorption producing toxic effects. Repeated skin contact may cause burning, itching, redness, blisters or dermatitis. Eye contact can cause burning, watering, redness and swelling.

High vapor concentration will result in similar symptoms in the eyes. Medical attention must be sought whenever symptoms of inhalation or ingestion are observed as many effects are delayed due to the slow rate of metabolism.

Methanol is classified as a flammable solvent and must be handled accordingly. Use methanol in a laboratory fume hood with appropriate personal protective equipment (laboratory coat, nitrile gloves and safety glasses). Store methanol in a flammable storage cabinet away from oxidizers and sources of ignition.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must either be capped or have a column connected at all times.

4.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Pollution Prevention Plan* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the volume of any reagent or standard described in Sections 7.2 or 7.3 so long as good laboratory practices are adhered to regarding the accuracy and precision of the glassware, syringes, and/or analytical balances used to prepare the solution. Reducing the concentration of a reagent is not allowed under this procedure because the impact of such a change on the chemistry of the procedure must be assessed prior to implementation.

Reduce the toxicity of waste by purchasing lower concentration stock standards, lower concentration stock reagents, and solutions to replace neat chemicals whenever possible. However, do not change the concentrations of standards and reagents

specifically designated in this SOP.

4.4 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedure* and City of Richmond Discharge Permit. Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

This procedure produces the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-regulated Waste	Not applicable
Sample extracts	Hazardous Waste	See methanol, gasoline and other analyte MSDSs

5 SAMPLE HANDLING AND PRESERVATION

5.1 Containers and Required Sample Volume

- Aqueous samples should be collected in 40-mL VOA vials and preserved with HCL to pH <2.
- Soil samples should be collected in EnCore™ containers, or pre-weighed vials preserved in the field with methanol. Soil samples may also be collected in glass jars or other containers.
- Volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, and minimize waste disposal. Three VOA vials of water or 15 g of solid sample should be sufficient to meet these objectives.

5.2 Internal Chain-of-Custody

- The sample custodian delivers water samples to a sample refrigerator in Room 201 or other area where the samples will be analyzed. The sample custodian delivers solid samples to a sample freezer in Room 201 or other area where the samples will be analyzed.
- When sample containers are moved from one location to another, the LIMS database internal custody form must be updated to indicate that the container disposition has changed as appropriate. At the end of the day, sample containers should be returned to the designated sample location. The LIMS database is then updated to change the container disposition to “available in”.
- Verify sample IDs and dates of collection against the chain-of-custody form.

5.3 Sample Storage

- Store water samples in a refrigerator maintained at $> 0^{\circ}\text{C}$ to 6°C .
- Store solid samples in a freezer maintained at $\leq -10^{\circ}\text{C}$.
- Return excess sample to the sample refrigerator in Room 201.

5.4 Holding Time

- Unpreserved water samples must be analyzed within 7 days of sampling; preserved water samples must be analyzed within 14 days of sampling.
- Solid samples must be extracted within 48 hours of sampling following Section 8.3.2. Sample extracts must be analyzed within 14 days.

6 INTERFERENCES

Chromatographic interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to anomalous peaks or elevated baselines in chromatograms, or by carryover when low concentration extracts are analyzed after high concentration extracts.

Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and dichloromethane) through the septum seal into the sample during storage and handling.

6.1 Carryover

Contamination by carryover can occur whenever high level and low level samples are

analyzed in sequence. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses.

For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging vessel with a detergent solution between analyses, rinse it with distilled water, then methanol. Dry in an oven at 105 °C. In addition, purge an aliquot of methanol through the affected port. Analyze a reagent water blank to show that the port is not contaminated before analyzing further samples. The trap and other parts of the system are also subjected to contamination; therefore, frequent bakeout and purging of the entire system may be required.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. Minor deviations may be made in specific apparatus and materials provided that they are documented and equivalency is maintained.

7.1 Instruments and Equipment

- Analytical balance - capable of measuring differences of 0.01 g.
- Gas chromatograph equipped with FID and PID detectors in series and a splitless injection port (Hewlett Packard 5890 Series II gas chromatograph, or equivalent).
- Data Acquisition and Processing System - Able to control the GC and to acquire, store, and process gas chromatographic data. The software must be able to calculate calibration factors and the concentrations of analytes in samples. Agilent Technologies EnviroQuant ChemStation software and data acquisition computers (or equivalent).
- Fused Silica Capillary Gas Chromatography Column - 75m x 0.53mm x 3µm DB-624 wide bore capillary column (or equivalent). Any capillary column that provides adequate resolution, capacity, accuracy, and precision, may be used. The column is interfaced to the purge and trap device through low dead-volume injector (OI Analytical).
- Purge and trap concentrator with autosampler. (OI DPM-16 autosampler and an OI 4560 purge and trap concentrator, or equivalent.)
- Tenax trap (OI #7) – alternate traps may be used, provided that the adsorption and desorption characteristics obtained achieve equivalent or better method sensitivity and precision.

7.2 Reagents

Record purchased reagents, such as methanol, in the Region 9 laboratory information management system (LIMS).

- Methanol, Burdick and Jackson purge and trap grade (232-1) or equivalent.
- Reagent Water: All references to water in this method refer to water in which method analytes or other interferences are at less than one-half the QL of the analytes of interest. The Region 9 laboratory organic-free deionized water is further cleaned by bubbling a contaminant-free inert gas through the water.
- Reagent Sand - Sand, sea washed (VWR Cat. # VW3358-3, or equivalent). Heat to 400°C for at least 1 hour before use. Store in a closed container.

7.3 Standards

All standards must be entered into the Region 9 LIMS.

Store unopened ampulated stock standard solutions, and all working standard solutions in glass bottles or vials with Teflon lined screw caps, at $\leq -10^{\circ}\text{C}$. Protect all standards from light. Fresh standards should be prepared every six months, or sooner if comparison with check-standards indicates a problem. The standard solution must be checked frequently for stability. Replace all working standard solutions after six months or sooner if QC results indicate a problem.

The following solution concentrations are recommended only; other concentrations can be used.

CAUTION: Allow all standard solutions to equilibrate to room temperature before use.

- Stock Standard Solutions: Individual solutions of analytes purchased from commercial suppliers, such as Restek #30213 (BTEX Standard at 2,000 $\mu\text{g}/\text{mL}$ each), Restek #30205 (XHc Unleaded Gasoline Composite Standard at 50,000 $\mu\text{g}/\text{mL}$), and Restek #30402 (Methyl tert-Butyl Ether at 2,000 $\mu\text{g}/\text{mL}$), or equivalent. These are concentrated solutions in P&T methanol and are diluted to make the primary dilution standards.
- Primary Dilution Standards (PDS): Prepare a solution to contain all single component method analytes, but not the surrogate compound, at a concentration of 10 $\mu\text{g}/\text{mL}$ in methanol. Prepare a solution of the gasoline composite standard at 100 $\mu\text{g}/\text{mL}$ in methanol.

- Water Surrogate Spike Solution: Solution of α,α,α -Trifluorotoluene in methanol at 125 $\mu\text{g/mL}$. Prepare from purchased solution such as Restek #30068, *α,α,α -Trifluorotoluene Mix*, α,α,α -Trifluorotoluene at 2,500 $\mu\text{g/mL}$ in P&T methanol, or equivalent.
- Soil Surrogate Spike Solution: Solution of α,α,α -Trifluorotoluene in methanol at 2,500 $\mu\text{g/mL}$. Use stock solution such as Restek #30068, *α,α,α -Trifluorotoluene Mix*, α,α,α -Trifluorotoluene at 2,500 $\mu\text{g/mL}$ in P&T methanol, or equivalent.
- Soil Matrix Spike/LCS Solution: Solution prepared at a concentration of 1,000 $\mu\text{g/mL}$ for BTEX/MTBE. For the gasoline, the matrix spike/LCS solution is the stock standard gasoline solution at 50,000 $\mu\text{g/mL}$.
- Water Matrix Spike/LCS Solution: Solutions prepared at concentrations of 10 $\mu\text{g/mL}$ for BTEX/MTBE and 100 $\mu\text{g/mL}$ for gasoline; equivalent to the PDS solutions.
- Calibration Verification (CCV) - Equivalent to the mid-point initial calibration solution of 8.0 $\mu\text{g/L}$ for BTEX/MTBE and 300 $\mu\text{g/L}$ for gasoline.
- Quantitation Limit Standard (QLS) - Equivalent to the lowest level calibration standard of 0.50 $\mu\text{g/L}$ for BTEX/MTBE and 50 $\mu\text{g/L}$ for gasoline.
- Second Source Verification (SCV) - Equivalent to the mid-point initial calibration solution of 8.0 $\mu\text{g/L}$ for BTEX/MTBE and 300 $\mu\text{g/L}$ for gasoline but prepared from a source different from the source of calibration standards.

7.3.1 Calibration Solutions

The following calibration solution concentrations are typical concentrations only; other concentrations can be used.

- Use the 10 $\mu\text{g/mL}$ primary dilution standards to prepare calibration solutions of the BTEX and MTBE analytes at five concentrations in organic free water at recommended concentrations of 0.5, 2.0, 8.0, 40, and 200 $\mu\text{g/L}$. Prepare the low level standard by adding 2.5 μL to 50 mL water in a volumetric flask; invert three times to mix and fill a 5 mL syringe with solution contained in the base of the flask. Prepare the remaining solutions by adding 1 μL , 4 μL , 20 μL , and 100 μL aliquots of the primary dilution standard directly to 5 mL of water in a syringe. Add 5 μL of the surrogate solution to each solution prior to injection into the sparge tube.

BTEX, MTBE Solution	Conc. $\mu\text{g/mL}$	Volume Used, μL	Final Volume, mL	Final Conc., $\mu\text{g/L}$
PDS Solution	10	2.5	50	0.50 (QLS)
Surrogate Spike	125	5	5	125
PDS Solution	10	1	5	2.0
Surrogate Spike	125	5	5	125
PDS Solution	10	4	5	8.0 (CCV)
Surrogate Spike	125	5	5	125
PDS Solution	10	20	5	40 (LCS)
Surrogate Spike	125	5	5	125
PDS Solution	10	100	5	200
Surrogate Spike	125	5	5	125

- Prepare the five gasoline calibration standards by adding 2.5 μL , 5 μL , 15 μL , 40 μL , and 100 μL aliquots of the gasoline primary dilution standard at 100 $\mu\text{g/mL}$ to 5 mL of water in a syringe to make standards at recommended concentrations of 50, 100, 300, 800, and 2000 $\mu\text{g/L}$. Add 5 μL of the surrogate solution to each solution prior to injection into the sparge tube.

TPH-Gas Solution	Conc. $\mu\text{g/mL}$	Volume Used, μL	Final Volume, mL	Final Conc., $\mu\text{g/L}$
PDS Solution	100	2.5	5	50 (QLS)
Surrogate Spike	125	5	5	125
PDS Solution	100	5	5	100
Surrogate Spike	125	5	5	125
PDS Solution	100	15	5	300 (CCV)
Surrogate Spike	125	5	5	125
PDS Solution	100	40	5	800 (LCS)
Surrogate Spike	125	5	5	125
PDS Solution	100	100	5	2,000
Surrogate Spike	125	5	5	125

7.4 Supplies

- pH paper (pH 0-14 range).
- Sand, white quartz - Aldrich Cat # 27,473-9, or equivalent.
- Gas-tight syringes (5- μL , 10- μL , 25- μL , 50- μL , 100- μL , 250- μL , 500- μL , 1-mL, 5-mL, and 25-mL).

- Volumetric flasks, Class A - Appropriate sizes with ground glass stoppers.
- Sparge Tubes - 18 mm x 150 mm Disposable Culture Tubes, VWR no. 60825-673 or equivalent.
- Stainless steel spatulas
- Microliter syringes (10- μ L, 25- μ L, 50- μ L, 100- μ L, 250- μ L, 500- μ L, and 1-mL).

8 ANALYTICAL PROCEDURES

8.1 Instrument Operation

Set up instruments using operating parameters provided in Appendix D. Adjust as needed to meet method and SOP requirements and chromatographic practice. Use a sparge volume of 5-mL.

Enter data into ChemStation using file naming conventions provided in Appendix E.

Bake the trap at 190°C (ensure an empty sparge tube is mounted on the autosampler at the selected position) and the GC oven at 250°C for at least 14 minutes each day before samples are analyzed.

Prior to analyzing calibration, QC, or field samples make a LIMS batch and sequence as required to obtain LIMS assigned IDs for the calibration and QC samples.

8.2 Calibration and Standardization

The calibration standards required depend on the analytical request, which may include BTEX/MTBE, gasoline, or both.

Set up the purge and trap concentrator for water analysis ensuring that the sparge needles reach to within 5 mm of the bottom of the sparge cells. The same calibration is used for the analysis of both water and soil methanol extracts.

8.2.1 Initial Calibration

Perform an initial calibration using a minimum of five calibration standards to establish an external standard linear calibration using the average calibration factor. Refer to Section 9.2.1 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

Check that compound type in ChemStation is set to H. This setting sums the area between the start and end of the analyte range. Inspect a chromatogram from the highest calibration standard from a previous ICAL to determine approximate times to start and stop integration. Enter these times in

ChemStation.

Analyze each of the initial calibration standards and an instrument blank as described in Section 8.3.1.

Example initial calibration sequence:

Sample Name		Sample Name	
1	IB	9	50 µg/L gasoline
2	0.5 µg/L BTEX/MTBE	10	100 µg/L gasoline
3	2.0 µg/L BTEX/MTBE	11	300 µg/L gasoline
4	8.0 µg/L BTEX/MTBE	12	800 µg/L gasoline
5	40 µg/L BTEX/MTBE	13	2000 µg/L gasoline
6	200 µg/L BTEX/MTBE	14	IB
7	IB	15	300 µg/L gasoline SCV
8	8.0 µg/L BTEX/MTBE SCV		

Spike the water with the appropriate amount of primary dilution standard for the specific calibration level being analyzed.

Inspect the high standard and update start and stop integration times in each calibration standard as needed.

Update each level of the ChemStation ICAL method. All target analyte and surrogate responses in the ICAL method should be replaced with the new responses.

Analyze a SCV standard immediately after each initial calibration. See Section 9.2.1 of this SOP for frequency and Appendix C for QC limits.

If the initial calibration, the SCV, and the IB meet all criteria specified in Appendix C, the remainder of the 12-hour analytical period may be used for the analysis of field and QC samples.

8.3 Sample Analysis

Check that the numbers on the vials coincide with the numbers on the routing forms to ensure that the correct sample is being analyzed.

If the sample has an unusual color, or other physical characteristic such as more than one phase, the presence of a precipitate, unusual viscosity, or physical signs of contamination a screening analysis is required to protect the analytical system from damage or contamination and to determine the appropriate subsequent dilution. If an

initial screening is necessary, analyze the sample at a 1:50 dilution, unless the group leader or Technical Director specifies otherwise. Also document anomalies in the LIMS MMO field.

Note in the LIMS MMO field in the work order window if there is headspace present in the sealed sample vial.

8.3.1 Water Sample Preparation

- Allow the samples to reach ambient room temperature before analysis.
- Break the chain of custody seal on the vial with a scalpel or other appropriate implement, and note if the seal is missing or compromised in any way.
- Check the pH of the sample using pH 0-14 range pH paper. Record the pH in the injection logbook. Note any samples that have a pH greater than 2 in the LIMS MMO field in the work order window.
- Fill a 5-mL syringe with the sample. Invert the syringe, remove any air bubbles, and bring the level to 5-mL by displacement with the plunger. Place any excess sample displaced from the syringe in the aqueous waste containers.
- Prepare MS, MSD, and LCS samples by spiking with the analytes of interest. Add 20 μL of the 10 $\mu\text{g}/\text{mL}$ BTEX/BTBE water matrix spike/LCS solution or 40 μL of the 100 $\mu\text{g}/\text{mL}$ gasoline water matrix spike/LCS solution to the matrix spike sample to prepare an MS/MSD or to reagent water to prepare an LCS.
- Spike the water with 5 μL of the 125 $\mu\text{g}/\text{mL}$ surrogate solution.
- Attach the syringe to the Luer lock mount on the purge and trap concentrator. Open the mount valve, inject the contents of the syringe into the spike cell, and close the valve. Remove the syringe from the mount.

8.3.2 Soil Sample Preparation

This section contains procedures for the extraction and analysis of soil samples collected as bulk samples in glass jars or other suitable containers, in EnCore™ sampler devices, or pre-weighed vials preserved in the field with methanol. The typical sample weight is 5 g (nominal).

The percent moisture is determined from a separate aliquot as described in EPA

Region 9 Laboratory SOP 460, *Percent Moisture Determination*.

Remove the sample from the refrigerator immediately prior to extraction or analysis. Samples should be extracted as soon as possible after receipt and within 48 hours of collection even if analysis will not to be performed immediately.

To prevent the loss of certain volatile organics the sample must not be allowed to reach room temperature. Break the chain of custody seal on the container with a scalpel or other appropriate implement, again making note in the logbook if the seal is missing or compromised in any way. Observe the sample closely for evidence of contamination. If the sample appears to contain hydrocarbons (an oily appearance or sheen) the sample must be analyzed at a dilution to prevent damage to the analytical system.

1. To extract bulk samples, use an EnCore™ sampler or a plastic syringe with the end cut off to subsample the soil container. Immediately transfer contents of the EnCore™ sampler or syringe into a 20-mL tared vial. Record the weight of soil added to the container to the nearest 0.1 g in the LIMS bench sheet. If possible, all samples within a sample delivery group should be extracted at the same time along with the MB preparation.

To extract samples collected with the EnCore™ sampling device, transfer the contents of the EnCore™ sampler into a 20-mL tared vial. Record the weight of soil added to the container to the nearest 0.1 g in the LIMS bench sheet.

2. Quickly add 10.0 mL of purge and trap grade methanol and 25 µL of the 2,500 µg/mL soil surrogate spike solution to the vial. Spike MS/MSD samples with the soil spiking solutions containing the analytes of interest at this time. Add 20 µL of the 1,000 µg/mL BTEX/MTBE soil matrix spike/LCS solution or 8 µL of the 50,000 µg/mL gasoline soil matrix spike/LCS solution. Cap the vial and vortex for 30 seconds. These steps must be done rapidly in order to prevent the loss of volatile organics from the sample.
3. Weigh samples collected in pre-weighed containers preserved in the field with methanol to the same level of precision as the weight recorded on the chain-of-custody or vial (0.1 g or 0.01 g). Enter the vial and methanol weight (the pre-weight) from the chain-of-custody or vial in the LIMS bench sheet and calculate the sample weight by subtraction. Quickly add soil surrogate spike solution to the vial at the rate of 2.5 µL per mL of methanol. An additional aliquot of the sample not preserved with methanol should have been collected to determine percent moisture.

Prepare a soil LCS by spiking 5 g of reagent sand in a 20-mL vial with the soil matrix spike/LCS solutions containing the analytes of interest. Add 20 μL of the 1,000 $\mu\text{g}/\text{mL}$ BTEX/MTBE soil matrix spike/LCS solution or 8 μL of the 50,000 $\mu\text{g}/\text{mL}$ gasoline soil matrix spike/LCS solution. Add 10.0 mL of purge and trap grade methanol and 25 μL of the 2,500 $\mu\text{g}/\text{mL}$ soil surrogate spike solution to the vial.

Transfer approximately 1 mL of extract to a GC vial for storage in the laboratory freezer at $\leq -10^{\circ}\text{C}$. Extracts must be analyzed within 14 days from sample collection. Use this extract for the analysis and any subsequent dilutions that may be necessary.

Analyze 100 μL of the extract in 5-mL of reagent water according to the instructions for 5-mL water analysis in Section 8.3.1.

8.3.3 Analytical Sequence and Sample Analysis

Set up a ChemStation data acquisition sequence from the LIMS sequence using the GC operating parameters in Appendix D. Include the client sample ID and the laboratory sample ID in the sample description field. Additional header information shall include the dilution factor, instrument ID, and the analyst's initials. Enter this sequence in the instrument run log, if used.

See Section 9.3 for batch quality control (QC) frequency and corrective action requirements. It is highly recommended that the MB, LCS, and MS/MSD extracts be analyzed as early as possible in the analysis of a batch.

If the initial calibration, the SCV, and the IB meet all criteria specified in Appendix C, the remainder of the 12-hour analytical period may be used for the analysis of field and QC samples.

Example Field Sample Analysis Sequence:

Sample Name		Sample Name	
1	IB	9	MS
2	8.0 $\mu\text{g}/\text{L}$ BTEX/MTBE CCV	10	MSD
3	0.5 $\mu\text{g}/\text{L}$ BTEX/MTBE QLS	11	Field sample
4	300 $\mu\text{g}/\text{L}$ gasoline CCV	12-16	Field samples
5	50 $\mu\text{g}/\text{L}$ gasoline QLS	17	Field sample
6	MB	18	IB
7	LCS	19	8.0 $\mu\text{g}/\text{L}$ BTEX/MTBE CCV
8	Matrix Spike Sample	20	300 $\mu\text{g}/\text{L}$ gasoline CCV

Enter the first and last sample positions in the concentrator and, with the ChemStation software in data acquisition mode, press the start button on the concentrator to begin purging the first sample. The purge and trap concentrator parameters are found in Attachment D.

Observe the initial purging of the sample to determine if the sample is liable to foam during the purging process. If the sample does foam, it can be analyzed as long as the foam does not enter the sparge vessel neck and enter the transfer line leading to the trap.

If it appears that the sample will foam excessively, discontinue the purging by pressing the [2nd], [on], [enter] keys. Drain the sparge cell; rinse it with methanol, then reagent water. Place the sample waste and rinsate in the aqueous waste container. Bake out the trap and the GC for 25 minutes before analyzing additional samples. Analyze a reagent water blank to show that the sampler is free from contamination before analyzing sample.

Analyze the sample at a 1:10 dilution of the sample, or other appropriate dilution to prevent foaming even though the detection limits are elevated. Document any sample foaming in the run log and the LIMS MMO field.

8.3.4 Analyte Identification and Quantitation

Update the center of the retention time window for each single response analyte and the surrogate by using the absolute retention times from the calibration verification standard at the beginning of the analytical shift.

All single response analytes and surrogates in the field and QC samples must fall within the established retention time windows.

If the retention time does not fall within the retention time window, then take corrective action to restore the system. If repairs to the system are required then a new initial calibration must be performed.

Quantitate the sample data using the ChemStation software using the appropriate initial calibration mean CFs. Quantitate methanolic extracts of soil samples with the same initial calibration used to quantitate water samples. If applicable, indicate degree of similarity of sample chromatogram to the gasoline standard. Print out quantitation reports and chromatograms for each field and QC sample.

8.3.4.1 Water Calculations

Calculate target analyte concentrations in aqueous samples using Equation 1:

$$\text{Concentration (ug/L)} = \frac{A_x \times DF}{RF}$$

Where:

A_x = area response for analyte x

DF = dilution factor

RF = mean response factor from the initial calibration
(area/concentration)

8.3.4.2 Soil Calculations

Calculate target analyte concentrations in soil samples using Equation 2:

$$\text{Concentration (mg / Kg dry weight basis)} = \frac{A_x \times V_t \times DF \times V_p \times 1,000}{RF \times W \times D \times V_i \times 1,000}$$

Where:

A_x = area response for analyte x

D = dry weight factor (Percent solids/100)

W = weight of sample in grams

RF = mean response factor from the initial calibration
(area/concentration)

V_t = total volume of methanol extract in mL

DF = dilution factor

V_i = volume of extract injected in μL

V_p = volume of extract purged in mL (i.e. 5mL)

1,000 (in numerator) = $\mu\text{L/mL}$

1,000 (in denominator) = mL/L

Yields concentration units of $\mu\text{g/g} = \text{mg/Kg}$

8.3.5 Manual Integration

Review the baseline drawn by the data system integrator to verify that it accurately reflects the area response of the sample components. If in the judgment of the analyst, it does not, then correct the integration using the ChemStation QEDIT software module. Document any manual integrations following the procedure described in U.S. Environmental Protection Agency Region 9 SOP 835, *Chromatographic Integration Procedures*.

8.3.6 QC Review

As soon as possible after analysis (typically prior to entry into LIMS), inspect sample and QC data for compliance with QC limits in Appendix C. If no significant problems are found, review the following QC data for compliance with SOP requirements:

- Target analyte results must be within range of initial calibration.
- Process and review the results for the IB, CCV, and QLS instrument QC samples. Print a ChemStation Evaluate Continuing Calibration Report using the appropriate settings to verify that the CCV and QLS QC sample results are within QC limits. See Section 9.2 for instrument QC requirements.
- Process and review the results for the MB, LCS, and MS/MSD batch QC samples and verify that the results are within QC limits. See Section 9.3 for Batch QC requirements.
- Check that surrogate compound retention times are within the window specified in Section 9.4.1 and Appendix C. Determine if surrogate recoveries for field and QC samples are within QC limits. Report the surrogate recovery from the FID when reporting TPH and from the PID when reporting BTEX/MTBE. If there is matrix interference with the surrogate response on the FID when determining TPH, report the surrogate recovery from the PID as the sample surrogate recovery instead. See Section 9.4 for Sample QC requirements.
- Review all sample results to determine if any samples need to be re-analyzed at a dilution.

If any of the target compounds in soil samples exceed the initial calibration range of the instrument, dilute by using a smaller aliquot of the extract combined with IB water to a total volume of 5 mL.

- If a run is rejected for any reason, mark the raw data “Not Used” in large print and document the reason on the quantitation report.

8.3.7 Data Export and LIMS Entry

- Generate epatemp.txt files for field and QC samples by also printing the report to the screen; these files are used by the LIMS DataTool module to import the instrument results into the Data Entry/Review table.

- Copy sample data files from the local drive to the appropriate instrument data subdirectory on the Region 9 LAN to make them available to LIMS and to archive them.
- Create an empty upload file containing the samples analyzed in the LIMS batch or sequence. Import and merge the data files using the LIMS DataTool module. Load the resulting merged data file into the LIMS Data Entry/Review table. See LIMS manual for detailed procedure.
- In order to take the dilution that occurs during soil sample preparation into account, the dilution factor for undiluted soil samples in the LIMS Data Entry/Review table must be 50. Any actual sample dilutions must be multiplied by 50 to obtain the effective sample dilution to be entered in LIMS. Edit dilutions in DataTool or LIMS entry table as needed.
- Review results in the LIMS. Qualify and flag results in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

8.4 Maintenance

The analyst should observe trends in the data such as declining response, erratic relative response, loss of classes of compounds, etc., which may signal the need for instrument maintenance. Document all routine maintenance or corrective actions taken in the maintenance logbook. Preventative maintenance procedures are listed in Appendix F.

The following sections describe possible causes and corrective actions for common problems. Refer to Appendix F for routine preventative maintenance procedures and schedule.

8.4.1 Purge and trap maintenance

Symptom:

- Carryover
Possible causes: Cold spot in system, especially the transfer lines between the sparge unit and the concentrator or between the concentrator and the GC or analyzing a sample containing high mole weight components or analyzing high-level and low-level samples sequentially.
Corrective action: Check temperatures of all heated zones. Adjust temperatures or replace heaters as required. Flush valve, gas lines, and sample lines with methanol or reagent water and bake out.

- Loss of sensitivity to selected analytes and increased pressure to maintain purge flow.
Possible cause: Degradation of trap.
Corrective action: Replace trap.
- Loss of all purged analytes.
Possible cause: Leak in system.
Corrective action: Leak check purge and trap system. Inspect sparge ferrules and replace them when worn or distorted.

8.4.2 GC Maintenance

Symptom

- Carryover
Possible causes: Analyzing a sample containing high mole weight components or analyzing high-level and low-level samples sequentially.
Corrective action: As necessary, replace inlet liner, clean inlet, bake out inlet, bake out column, clip column, replace septum, replace column.
- Shorter retention time.
Possible cause: column flow rate problem.
Corrective action: check flow rate and adjust as necessary.
- Longer retention time and or smaller peaks.
Possible causes: column flow rate problem, injection port leak, or column contamination.
Corrective action: as necessary, check for leaks, replace septum, replace the liner, replace the lower injection port seal, and cut the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.
- Loss of resolution.
Possible causes: column flow rate problem, injection port leak, or column contamination.
Corrective action: check for leaks, replace septum, replace the liner, replace inlet seal, clip the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.
- Loss of sensitivity (for PID analytes).
Possible causes: dirty PID window or defective PID lamp.
Corrective action: clean with mild abrasive such as iron oxide slurry (do not use alumina). If cleaning the window does not improve sensitivity, the lamp may need to be replaced.

9 QUALITY CONTROL

9.1 Demonstration of Capability

The EPA Region 9 Laboratory operates a formal quality control program. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated. A summary of QC criteria is provided in Appendix C.

A Demonstration of Capability must be in place prior to using an analytical procedure and repeated if there is a change in instrument type, personnel, or method. Follow procedures described in EPA Region 9 Laboratory SOP 880, *Demonstration of Laboratory Capability and Analyst Proficiency* for more details.

9.1.1 Retention Time Windows

- Establish retention time windows for the single response analytes and the surrogate whenever a new GC column is installed or a new DOC is required on each chromatographic column and instrument. Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. See Section 9.2.1 for retention time window criteria.
- Make three injections of the mid-level BTEX/MTBE calibration standard over the course of at least a 72-hour period.
- Record the retention time to three decimal places (e.g., 9.007) from three injections. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too narrow.
- Calculate the mean and standard deviation of the three absolute retention times using Equation 4. If the standard deviation of the retention times for a target compound is less than 0.01 minutes then use a default standard deviation of 0.01 minutes.
- The width of the retention time window is defined as ± 3 times the standard deviation of the mean retention time. If the default standard deviation is employed, the width of the window will be ± 0.03 minutes.
- For samples run during the same shift as an initial calibration, use the

retention time of each analyte and surrogate in the mid-point standard of the initial calibration as the center of the retention time window.

- Document the RT window calculations in a spreadsheet and store them in the laboratory where the samples are analyzed.

9.2 Instrument QC

9.2.1 Initial Calibration

Demonstration and documentation of an acceptable initial calibration are required before any samples are analyzed

The GC system must be calibrated whenever corrective action changes instrument response (e.g., detector gas adjustment, column replacement, etc.) is performed or if the calibration verification criteria cannot be met.

- The data system calculates the calibration factor (CF) using Equation 3.

Equation 3

$$CF = (A_x) / (C_x)$$

Where

A_x = Area of analyte x, or area sum response of gasoline

C_x = Concentration of the standard injected (μg/L)

- The data system calculates the percent relative standard deviation (%RSD) of the CF values for each analyte using Equation 4.

Equation 4

$$\%RSD = (SD / CF_{avg}) \times 100$$

Where SD is the sample standard deviation and is calculated as:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - CF_{avg})^2}{n - 1}}$$

Where:

CF_{avg} = Mean calibration factor from the initial calibration.

CF_i = Calibration factor for a calibration level.

- Print a ChemStation Response Factor Report. Verify that the %RSD of the target analytes and the surrogate are within QC limits immediately after the initial calibration is finished. See Appendix C for QC limits.
- If an ICAL fails because of one standard, a fresh solution of that standard may be re-analyzed and substituted for the failed one in the ICAL. If more than one standard fails, corrective action is required.
- Analyze an SCV sample immediately after each initial calibration. Calculate the calibration factor (CF) for the target analytes and the surrogate compound using Equation 3.
- Calculate the percent difference (%D) between the SCV CF and the initial calibration average CF for the target analytes and the surrogate using Equation 5.

Equation 5:

$$\%D = \frac{CF_c - CF_{avg}}{CF_{avg}} \times 100$$

Where:

CF_c = SCV or CCV CF

CF_{avg} = ICAL mean CF

- See Appendix C for QC limits. If the SCV sample fails it may be repeated once. If the second SCV fails, the cause for failure must be determined and corrected before analysis of samples can proceed.

Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

9.2.2 Continuing Calibration Verification

- Analyze a CCV standard at the beginning of each 12-hour analytical period and at the end of the 12-hour analytical period. The 12-hour analytical period begins with the injection of the CCV standard and ends with the injection of the last sample that can be injected within 12 hours of the beginning of the period.

- Calculate the calibration factor (CF) for the target analytes and the surrogate compound using Equation 3.
- Calculate the percent difference (%D) between the calibration verification CF and the initial calibration average CF for the target analytes and the surrogate using Equation 5.
- The %D must be within QC limits. See Appendix C for QC. If an analyte fails this criterion a second calibration verification may be analyzed. Repeated failure requires that corrective action be taken to restore the system before any additional samples are analyzed. All affected samples must be re-analyzed.

If repairs to the system are required then a new initial calibration must be performed. The analyst should observe trends in the data such as declining response, erratic response, etc., which may signal the need for instrument maintenance.

- Acceptable sample analyses must be bracketed by the analyses of calibration verification standards that meet QC limits.

9.2.3 Quantitation Limit Standard

- Analyze a quantitation limit standard (QLS) for the analytes of interest each day when analyses of field or QC samples are performed. The QLS is used to verify analytical system response at the quantitation limit.
- Calculate the concentration of the target analytes using Equation 1.
- Calculate the percent of true value (TV) for the target analytes using Equation 6.

Equation 6:

$$\% \text{ True Value} = (C_d / T_v) \times 100$$

Where:

C_d = Concentration determined by analysis

T_v = True value of standard

- If the % TV is not within the QC limits in Appendix C, analyze a second QLS sample. Repeated failure requires that the cause be determined and corrected before analysis of samples can begin. If repairs to the system are required then a new initial calibration must be performed.

9.2.4 Instrument Blank

- At a minimum, one acceptable IB is required for each 12-hour analysis period.
- Evaluate the IB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- If the IB results are not within QC limits, analyze a second IB. If the second IB also fails but the system is significantly cleaner, another IB may be analyzed; if not, take corrective action.
- Corrective action - If the IB is not acceptable the source of the contamination must be found and eliminated and the problem documented before analysis can proceed.
- Surrogate recovery is not evaluated for IB QC samples.

9.3 Batch QC

9.3.1 Method Blank

- Extract and analyze a method blank (MB) with each extraction batch or every 20 samples, whichever is more frequent, to demonstrate that the entire analytical system - from extraction through GC analysis - is free of contamination.
- For aqueous samples a MB is identical to an IB. For soil sample analysis it is necessary to prepare an extracted MB.
- Evaluate the MB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- Corrective action - If the MB result exceeds QC limits and the sample result is less than five times the MB analyte result, re-analyze the MB. If the MB result still exceeds QC limits then the MB and all associated samples must be re-prepared and re-analyzed. If the MB result exceeds QC limits and the sample result is \geq five times the MB result or is not detected then report the sample result.
- If the surrogate recovery does not meet acceptance criteria, re-analyze the MB. If the surrogate recovery still does not meet acceptance criteria, the batch may have to be re-extracted

9.3.2 Laboratory Control Sample

- Analyze a laboratory control sample (LCS) to demonstrate that the analytical system is in control. An LCS is extracted and analyzed once per extraction batch or every 20 samples, whichever is more frequent. The LCS is an MB spiked with matrix spiking solution.
- Calculate the percent recovery (%R) using Equation 7.

Equation 7:

$$\% \text{ Rec} = ((\text{SSR} - \text{SR})/\text{SA}) \times 100$$

Where,

SSR = Spiked sample result

SR = Unspiked sample result

SA = Spike added

- The %R must be within the QC limits in Appendix C. If acceptable accuracy cannot be achieved, the problem must be located and corrected prior to reporting any sample data and before additional samples are analyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate

- Matrix spike (MS) and matrix spike duplicate (MSD) samples are extracted and analyzed for each batch of twenty or fewer samples extracted as a group. Matrix QC samples are usually designated in the field. In the event that a sample was not designated as the matrix spike sample and adequate sample volume exists, the analyst will choose one representative sample from the SDG for QC analysis. Do not choose any obvious field blanks as the QC sample.
- Calculate the recovery of each analyte using Equation 7.
- Calculate the relative percent differences (RPD) of the recoveries of each analyte in the MS and MSD using Equation 8.

Equation 8:

$$\text{RPD} = \frac{(\text{MSC} - \text{MSDC})}{(\text{MSC} + \text{MSDC}) / 2} \times 100$$

Where,

MSC = Measured concentration of analyte in MS

MSDC = Measured concentration of analyte in MSD

- See Appendix C for QC limits.

The MS/MSD recovery limits are advisory limits only. If the limits are not met, then no further action is required, as long as the LCS is within limits, since the purpose of these analyses is to determine matrix effects on compound recovery. However, frequent failure to meet the recovery or RPD criteria should alert the analyst that a problem may exist and must be investigated. The analyst should analyze the matrix spike solution and check the recoveries of the spike compounds. A new solution should be prepared if the recoveries are not within 20% of expected.

- The table below lists the action to be taken based on the LCS and MS/MSD results.

QC ACCEPTANCE MATRIX			+ = PASS		- = FAIL			
CASE	1	2	3	4	5	6	7	8
LCS - % REC	+	+	+	+	-	-	-	-
MS/MSD -% REC	+	-	+	-	+	-	+	-
MS/MSD - RPD	+	+	-	-	+	+	-	-

Case 1: Extraction batch acceptable.

Case 2: Extraction batch acceptable; matrix effect confirmed.

Cases 3 & 4: Extraction batch is unsatisfactory. Investigate MS/MSD problem and document findings in report narrative.

Case 5: Extraction batch rejected. Batch may have to be re-extracted unless LCS problem is determined and documented.

Cases 6, 7 & 8: Extraction batch rejected. Re-extract batch.

9.3.4 Storage Blank

- Every Monday morning, or the first workday of the week, fill three 40-mL screw-cap volatile vials with PTFE-faced silicone septum with reagent water and store them with the samples, in the sample storage refrigerator in

Room 201.

- Analyze a storage blank (SB) once every week while samples are being stored waiting for analysis. The storage blank indicates whether contamination may have occurred during sample storage.
- If samples have been stored in the refrigerator during the previous week, analyze the storage blank the following Monday, or on the first work day of that week. If samples have not been stored in the refrigerator during the previous week, discard the blanks and place new storage blanks in the refrigerator.
- Evaluate the SB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- If the SB does not meet QC criteria all affected data must be qualified.

9.4 Sample QC

9.4.1 Surrogate Recovery

- Calculate the surrogate recovery in all field and QC samples immediately after analysis using the following formula:

Equation 9:

$$\%R = (\text{Amount Found}/\text{Amount Spiked}) \times 100.$$

- The surrogate recovery must be within QC limits. See Appendix C for QC limits.
- Take the following steps if surrogate recovery is not within the limits:
 1. Ensure that there are no calculation errors, and check the system performance.
 2. Re-analyze the extract if a system performance problem or calculation error is not evident. Distinguish between the analysis and re-analysis by adding an "RE[X]" suffix to the laboratory ID on the re-analysis, where X is a sequential number that identifies the reanalysis. The extract may be diluted for re-analysis if examination of the chromatogram so indicates.
 3. If re-analysis of the extract does not solve the problem, the sample may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case-by-case basis.

- Do not re-extract undiluted samples with surrogate recoveries outside the limits if the diluted analysis with acceptable surrogate recoveries is being submitted. Report the event in the run log.
- Do not re-analyze the MS/MSD samples, even if surrogate recoveries are outside the limits.
- If the sample associated with the MS/MSD analyses does not meet the surrogate recovery criteria, it should be re-analyzed only if the matrix spike and duplicate surrogate recoveries are within the limits. If the sample and spikes show the same pattern (i.e., outside the limits), then the sample does not need re-analysis.
- If the surrogate recoveries of the re-analysis of the extract are within limits, then:
 1. If the re-analysis was undiluted, the problem was within the laboratory's control. Report the results from the re-analysis and submit the data from both analyses. Distinguish between the analysis and re-analysis by adding an "RE" suffix to the sample ID on the re-analysis.
 2. If the re-analysis was diluted, the problem was a matrix effect. Report the results from the re-analysis and submit the data from both analyses and discuss the result in the report narrative. Distinguish between the analysis and re-analysis by adding an "RE" suffix to the sample ID on the re-analysis.
 3. If the surrogate recoveries of the re-extraction are within limits, then the problem was within the laboratory's control. Report the results from the re-extraction
 4. If the re-extraction does not solve the problem, report the results from the first analysis and submit the data from both analyses

9.5 Method Performance

Region 9 Laboratory performance for this procedure from January 1, 2003 to February 28, 2005 is summarized in the following table.

Method Performance

Analyte	Matrix	QC Type	Number of Measurements	Mean Recovery, %	95% Confidence Interval (2 σ)
Benzene	Water	LCS	7	105	83.7-126

Analyte	Matrix	QC Type	Number of Measurements	Mean Recovery, %	95% Confidence Interval (2σ)
Ethyl Benzene	Water	LCS	7	109	87.2-130
TPH as Gasoline	Water	LCS	28	95.3	81.1-110
Toluene	Water	LCS	7	105	83.6-127
m&p-Xylene	Water	LCS	7	105	95.1-115
o-Xylene	Water	LCS	7	99.3	96.8-102
MTBE	Water	LCS	7	96.6	81.8-111
Benzene	Solid	LCS	7	106	83.7-126
Ethyl Benzene	Solid	LCS	7	104	87.2-130
TPH as Gasoline	Solid	LCS	32	104	77.4-130
Toluene	Solid	LCS	7	107	90.5-124
m&p-Xylene	Solid	LCS	7	105	97.7-112
o-Xylene	Solid	LCS	7	103	90.4-115
MTBE	Solid	LCS	7	96.7	81.3-112

The following functional areas of the SOP may be significant sources of analytical error:

- Poor purge efficiency due to specific analyte characteristics or other problems.
- Standard degradation
- Volatile compound losses in spike solutions and standards.
- Chromatographic separation and peak integration.

10 DOCUMENTATION

10.1 Standards

Record the preparation of all standards in the Element database. Include a copy of each Analytical Standard Record associated with sample analysis in the data package.

10.2 Analytical sequence

The analytical sequence is documented in the Element database in the instrument Run Log. Case Number, SDG number, date of analysis, QC solution IDs, analyst initials, laboratory sample IDs, client sample IDs, dilution factors and comments, if any, are recorded.

10.3 Analytical Report and Data Package

Analytical reports are produced using the Element database. The data package is

produced from Element database and manual log records. Appendix G provides the typical format for data package deliverables.

10.4 Maintenance Logbook

Maintain a maintenance logbook for each instrument. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control. Document all preventive or routine maintenance performed, as well as repairs or corrective or remedial actions in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control*.

10.5 SOP Distribution and Acknowledgement

Distribute the approved SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. Document using the SOP Distribution and Acknowledgement List as shown in Appendix H.

11 REFERENCES

Agilent Technologies EnviroQuant ChemStation User's Guide

HP 5890 Gas Chromatograph Users Manual

OI 4560 and DPM16 Operator's Manuals.

U.S. Environmental Protection Agency, *Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December 1996*.

U.S. Environmental Protection Agency, *Method 5035, Closed-system Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December 1966*

U.S. Environmental Protection Agency, *Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003*.

U.S. Environmental Protection Agency, *Method 8015B, Nonhalogenated Organics Using GC/FID, Revision 2, December 1996*.

U.S. Environmental Protection Agency, *Method 8021B, Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors, Revision 2, December 1996*.

U.S. Environmental Protection Agency Region 9 *Laboratory Quality Assurance Plan*,

Revision 7, June 7, 2004

USEPA Region 9 Laboratory SOP 110, *Sample Receiving and Login.*

U.S. Environmental Protection Agency Region 9 SOP 125, *Disposal Procedures for Unused Aqueous Environmental Samples*

U.S. Environmental Protection Agency Region 9 SOP 706, *Laboratory Waste Management Procedures*

U.S. Environmental Protection Agency Region 9 SOP 805, *Refrigerator Temperature Monitoring*

U.S. Environmental Protection Agency Region 9 SOP 820, *Laboratory Discrepancy and Corrective Action Reporting Procedures*

U.S. Environmental Protection Agency Region 9 SOP 835, *Chromatographic Integration Procedures*

U.S. Environmental Protection Agency Region 9 SOP 840, *Notebook Documentation and Control*

U.S. Environmental Protection Agency Region 9 Laboratory SOP 880, *Demonstration of Capability*

APPENDIX A.
DEVIATIONS FROM THE REFERENCE METHOD

1. The CF is area/concentration unit ($\mu\text{g/L}$) not area/mass (ng) as in the reference method. The formulas for determining sample analyte concentrations have been modified to reflect this change.
2. Control limits for surrogate, LCS, and MS/MSD recoveries are specified in the SOP, not from evaluation of laboratory data.
3. The retention time range for gasoline is established from the retention time range over which it elutes, not the retention times of 2-methylpentane and 1,2,3-trimethylbenzene as specified in the reference method.

APPENDIX B.
ANALYTES AND QUANTITATION LIMITS

Analyte	QL, on column, $\mu\text{g/L}$	QL, 5g Solid, mg/kg	QL, 5 mL Water, $\mu\text{g/L}$
TPH as gasoline	50	5.0	50
Benzene	0.5	0.05	0.5
Toluene	0.5	0.05	0.5
Ethyl benzene	0.5	0.05	0.5
o-Xylene	0.5	0.05	0.5
m&p-Xylene	1.0	0.10	1.0
tert-Butyl methyl ether (MTBE)	0.5	0.05	0.5

**APPENDIX C.
QUALITY CONTROL MEASURES AND CRITERIA**

QC MEASURE	CRITERIA
Initial Calibration (ICAL) RSD	≤ 20
Second Source Verification (SCV) %D	$\leq \pm 30$
Calibration Verification (CCV) %D	$\leq \pm 15$
Quantitation Limit Standard (QLS)	$\pm 40\%$ of TV
Blanks – MB, IB, SB	$< \frac{1}{2}$ QL
Laboratory Control Sample (LCS) %R	70 - 130
MS/MSD %R	70 - 130
MS/MSD RPD	≤ 25
Surrogate Recovery of QC and field samples (except IB) %R	70 – 130
Retention Time Windows	± 0.03 minutes, or as determined

APPENDIX D.
RECOMMENDED INSTRUMENT PARAMETERS

OI 4560 Concentrator

Recommended operating settings for the OI 4560 purge & trap concentrator that is interfaced with the HP 5890 Series II GC and the DPM-16 autosampler is as follows.

<u>PARAMETER</u>	<u>SETTING</u>
Purge temperature	20°C
Sample temperature	ambient
Purge Time	11 minutes
Dry purge	2.3 minutes
Purge Flow	35 - 40 mL/min
Desorb	2.00 minutes @ 180°C
Bake	14 minutes @ 190°C
Valve temperature	100°C
Mount temperature	40°C
Line temperature	100°C
DPM16 transfer line	100°C
DPM Valve temperature	100°C
Water Management	ON
Purge Temperature	100°C
Desorb Temperature	20°C
Bake Temperature	240°C

HP 5890 Series II Gas Chromatograph

<u>PARAMETER</u>	<u>SETTING</u>
Injector temperature	225°C
Column Equilibration time	0.5 minutes
Initial Oven Temp	35°C
Initial Oven Time	3.0 minutes
Temperature Ramp	10°C/minute
Final Oven Temp	250°C
Final Hold Time	0 minutes
Column Flow rate	~ 8 mL/min
Detector B (PID) Temp	280°C
Signal 1 (A)	FID
Signal 2 (B)	PID

APPENDIX E.
CHEMSTATION FILE NAMING CONVENTIONS

ChemStation File Naming Convention

File data, methods, and sequences on ChemStation computers and the LAN using the following naming conventions:

Directories

On the Workstation:

Data: C:\HPCHEM\1\Data\MDDY or D:\HPCHEM\1\Data\MDDYS

Methods: C:\HPCHEM\1\Methods or D:\HPCHEM\1\Methods

Sequences: C:\HPCHEM\1\Sequence or D:\HPCHEM\1\Sequence

For system controlling multiple instruments, 1 may be changed to reflect the instrument number

System running ChemStation versions C & D HPCHEM is named as MSDCHEM

On the LAN:

Data: I:\Room Number\Instrument\Year\MDDYS

Methods: I:\Room Number\Instrument\Methods

Sequences: I:\Room Number\Instrument\Sequence

Methods

MDDYITA

Sequence

MDDYS

Data Files

For GC:

MDDYICSS

For GC/MS

MDDYIQSS

Variables

A: Enter analysis, as follow:

1,4-Dioxane X

504 E

TO15 A

BNA B

BNA-L (SIM) L

Congeners C

P/P P

PCB P

RSK175	R
Soil Gas	A
TPH-G	G
TPH-D	D
VOA	V

C: Channel: A = front
B = back (if applicable)

DD: Day

I: Instrument
6890 series GCs by last number in name: e.g. 6890-1 = 1 except 580-2 = A
All GC/MSs by last letter in name: e.g. 5973L = L

M: Month 1-9, A: October, B: November, C: December

Q: QC type

BFB	F
Blank	B
CV	C
Degradation	P
DFTPP	D
IB	Z
IC	I
LCS	L
LCV	Q
Second Source	S
MS/MSD	M

S: Sequential number 1,2 3,

T: Matrix Type (if applicable)
Water W
Solid S
Air A
Oil O
Other X

Y: Year i.e. 5 for 2005

APPENDIX F.
PREVENTIVE MAINTENANCE REQUIREMENTS

Item	Frequency	Actions/Comments
Gas purifiers (carrier gas & detector gas)	Annually	Replacement schedule is based on capacity and grade of gases. In general, replace non-indicating traps every 6-12 months or when indicating traps start to change color. Replace indicating traps when indicating material is spent.
Flowmeter calibration	2 years	Manual flowmeters only.
Syringes and/or syringe needles	As Needed	Replace syringe if dirt is noticeable in the syringe, if it cannot be cleaned, if the plunger doesn't slide easily, or if clogged. Replace needle if septa wear is abnormal or the needle becomes clogged.
Inlet liner	With each ICAL	Check often. Replace when dirt is visible in the liner or if chromatography is degraded.
Liner O-rings	With each ICAL	Replace with liner or with signs of wear.
Inlet septum	Daily (when analyzing samples)	Check often. Replace when signs of deterioration are visible (gaping holes, fragments in inlet liner, poor chromatography, low column pressure, etc.).
Inlet Hardware	Annually	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Column Maintenance	With each ICAL	Remove 1/2-1 meter from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.).
Solvent rinse	As needed	When chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Replacement	As needed	When trimming and/or solvent rinsing no longer return chromatographic performance.
Ferrules		Replace ferrules when changing columns and inlet/detector parts.
FID Jets & Collector	As needed	Clean when deposits are present. Replace when they become scratched, bent, or damaged, or when having difficulty lighting FID or keeping flame lit.

Item	Frequency	Actions/Comments
Purge/Sample Lines	Annually or as needed	Bake out and purge. Clean with organic free water if necessary.
Trap	As needed	Replace when loss of performance.
PID	Annually or as needed	Clean window

**APPENDIX G.
TYPICAL DATA PACKAGE FORMAT**

Data package contents, in order. Optional sections are shown in *italic text*. Separator pages are underlined.

Draft Report (from LIMS)

Data Package Cover [First numbered page in the data package]

Review Forms

Daily folder review forms or checklists
Other review forms as applicable

Tracking Forms

Work Order(s)
COC(s)

Sample Preparation (for projects that require extraction or digestion)

Bench Sheets (and extraction logs, where used)
Sample cleanup data and records (e.g., GPC logs)
Moisture data as applicable
Analysis matrix (for organics)

[Analysis Method] Data (For each method where multiple methods in package)

Bench sheet(s) where not used in Sample Preparation section
Sequence logs and instrument or other data as applicable, in run order and grouped by day.

Alternatively, separate calibration and sample data as:

Initial Calibration Data

Sample Data

Miscellaneous Data

Other data as applicable (e.g., conductivity for perchlorate)

Standard Records

Standards records from LIMS (and logbook pages as needed)

**USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA**

STANDARD OPERATING PROCEDURE 290

**EXTRACTION OF SOIL SAMPLES USING
PRESSURIZED FLUID EXTRACTION**

Signature & Title:

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Date

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Date

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Date

STANDARD OPERATING PROCEDURE 290

**EXTRACTION OF SOIL SAMPLES USING
PRESSURIZED FLUID EXTRACTION**

This SOP was prepared by ICF Consulting for the United States Environmental Protection Agency (USEPA) under the Environmental Services Assistance Team (ESAT) contract (EPA contract No. 68-W-01-028).

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ESAT Document Control Number: B0104013-3347

STANDARD OPERATING PROCEDURE 290

**EXTRACTION OF SOIL SAMPLES USING
PRESSURIZED FLUID EXTRACTION**

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STANDARD OPERATING PROCEDURE 290

EXTRACTION OF SOIL SAMPLES USING PRESSURIZED FLUID EXTRACTION

1 SCOPE AND APPLICATION

This SOP describes the procedures for the extraction of organochlorine pesticides, PCBs, semivolatile organic compounds (including 1,4-dioxane), and hydrocarbon fuels or oils from soils, sediments, sludges, and solid wastes by pressurized fluid extraction. This SOP is based on procedures in SW-846 Method 3545, *Pressurized Fluid Extraction*, Revision 0, December 1996.

2 METHOD SUMMARY

A measured weight of sample, approximately 30 g, is mixed with anhydrous sodium sulfate, loaded into the extraction cell, and spiked with surrogates. The cell is placed in the extraction apparatus and heated to the extraction temperature, pressurized with the solvent system, and extracted. The extraction apparatus collects the solvent from the heated extraction cell. The extract is dried, cleaned up, if necessary, and concentrated to the required volume before analysis by GC or GC/MS methods specific for the analytes of interest as summarized below.

Extracts for SVOC determination, including 1,4-dioxane, are prepared for analysis using gel permeation chromatography (GPC) cleanup following Region 9 Laboratory SOP 260. The concentrated extracts are analyzed by gas chromatography/mass spectrometry (GC/MS) according to Region 9 Laboratory SOP 315.

Extracts for organochlorine pesticides and PCBs determination are prepared for analysis using GPC cleanup following procedures in EPA Region 9 Laboratory SOP 260. Extracts are cleaned up using Florisil cartridges unless the project does not require the additional cleanup. The concentrated extracts are analyzed by gas chromatography/electron capture detector (GC/ECD) using EPA Region 9 Laboratory SOP 330. Extracts may be prepared for the analysis of PCBs only, which excludes the GPC cleanup step and includes an acid cleanup prior to analysis by EPA Region 9 Laboratory SOP 335.

Extracts for extractable petroleum hydrocarbons are dried with sodium sulfate and concentrated to 3.0 mL. The concentrated extracts are analyzed by GC with a flame ionization detector according to EPA Region 9 Laboratory SOP 385.

3 DEFINITIONS

- 3.1 Laboratory Control Sample (LCS) - An aliquot of reagent water, sand, or sodium sulfate to which known quantities of the method analytes are added. The LCS is treated exactly as a sample. The LCS is used to determine whether the methodology is in control and to indicate the accuracy associated with laboratory procedures. This is equivalent to a laboratory fortified blank QC sample.
- 3.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) - Two aliquots of the same sample to which known quantities of the method analytes are added. The MS and MSD are treated exactly as samples. The MS and MSD are used to determine whether the sample matrix contributes bias to sample results and to measure the precision associated with laboratory procedures. These are equivalent to laboratory fortified matrix and laboratory fortified matrix duplicate QC samples.
- 3.3 Method Blank (MB) - An aliquot of reagent water, sand, or sodium sulfate that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the solvents, or the equipment. This is equivalent to a laboratory reagent blank.
- 3.4 Surrogate - Compounds which are extremely unlikely to be found in any sample that is added to a sample aliquot in a known amount before extraction or other processing, and measured with the same procedures used to measure other sample components. The purpose of the surrogate is to monitor method performance with each sample.
- 3.5 TPH - Total Petroleum Hydrocarbons, also referred to as extractable petroleum hydrocarbons.

4 HEALTH & SAFETY

- 4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be minimized through the use of personal protective equipment and laboratory engineering and design. ESAT personnel should contact the Group Leader or Health and Safety and Environmental Compliance Task Manager and EPA staff should see the Team Leader or the Laboratory Safety, Health and Environmental Compliance Manager if exposure is suspected. Refer to the Material Safety Data Sheets located in the library and the local area network (LAN) for additional information.
- 4.2 Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Stock standard solutions of these compounds must be

prepared in a fume hood. Routine procedures in this SOP do not require contact with concentrated solutions or neat materials. All standard preparation procedures associated with this SOP should be performed in a fume hood wearing protective clothing (lab coats) and safety glasses.

- 4.3 Dichloromethane is a suspected carcinogen. Effects of overexposure: acute inhalation or ingestion causes mild central nervous system depression. The primary toxic effect is narcosis. Other toxic effects are pulmonary edema, encephalopathy, and hemolysis. Dichloromethane irritates the eyes, skin, and respiratory tract. No systemic effects have been reported in humans, although excessive concentrations have caused cancer and liver and kidney damage in animals. Emergency and first aid - Inhalation: immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer cardiopulmonary resuscitation (CPR), contact physician immediately. Eye contact: rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance. Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance. Ingestion: call local poison control center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.
- 4.4 Hexane liquid and vapors are extremely flammable, keep it away from ignition sources. Hexane is harmful if inhaled or swallowed and may cause damage to kidneys, nerves, and respiratory system. It is irritating to skin, eyes, mucous membranes, and is toxic if ingested inhaled. Vapor inhalation causes irritation of nasal and respiratory passages, headache, dizziness, nausea, central nervous system depression. Chronic overexposure can cause severe nerve damage. No systemic toxicity has been reported.
- 4.5 Emergency First Aid - Inhalation: immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer CPR. Contact physician immediately. Eye contact: Rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance. Skin contact: Flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance. Ingestion: Call local Poison Control Center for assistance. Contact physician immediately. Aspiration hazard - do not induce vomiting.
- 4.6 Sulfuric acid - Sulfuric acid is a corrosive poison. Liquid and mist cause severe burns to all body tissues and may be fatal if swallowed or inhaled. Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms of exposure by inhalation may include irritation of the nose and throat, and labored breathing. Do not get acid in eyes, on skin, or on clothing. Skin contact can cause redness, pain, and severe skin burns. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and

shoes. When diluting an acid, the acid should always be added slowly to water and in small amounts. Never use hot water and never add water to the acid. Water added to acid can cause uncontrolled boiling and splashing. Sulfuric acid is incompatible with water, bases, organic material, halogens, metal acetylides, oxides and hydrides, strong oxidizing and reducing agents and many other reactive substances.

- 4.7 Sodium hydroxide - Sodium hydroxide is a corrosive poison and may be fatal if swallowed. It is harmful if inhaled. Effects from inhalation of mist vary from mild irritation to serious damage of the upper respiratory tract. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. When preparing a solution, always add the caustic (pellets or a concentrated solution) to water while stirring; never the reverse. Sodium hydroxide in contact with acids and organic halogen compounds, especially trichloroethylene, may causes violent reactions. Contact with nitromethane and other similar nitro compounds causes formation of shock-sensitive salts. Contact with metals such as aluminum, magnesium, tin, and zinc cause formation of flammable hydrogen gas. Sodium hydroxide, even in fairly dilute solution, reacts readily with various sugars to produce carbon monoxide, a poisonous gas which is odorless and colorless.
- 4.8 Sodium sulfate - May be harmful if swallowed and may be irritating to skin, eyes, and mucous membranes. Get medical assistance for all cases of overexposure. If skin is exposed, wash thoroughly with soap and water. If eyes are exposed, immediately flush with water for at least 15 minutes. For dust inhalation remove to fresh air. For ingestion, if the victim is conscious, have them drink water and induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. Do not heat sodium sulfate in an aluminum as an explosive reaction may occur.
- 4.9 Acetone liquid and vapors are highly flammable. Avoid heat, sparks, open flame, open containers, and poorly ventilated areas when using acetone. Effects of overexposure: acetone is a mild eye and mucous membrane irritant, primary skin irritant, and central nervous system depressant. Acute exposure irritates the eyes and upper respiratory tract. Direct skin contact produces dermatitis, characterized by dryness and erythema through defatting of skin. High concentrations produce narcosis and hypoglycemia. Emergency first aid - Inhalation: immediately remove to fresh air. If the victim is not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer CPR. Contact a physician immediately. In case of eye contact, rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance. Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance. Ingestion: call local poison control center for assistance. Contact a physician immediately. Never induce

vomiting or give anything by mouth to a victim who is unconscious or having convulsions.

5 SAMPLE HANDLING AND PRESERVATION

- 5.1 The extracts are marked with the EPA Region 9 Laboratory number, which can be checked against the tracking sheets and chain-of-custody record to determine the client sample identification, case number, and sample delivery group (SDG) number.
- 5.2 Store samples in the dark in the freezer in Room 406 at $\leq -10^{\circ}\text{C}$. Samples must be extracted within 14 and 7 days for soil and water respectively.
- 5.3 Sample are received in Room 503 and maintained under custody. Remove the samples from the walk-in cooler in Room 503 and fill out the sign-out sheet which is located next to the refrigerator. Take the samples to Room 406 for extraction.
- 5.4 Verify that the following information on the sample containers corresponds to the information on the tracking sheets and the chain-of-custody record. Any discrepancies must be resolved prior to beginning extraction.
 - Client sample ID.
 - Region 9 Laboratory ID.
 - Case number.
 - Sample Delivery Group (SDG) number.
- 5.5 Verify that the integrity of the samples has not been compromised by checking the samples for the following items. Any problems should be noted in the extraction logbook and LIMS and a comment made in the report.
 - Broken chain-of-custody seals on the sample containers.
 - Leaking or broken sample containers.
 - Altered sample information on the sample containers.
- 5.6 Sort samples by date sampled so that samples can be analyzed chronologically according to date sampled (not date received) to prevent exceeding the extraction holding time.
- 5.7 Extract samples within 14 days of the time of sampling. If this requirement is not met, the data must be flagged and the EPA Chemistry Team Leader notified.
- 5.8 Excess samples and empty sample containers must be returned to Room 503 and replaced in their original location. Record the return of the samples in the sample custody log.

5.9 Store sample extracts in the following locations and conditions:

Analysis	Location
SVOCs and 1,4-dioxane	Room 406 at $\leq -10^{\circ}\text{C}$
Pesticides/PCB and PCBs only	Room 400 at $4 \pm 2^{\circ}\text{C}$
Pesticides/PCB and PCBs only (reserved volume)	Room 400 at $4 \pm 2^{\circ}\text{C}$
TPH/E	Room 406 at $4 \pm 2^{\circ}\text{C}$

5.10 Store extracts following analysis and submission of the data deliverables for an SDG for 90 days from the extraction date before segregating for disposal.

5.11 Sample extracts for analysis are received from the extraction lab personnel and custody is transferred to the GC/MS laboratory staff. The GC/MS analyst acknowledges the receipt of the sample extracts by signing the appropriate sections of the completed LIMS bench sheet. Copies of tracking sheets, chain-of-custody records, and the original LIMS bench sheet extraction should accompany the sample extracts.

6 INTERFERENCES

- 6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to anomalous peaks or elevated baselines in gas chromatograms.
- 6.2 Phthalate esters are commonly used as plasticizers and are easily extracted from plastic materials. Contact of samples, solvents, reagents, glassware, extracts, or other sample processing apparatus with plastics must be avoided.
- 6.3 Baseline interference has been observed from excess sodium sulfate added to soil samples and from ASE frit contamination. Minimal amounts of sodium sulfate should be added to soil samples placed on the ASE to minimize contamination. The ASE frits should be processed through the ASE, in addition to three sonication cleanups (refer to Appendix C).

7 APPARATUS AND MATERIALS

7.1 Equipment and supplies

- 7.1.1 Automated pressurized fluid extractor - Dionex ASE 200 Accelerated Solvent Extractor, or equivalent, with 22 and 33 mL size extraction cells. Cells must be

made of stainless steel or other material capable of withstanding the pressure requirements (1750+ psi) necessary for this procedure.

7.1.2 Analytical balance - capable of weighing accurately to " 0.001 g, (Mettler top loading balance, or equivalent).

7.1.3 Drying oven - capable of reaching 105EC

7.1.4 Aluminum weighing boats.

7.1.5 Nitrogen evaporation device - with the capability of temperature control in a heated dry media bath (N-EVAP, Organomation Model 111, or equivalent).

7.1.6 Apparatus for eluting Florisil cartridges

7.1.6.1 Vacuum manifold system to include glass chamber with vacuum valve and top plate with flow control valves. Restek Resprep™-12T Catalog No. 24001 or equivalent.

7.1.6.2 Florisil cartridges - 1 g cartridges with stainless steel or Teflon frits. Supelco ENVI-FLORISIL Catalog No. 57053 or equivalent.

7.2 Glassware and Incidentals

- Beakers – 400 mL.
- Spatula - stainless steel.
- pH meter
- Pasteur pipettes - disposable.
- Pipette, disposable - 1.0 mL, 5.0 mL, 10.0 mL.
- Extraction cells – 22 mL and 33 mL, complete with end caps
- Collection vial – 40 mL and 60 mL
- Glass vial with screw cap tops – 1 mL, 5 mL, 10 mL.
- Graduated cylinder – 1000 mL.
- Syringes, gastight - 1.0 mL, 2.5 mL, 5.0 mL, 10.0 mL.
- Glass funnel - 10 cm ID, long stem
- Sand, seawashed or equivalent

Note: Before use, heat sand at 400°C for 1 hour, cool in a desiccator, and store in a glass bottle.

7.3 Reagents and Standards

7.3.1 Document the receipt and preparation of all standards in the Element database. All standards must be maintained in the freezer at #-10EC and protected from

light. CAUTION: Analysts must allow all standard solutions to equilibrate to room temperature before use.

7.3.2 Sodium sulfate - granular, anhydrous, 10-60 mesh, reagent grade.

Note: Before use, heat at 400EC for 4 hours, or at 120EC for 16 hours, cool in a desiccator, and store in a glass bottle.

7.3.3 Dichloromethane - capillary GC/GC-MS solvent grade and recycled.

Caution: Dichloromethane is a suspected carcinogen. See Health and Safety section for precautions (Section 4.3).

7.3.4 Hexane - capillary GC/GC-MS solvent grade.

Caution: Hexane is extremely flammable. See Health and Safety section for precautions (Section 4.4)

7.3.5 Acetone - capillary GC/GC-MS solvent grade.

Caution: Acetone is highly flammable. See Health and Safety section for precautions (Section 4.9).

7.3.6 Dichloromethane/acetone (1:1 v/v) - Using 1-liter graduated cylinder, measure 1 liter of dichloromethane and transfer to a clean 4-liter glass bottle (solvent container). Use the same 1-liter graduated cylinder and add 1 liter of acetone to the bottle containing the dichloromethane. Cap the bottle and mix well.

7.3.7 Hexane/acetone (4:1 v/v) - Using 1-liter graduated cylinder, measure 1.6 liter of hexane and transfer to a clean 4-liter glass bottle (solvent container). Use the same 1-liter graduated cylinder and add 0.4 liter of acetone to the bottle containing the hexane. Cap the bottle and mix well.

7.3.8 Surrogate Analyte solution - A surrogate analyte solution is added to each field and QC sample prior to extraction. The following table lists the surrogates and the amounts to add for each method.

Analytes	Conc. ($\mu\text{g}/\text{mL}$)	Vol. to add (μL)
OC Pesticides	0.2	1,000
Semivolatile Organics (w/ 1,4-dioxane)	10/100/150	500
1,4-Dioxane	10	500
PCBs (use OC Pest. mix)	0.2	1,000
TPH/E	2,500	60

7.3.9 Matrix Fortifying solution - Use the matrix fortifying solutions listed in the following table to add to each LCS and MS/MSD sample.

Analytes	Conc. (µg/mL)	Vol. to add (µL)
OC Pesticides	0.5/1.0	1,000
Semivolatile Organics with 1,4-dioxane	20/20/80	500
1,4-Dioxane	20	500
PCBs	5	100
TPH/E	2,500	1,000

7.3.10 Nitrogen - 99.999 grade, at 150psig.

7.3.11 Florisil Cartridge Check Solutions

7.3.11.1 A solution of 2,4,5-trichlorophenol in acetone at a concentration of 0.1 µg/mL (Restek 32017-500 or equivalent).

7.3.11.2 Pesticide Mix - A solution of the following pesticides at the indicated concentrations. Used to check Florisil cartridge recovery of analytes. Made from commercial mixture such as Restek Pesticide Standard Mix A. 32003-500 or equivalent.

Compound	Concentration
A-BHC	20 ng/mL
Heptachlor	20 ng/mL
Γ-BHC	20 ng/mL
Endosulfan I	20 ng/mL
Dieldrin	40 ng/mL
Endrin	40 ng/mL
4,4'-DDD	40 ng/mL
4,4'-DDT	40 ng/mL
Methoxychlor	200 ng/mL
Tetrachloro-m-xylene	20 ng/mL
Decachlorobiphenyl	40 ng/mL

8 QUALITY CONTROL

Assessment of QC sample results is performed under EPA Region 9 Laboratory SOPs 315, 330, 335, and 385 where corrective action, which may include re-extraction or isolation of a source of contamination in the extraction laboratory, is defined.

8.1 Method Blank

8.1.1 Method Blanks are used to determine the level of contamination introduced by the laboratory during the extraction procedure. The blanks are subjected to the same extraction and cleanup procedures that are used for samples. One MB is extracted with each group of twenty or fewer samples extracted together.

8.1.2 Acceptance criteria: in general, contamination should be less than one-half the method quantitation limits. Interfering contamination in the blank may warrant re-extraction of the extraction batch.

8.2 Laboratory Control Sample

8.2.1 The LCS is a MB spiked with matrix fortifying solution. The matrix fortifying compounds are used as indicators of extraction efficiency in the absence of matrix interferences. One LCS is extracted with each group of twenty or fewer samples extracted together.

8.2.2 Acceptance criteria: see EPA Region 9 Laboratory SOP 315, 330, 335, or 385 for percent recovery (%R) acceptance limits and corrective actions.

8.3 Matrix Spike Sample Matrix and Matrix Spike Duplicate

8.3.1 MS and MSD analyses provide information about the effect of the sample matrix on sample preparation and measurement. Poor %R results and large RPD between duplicates may indicate inconsistent laboratory technique, sample nonhomogeneity, or matrix effects which may interfere with analysis. A solution of matrix fortifying compounds is spiked into the QC samples designated by the samplers or the sample custodian. A set of MS/MSD samples are prepared with each sample delivery group.

8.3.2 Acceptance criteria: see EPA Region 9 Laboratory SOP 315, 330, 335, or 385 for %R acceptance limits, RPD acceptance limits, and corrective actions.

8.4 Surrogate

8.4.1 Each field or QC sample is fortified with a surrogate solution prior to extraction. Surrogate %R provides information about both the laboratory performance on individual samples and the possible effects of the sample matrix on the analytical results.

8.4.2 Acceptance criteria: see See EPA Region 9 Laboratory SOP 315, 330, 335, or 385 for %R acceptance limits and corrective actions.

8.5 Florisil Cartridge Performance Check

- 8.5.1 Each lot number of Florisil cartridges must be tested before they are used for sample cleanup. Follow procedures outlined in Appendix D.
- 8.5.2 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80-120%, the recovery of 2,4,5-trichlorophenol is less than 5% (compound should be retained on Florisil cartridge), and no peaks interfering with the target analytes are detected.

9 ANALYTICAL PROCEDURES

9.1 Recording Organic Extraction Information

The LIMS must be used as an integral part of the extraction process. The analyst must query the backlog to schedule extractions so that holding times are not exceeded. Samples must be organized into batches and bench sheets completed as the work progresses. It is an unacceptable practice to record data or notes on loose paper for later entry into the LIMS.

9.2 Extraction of Soils by ASE 200

- 9.2.1 Follow procedures in EPA Region 9 Laboratory SOP 150 *Soil and Sediment Homogenization* before weighing an aliquot for extraction.
- 9.2.2 Unless project specific instructions are provided which indicate a smaller aliquot, weigh approximately 30 g (do not exceed 30 g) of the sample into a 400 mL beaker and record the weight to nearest 0.1 g. Do not try to obtain an exact predetermined weight such as 30.0 g.
- 9.2.3 Add enough sodium sulfate to the sample aliquot to absorb the moisture in the sample, usually about 5 g. Stir the mixture until a sandy texture is observed. Use the minimum amount of sodium sulfate required to achieve this texture because the drying agent can cause interferences and may damage the instrument.
- 9.2.4 Caution: The total volume of the sample plus sodium sulfate must not be more than the extraction cell volume. Do not fill the cell with the sample plus sodium sulfate mixture and discard the excess. If the sample volume is greater than the cell volume, discard the sample aliquot and start over.
- 9.2.5 Use a 33 mL cell for 30 g of sample and 5 g of sodium sulfate. Hand-tighten the bottom cell cap onto the cell body. Then insert a disposable cellulose filter in the bottom of the cell. The cellulose filter prevents blockage of the bottom cap's stainless steel frit. Label the extraction cell with the laboratory assigned sample ID, and the type of sample (i.e. MB, LCS, MS/MSD).

- 9.2.6 Transfer the sample into the cell, being careful to keep the threads clean on the cell body and cap.
- 9.2.7 Using a syringe, add the appropriate amount of the required surrogate solution to the sample mixture (Section 7.3.7). If the samples are the designated QC samples, also add the appropriate amount of the required matrix fortifying solution to those samples labeled "MS" and "MSD" and "LCS" (Section 7.3.8).
- 9.2.8 Note: Allow surrogate and matrix spike solutions to come to room temperature before using. Re-mix the solutions by shaking it or agitating with a Vortex mixer. Make sure that the solutions do not contain any precipitate. This is important to ensure acceptable surrogate and spike recovery.
- 9.2.9 Fill any void volume in the cell with an inert material, such as sand. This reduces the amount of solvent used during the extraction. Place a cellulose filter at the top of the cell. Screw the top cap on to the cell body and hand-tighten. Do not use a wrench or other tool to tighten the cap.
- 9.2.10 Prior to loading sample vials onto the ACE, complete the startup procedure provided in Appendix B.
- 9.2.11 Load the tray slots in numerical order with all of the full sample cells, reserving the first slot for an ASE instrument blank. Hang the cells vertically in the tray slots from their top caps.
- 9.2.12 Note: The ASE instrument blank is used solely to prime the ASE and will be discarded. Poor recoveries have been observed consistently with the first ASE run of a batch.
- 9.2.13 Load the rinse tubes into the four open slots, labeled R1 through R4, located between positions 1 and 24, 6 and 7, 12 and 13, and 18, and 19.
- 9.2.14 Note: Check the end of each rinse tube to verify that the O-rings are in place and in good condition. Install or replace if necessary. Do not use a wrench or other tool to tighten the cap.
- 9.2.15 Load a collection vial onto the corresponding vial tray position. Label the vials with laboratory assigned sample number and the type of sample. Load four vials into the rinse slots (labeled R1 through R4).
- 9.2.16 Note: During the extraction process, sensors determine if a vial is present, contains 1 mL of solvent, or is full. Because of this, vial labels must be placed where they do not block areas of the vial read by the sensors. To accomplish this, turn the labels in toward the tray. The caps should extend above the tray inserts. Make sure the vial size used matches the size of the loaded sample cells.

9.3 Typical extraction parameters:

Parameter	Condition
Oven temperature:	100EC
Pressure:	1750 psi
Static time:	5 min (after 5 min pre-heat equilibration)
Flush volume:	0.6 times the cell volume
Nitrogen purge:	45 sec at 150 psi

9.3.1 Extraction solvent combinations:

Analytes	Extraction solvent
OC Pesticides/PCBs	Dichloromethane/acetone 1:1 v/v
Semivolatile organics	Dichloromethane/acetone 1:1 v/v
PCBs only	Hexane/acetone 4:1 v/v
TPH/E	Dichloromethane/acetone 1:1 v/v

CAUTION: For best results with very wet samples (e.g., <70% solids), reduce or eliminate the quantity of hydrophilic solvent (acetone) used. If this occurs, a note must be placed in the "Comments" section of both the ASE run log and the LIMS batch record.

IMPORTANT: Make sure that the gas (N₂) supply pressure is 150 psig. The ASE unit may not extract samples reliably with the N₂ supply pressure below 150 psig.

9.3.2 Begin the extraction according to the manufacturer's instructions.

9.3.3 Each sample extraction requires 45 minutes. Allow the extracts to cool after the extractions are complete. Sample extract volumes collected from the ASE are typically 20-30 mL.

9.3.4 Discard the samples into laboratory solid waste container. Clean the cells according to the procedure in Appendix C.

9.4 Drying - residual water must be removed from all sample extracts before proceeding.

9.4.1 Use a long-stemmed funnel to prepare the drying tube. Place a 0.25-0.5 inch plug of glass wool in the top portion of the stem. Add anhydrous sodium sulfate (Na₂SO₄), about 10 grams. For very wet samples, use more anhydrous sodium sulfate (Na₂SO₄).

- 9.4.2 Rinse a prepared drying tube with 5 mL of same solvent mixture as used for the extraction (see Section 9.3.1). Discard the rinsate in the waste solvent container.
- 9.4.3 Use a 40-mL vial for extract collection. Quantitatively transfer the extract to the drying tube. Rinse the drying tube with two 3 mL portions of the final solvent (dichloromethane or hexane, as defined in Section 9.3.1). Collect the sample extract and the rinses.

9.5 Concentration

- 9.5.1 Adjust the dry media bath temperature to about 2-5°C below the lower of the two boiling points of the extraction solvents (see table below); measure the dry media bath temperature where the vials are placed in the sand bath. Set the vial with the sample in the sand bed in a secure position. At least 1" of the sample vial should be below the surface of the sand. Place the needle inside the vial without contacting the solvent or the vial itself.

Solvents	Boiling Point, °C	Bath Temp, °C
Dichloromethane/ acetone	39 56	34-37
Hexane/ acetone	69 56	51-54

- 9.5.2 Verify that the valves on the N-EVAP manifold are off and the needle valve controlling the nitrogen flow to the N-EVAP manifold is off. Open the nitrogen valve on the left side of the hood and control the gas flow going into the tube using the needle valve and the valve on the N-EVAP manifold. Maintain a gentle nitrogen flow and monitor it frequently to ensure the solvent does not splash and that the tube does not go dry.
- 9.5.3 Evaporate the extract volume to about 1 mL. The predominant solvent at this point will be the solvent with the higher boiling point (i.e., acetone for semivolatiles and organochlorine pesticides; hexane for PCBs).
- 9.5.4 The next concentration step varies by class of contaminants:
- 9.5.4.1 For organochlorine pesticides and semivolatile organics, add about 20 mL of dichloromethane. Evaporate the extract to 9 mL or less. Using a clean 10.0 mL syringe, draw all the extract from the vial. Rinse the internal wall of the vial three times with approximately 0.2 mL dichloromethane, using a Pasteur pipette to add solvent to the vial. Draw the rinsates into the syringe with the extract.

9.5.4.2 For PCBs, using a clean 5.0 mL or 10 mL syringe, draw all the extract from the vial. Rinse the internal wall of the vial three times with approximately 1.0 mL of hexane, using a Pasteur pipette to add solvent to the vial. Draw the rinsates into the syringe with the extract.

9.5.4.3 For TPH/E, add about 10 mL of dichloromethane. Evaporate the extract to 2 mL or less. Using a clean 5.0 mL syringe, draw all the extract from the vial. Rinse the internal wall of the vial three times with approximately 0.2 mL dichloromethane, using a Pasteur pipette to add solvent to the vial. Draw the rinsates into the syringe with the extract.

9.5.5 Transfer a small volume of the appropriate solvent (as indicated in table below), approximately 1.2 mL for PCBs and 0.5 mL for the other analytes, to the vial. Adjust the volume in the syringe to the final volume listed in the table below.

Analyte	Solvent	Evaporation Volume, mL	Final Volume, mL
OC Pesticides	Dichloromethane	9	10.0
Semivolatile Organics	Dichloromethane	9	10.0
PCB's	Hexane	1	5.0
TPH/E	Dichloromethane	2	3.0

NOTE: IF THE VOLUME IN THE SYRINGE ACCIDENTALLY EXCEEDS THE VOLUME, RETURN THE EXTRACT TO THE VIAL AND BEGIN THE EVAPORATION PROCESS AGAIN.

9.5.6 Turn off the N-EVAP manifold valve.

9.5.7 The TPH/E extracts are now ready for analysis. Transfer the extracts to a Teflon screw-cap labeled with the laboratory sample number and the analysis. Store at 4±2°C prior to analysis. The other extracts are now ready for cleanup and subsequent concentration and solvent exchange.

9.6 Extract Cleanup

The following table lists the cleanup procedures used for each of the extraction procedures.

Analyte	Cleanup	Procedure
OC Pesticides	GPC; Florisil as required	SOP 260, then Section 9.6.1
Semivolatile Organics	GPC	SOP 260, then Section 9.6.2
PCBs	Acid	Section 9.6.3
TPH/E	None	

9.6.1 Organochlorine Pesticide Cleanup

9.6.1.1 For OC Pesticides, concentrate the GPC extract to approximately 20 mL. Solvent exchange to hexane by adding approximately 40 mL of hexane and concentrating to 3 mL then use the syringe technique described in Section 9.5.3 to adjust final volume to 5.0 mL. The extract is now ready for Florisil cartridge cleanup if required.

Determine if the extracts may require Florisil cleanup by inspecting the color and clarity of the extracts. If the extracts are clear and colorless, analyze a typical extract. If no chromatographic interferences are present, request approval from the EPA Chemistry Team Leader to analyze the samples without Florisil cleanup. If the samples are colored or if interferences are present, proceed with Florisil cleanup.

9.6.1.2 Florisil cartridge clean up of pesticide /PCB extracts.

Place one Florisil cartridge into a valve on the vacuum manifold for each sample extract. Place a waste collection container below each Florisil cartridge.

Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (4:1) v/v. Pass at least 6 mL of the hexane/acetone solution through the cartridge. Use vacuum to elute the cartridges if necessary. Allow most of the solvent to pass through the cartridge filter and close the valve when there is a thin layer of solvent above the top of the cartridge filter. Remove the waste collection container and discard the rinsate in appropriate waste container.

DO NOT ALLOW THE CARTRIDGE FILTERS TO GO DRY AFTER THEY HAVE BEEN WASHED. If the cartridge filters go dry before the addition of the extract, discard them and begin again. If they go dry after the extract has been added, make a note in the LIMS bench sheet and contact the organics group leader.

Place labeled vials inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside of the appropriate vial as the manifold top is replaced.

Transfer a 2.0 mL aliquot of each field or QC sample extract to the top frit of the appropriate Florisil cartridge. (Reserve 3.0 mLs of the sample extract in the original sample extract container and store refrigerated in Room 406.) Open the valve; allow most of the extract aliquot to pass through the cartridge filter and close the valve when there is a thin layer of

solution above the top of the cartridge filter. In an analogous manner, rinse the syringe with two 0.5 mL portions of hexane that have passed through the Florisil cartridge and collect in the labeled sample vials to complete the quantitative transfer. Close the valve beneath the cartridge.

Open the valve beneath the Florisil cartridge and elute the pesticides and PCBs in the extracts from the cartridge with at least 20 mL of hexane/acetone (4:1 v/v). Use vacuum to elute the cartridges if necessary.

Concentrate the extract to the same 2.0 mL aliquot volume as was taken for cleanup using nitrogen blow down (Section 9.5). Measure the final volume using the syringe technique in Section 9.5.4. Record the final volume as 10 mL.

9.6.2 Semivolatiles Final Concentration

For semivolatiles, concentrate the GPC extract to approximately 0.4 mL. The 0.4 mL is drawn into a clean 0.5 mL or 1.0 mL syringe and this volume is then used to rinse the sides of the concentrator tube. The extract is again drawn into the syringe. A 75 μ L aliquot of dichloromethane is used for the final rinse of the concentrator tube. Adjust the volume to 0.5 mL using the syringe technique described in Section 9.5.4.

Note: Do not allow the concentrator tube to go dry during the concentration step. The tube should be cool as it is rinsed down or analyte loss will result. Adjust final volume very carefully as a small error will result in a large analytical error.

9.6.3 Acid Cleanup for PCB Extracts

9.6.3.1 Take the 5.0 mL aliquot extract (now in hexane). In a 40 mL vial, very slowly add 10 mL of concentrated sulfuric acid.

9.6.3.2 Vortex 1 minute (vortex must be visible in the extract). Allow phases to separate at 5 to 10 minutes. The hexane layer (the top) should be colorless and not have any visible emulsion or cloudiness. If the extract fails these criteria complete Sections 9.6.3.2.3 and 9.6.3.2.4. If it passes the criteria proceed to Section 9.6.3.2.5.

9.6.3.3 Draw off the hexane extract (top layer) and pipet into a clean 40 mL vial. Discard the bottom layer in acid waste container.

9.6.3.4 Repeat Sections 9.6.3.2.1 and 9.6.3.2.2. If the extract still fails the criteria, seek assistance from the extractables group leader.

9.6.3.5 Transfer the hexane layer (the top) to a Teflon-sealed screw-cap bottle. Label the bottle with the laboratory assigned sample number and the type of sample (PCB) and store at 4E" 2EC in Room 406. The extract is now ready for analysis for PCBs.

9.7 Percent Solids Determination

Follow EPA Reg. 9 Laboratory SOP 460 for the determination of solids. Include both the logbook page with the recorded weights and the spreadsheet with the copy of the extraction and ASE logbooks delivered to the analyst. Percent solids results are recorded in the LIMS and used for draft and final report generation.

10 DOCUMENTATION

10.1 Bench Sheet

Complete a LIMS bench sheet and for each extraction batch. Submit one copy to the analyst with the sample extracts. This copy is included in the data package. Make sure all information requested on the bench sheet is completed fully (ie. dates, amounts, initials, and comments) and that the page has been peer reviewed before delivering to the analyst.

10.2 ASE logbook

Complete one page for each extraction batch. Make sure all information requested on the sample extraction form is completed fully (ie., EPA numbers, laboratory IDs, case, SDG , dates, amounts, initials, etc.). Submit one peer-reviewed copy to the analyst with the sample extracts. This copy is included in the data package.

10.3 Solids Determination Logbook

Complete documentation as required in EPA Region 9 Laboratory SOP 460 and submit a peer reviewed copy to the analyst with the sample extracts.

10.4 Maintenance Logbook

Maintain a maintenance logbook for each instrument. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control. Document all preventive or routine maintenance performed, as well as repairs or corrective or remedial actions in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control*.

11 REFERENCE

1. EPA Method 3500B, *Organic Extraction and Sample Preparation*, Revision 2, Dec. 1996.
2. EPA Method 3545, *Pressurized Fluid Extraction*, SW-846, Revision 0, December 1996.
3. EPA Method 3620B, *Florisil Cleanup*, SW-846, Revision 2, December 1996.
4. EPA Method 3665A, *Sulfuric Acid / Permanganate Cleanup*, SW-846, Revision 1, December 1996.
5. EPA Method 8270C, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, Revision 3, December 1996.
6. EPA Region 9 Laboratory SOP 150, *Soil and Sediment Homogenization*
7. EPA Region 9 Laboratory SOP 212, *Extraction of Water Samples by Liquid-Liquid Extraction*.
8. EPA Region 9 Laboratory SOP 222, *Extraction of Organic Compounds from Drinking Water*.
9. EPA Region 9 Laboratory SOP 260, *Gel-Permeation Chromatography (GPC) Clean-Up*
10. EPA Region 9 Laboratory SOP 315, *Semivolatile Organics Analysis*.
11. EPA Region 9 Laboratory SOP 330, *Pesticides & PCB (Water, Sediment, Soil) by GC*.
12. EPA Region 9 Laboratory SOP 335, *Polychlorinated Biphenyls (PCBs) by GC*.
13. EPA Region 9 Laboratory SOP 385, *Extractable Petroleum Hydrocarbons by GC/FID*.
14. EPA Region 9 Laboratory SOP 805 *Refrigerator Temperature Monitoring*
15. EPA Region 9 Laboratory SOP 805, *Refrigerator Temperature Monitoring*.
16. EPA Region 9 Laboratory SOP 840 *Notebook Documentation and Control*
17. *Operator's Manual for ASE 200 Accelerated Solvent Extractor*, Dionex Corporation, Document No. 031149, Revision 03, March 1997.

APPENDIX A
DEVIATIONS FROM THE REFERENCE METHOD

1. SOP 290 does not include a requirement to grind samples to less than 10 mesh.

APPENDIX B
START-UP PROCESS FOR ASE 200

- 1 Check the in coming nitrogen gas pressure. The pressure gauge should be approximately 150 PSI. If it is not, adjust the gas flow before continuing. If the pressure will not reach 150 PSI, check the gas tank – it may be empty.
- 2 Check the 3 regulators inside the ASE solvent cabinet. They are labeled:
 - a Solvent bottle (10 PSI)
 - b System Air (50 PSI)
 - c Compression Oven (140 PSI)

There are knobs to adjust the regulators inside the instrument located against the back panel. Adjust each regulator to the appropriate pressure before continuing (to adjust, pull knobs out and then turn).

- 3 Load 4 clean empty rinse vials in locations R1-R4.
- 4 Push Rinse button and check for the following:
 - a The solvent arm comes out and picks up rinse tube.
 - b Vial tray rotates to appropriate location.
 - c The vial door swings out and solvent needles lower into (and through) vial top.
 - d Pump is working (distinctive clicking sound) and solvent is going into rinse vial.
- 5 Check Hydrocarbon Sensor:
- 6 Pressure Test: These steps will ensure that the instrument is able to reach and maintain cell pressure. On the display screen:
 - a. Go to Main menu and select “7”, the Diagnostic menu.
 - b. Push “.”(dot) three times (located on front of instrument just to the left of “Enter” button). A box should open up in upper right corner of display screen.
 - c. Enter “9137” in box. This should open Service Diagnostic Menu.
 - d. Select “8”, the Manual Control menu.

ATTENTION: The following steps must be done in this order.

- a. Select Cell number “25” and push “Enter”.
- b. Select Vial number “27” and push “Enter”.
- c. Go to A/S IN/OUT use the “Select” button and select OUT and press “Enter” button.
- d. Go to A/S UP/DN, use the “Select” button and select UP and press “Enter”button.
- e. Go to NEEDLE, use “Select” button and select DOWN and press “Enter” button.

- f. Go to PRESSURE: and enter “3000” and then press “Enter”.

At this point the pump should come on briefly and the pressure display (just to right of where you entered 3000) should read approximately 3000 PSI. Run the instrument for a few minutes and **if it does not hold pressure, then there is a problem with the instrument and it should not be run.**

WARNING: Before you leave “Manual Control menu” you must do the following:

- a. Go to PRESSURE: and enter “A” and then press “Enter”.
- b. Go to STATIC: and use “Select” button to open (“O”) valve and press “Enter” button. Leave valve open for several minutes. **Use “Select” button to close (“C”) valve and press “Enter” button before continuing.**
- c. Go to PURGE: and use “Select” button to open (“O”) valve and press “Enter” button. Leave valve open for ~10 seconds. **Use “Select” button to close (“C”) valve and press “Enter” button before continuing.**
- d. Go to NEEDLE, use “Select” button and select UP and press “Enter” button.
- e. Go to A/S UP/DN, use the “Select” button and select DOWN and press “Enter” button.
- f. Go to A/S IN/OUT use the “Select” button and select IN and press “Enter” button.

If all of the above criteria are met, then the ASE 200 instrument is ready to run samples. Press the “Menu” button several times to get to the Main menu.

APPENDIX C
CLEANING PROCEDURE FOR ASE EXTRACTION CELLS

1. Unscrew an end cap from the extraction cell body and remove the extracted soil/ Na_2SO_4 . Discard the soil/ Na_2SO_4 into a laboratory solid waste container.
2. Unscrew the other end cap. Send the extraction cell body to glass washing to be cleaned.
3. Remove and discard the cellulose filters from the end cap.
4. Disassemble the end cap.
 - A. Insert the pointed ends of the snap ring tool into the two holes in the snap ring and squeeze the handles of the tool together to release the tension on the ring.
 - B. While continuing to squeeze the handles, pull the ring out of the cap.
 - C. After the snap ring is out, carefully release the handles of the tool and remove the ring from the tool.
 - D. Remove the cap insert by inverting the end cap and striking it on the bench top.
5. Remove the stainless steel frit.
 - A. Clean by sonicating in a beaker with 50:50 V/V DCM/Hexane for 5 min. Add enough solvent to completely cover the frits.
 - B. Pour off and discard solvent.
 - C. Rinse frits with DCM/Hexane. Discard rinsate.
 - D. Add solvent. Sonicate a second time for 5 min.
 - E. Pour off and discard solvent.
 - F. Rinse frits with DCM/Hexane. Discard rinsate.
 - G. Add solvent and sonicate a third time for 5 min.
 - H. Pour off and discard solvent.
 - I. Put the frits in a 33-mL extraction cell. Extract using ASE, dichloromethane/acetone 1:1 v/v.
 - J. Put frits in 105EC oven for 1 hour.
 - K. Store clean dry frits in a clean glass jar with a Teflon-lined screw-cap lid until ready to assemble for sample extraction.
6. Send end cap, cap insert with PEEK seal, and snap ring to glass washing to be cleaned.
7. When extraction cell bodies and end cap parts come back from glass washing, rinse them with solvent, air dry in fumehood, and put them in 105EC oven for 1 hour.
8. Assemble the end cap.
 - A. Check the end cap for the white O-ring. Replace if missing or damaged.
 - B. Center a frit in the groove at the bottom of the end cap.
 - C. Inspect the cap insert and PEEK seal. If PEEK seal has come off during washing, put it back, making sure that the grooves in the PEEK seal are correctly placed. If

the PEEK seal is deeply grooved - about ½ the height of the seal - discard it and replace it with a new one.

- D. Align the pins in the cap insert with the grooves in the end cap and place the cap insert, with the PEEK seal facing down, into the end cap. Make sure that the cap insert is seated in the bottom of the end cap; you will not be able to install the snap ring if the cap insert is not seated correctly. If you can't seat the cap insert, make sure that the frit is still centered in the groove at the bottom of the end cap.
 - E. Set the end cap upright on clean foil on the bench.
 - F. Insert the snap ring tool into the holes on a snap ring. Do not allow the pointed ends of the snap ring tool to protrude too far past the holes in the snap ring otherwise you will not be able to place the snap ring in the groove in the end cap. Squeeze the tool handles to bring the ends of the snap ring together.
 - G. Insert the snap ring into the groove in the end cap. After making sure the entire ring is in the groove in the end cap, slowly release the tension on the tool and remove the tool from the ring.
9. This completes the cleaning of the ASE extraction cells.

APPENDIX D
FLORISIL CARTRIDGE PERFORMANCE CHECK

1. Each lot number of Florisil cartridges must be tested before they are used for sample cleanup.
2. Add 0.5 mL of 2,4,5-trichlorophenol solution (0.1 µg/mL in acetone) and 0.5 mL of the Pesticide Mix at 20/40/200 ng/mL in hexane (See Section 7.3.10) to 4 mL of hexane. Reduce the final volume to 0.5 mL using nitrogen.
3. Place the mixture onto the top of a washed Florisil cartridge, and elute it with 18 mL of hexane/acetone 4:1 v/v. Use two additional 1 mL hexane rinses to ensure quantitative transfer of standard from the cartridge. Reduce the final volume to 1.0 mL using nitrogen.
4. Analyze the solution by GC/ECD using at least one of the GC columns specified for sample analysis. Determine the recovery of each analyte for evaluation and reporting purposes. Calculate the percent recovery using the following equation:

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Quantity determined by analysis

Q_a = Quantity added

6. The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 120 percent, the recovery of 2,4,5-trichlorophenol is less than 5 percent (compound should be retained on Florisil cartridge), and no peaks interfering with the target analytes are detected.

**USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA**

STANDARD OPERATING PROCEDURE 385

EXTRACTABLE PETROLEUM HYDROCARBONS BY GC/FID

Revision 3

Effective Date: March 28, 2005

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1 PURPOSE AND APPLICABILITY

This method describes the procedures used to analyze dichloromethane extracts for total petroleum hydrocarbons (TPH). Gas chromatography (GC) with a flame ionization detector (FID) is used for the quantitative and qualitative determination of hydrocarbons. Water samples are prepared using SOP 275 *Extraction of Water Samples by Continuous Liquid-Liquid Extraction*. Solid samples are prepared using SOP 290 *Extraction of Soil Samples Using Pressurized Fluid Extraction*.

This method is applicable to the determination of TPH as diesel and TPH as motor oil and in extracts prepared from solid or liquid samples. The method may also be used to determine kerosene (jet fuel) in these matrices. This SOP is based on procedures contained in EPA SW-846 method 8015B, Revision 2, December 1996. Deviations from the reference method are described in Appendix A.

Quantitation limits are provided in Appendix B by matrix and analyte.

2 SUMMARY

Sample extracts, which have been fortified with surrogate analytes, are injected into a GC with FID. Sample components are separated in a fused-silica capillary GC column during temperature programming and detected by the FID.

The fuel of interest is quantitated by comparing its area sum response over the retention time range which it elutes to the area sum response of a fuel standard analyzed under the same conditions as the sample. Probable identification of fuels in samples is done by comparing the chromatographic pattern generated by analysis of the sample to the chromatographic pattern of fuels analyzed under the same conditions as the standard. The identification of specific fuel types may be complicated by environmental processes such as evaporation, biodegradation, or the presence of more than one fuel type.

3 DEFINITIONS

FID - Flame Ionization Detector.

Laboratory Control Sample (LCS) - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine if the methodology is in control, and if the laboratory is capable of making accurate and precise measurements. The LCS is also known as a blank spike (BS).

LIMS - Laboratory Information Management System. The Element database.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD) - Two aliquots of the same environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are treated exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results and to indicate the precision associated with laboratory procedures. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for background concentrations.

Method Blank (MB) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit (MDL) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

Second Source Verification (SCV) - A solution of method analytes of known concentrations which are used to prepare mid level standard(s). The SCV solution is obtained from a source different from the source of calibration standards. It is used to check the accuracy of the initial calibration solutions.

Quantitation Limit (QL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The QL is the concentration of the lowest non-zero standard in the calibration curve. Sample QLs are highly matrix-dependent.

Quantitation Limit Standard (QLS) - The lowest level CAL solution. The QLS is used to verify analytical system response at the quantitation limit.

Surrogate Analyte (SA) - A pure analyte which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in a known amount before extraction or other processing, and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

Stock Standard Solution (SSS) - A concentrated solution containing one or more method analytes purchased from a reputable commercial source.

Total Petroleum Hydrocarbons (TPH).

4 HEALTH & SAFETY

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation must be performed in a well-vented laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN for additional information.

4.1.1 Dichloromethane

Dichloromethane is a suspected carcinogen. Effects of overexposure: acute inhalation or ingestion causes mild central nervous system depression. The primary toxic effect is narcosis. Other toxic effects are pulmonary edema, encephalopathy, and hemolysis. Dichloromethane irritates the eyes, skin, and respiratory tract. No systemic effects have been reported in humans, although excessive concentrations have caused cancer and liver and kidney damage in animals. Emergency and first aid - Inhalation: immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer cardiopulmonary resuscitation (CPR). Contact physician immediately. Eye contact: flush with water continuously for 15 minutes. Get emergency medical assistance. Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothes and shoes. Get emergency medical assistance. Ingestion: call local poison control center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

4.1.2 Acetone

Acetone liquid and vapors are highly flammable. Avoid heat, sparks, open flame, open containers, and poor ventilation. Effects of overexposure: Acetone is a mild eye and mucous membrane irritant, primary skin irritant, and central nervous system depressant. Acute exposure irritates the eyes and upper respiratory tract. Direct skin contact produces dermatitis, characterized by dryness and erythema through defatting of skin. High concentrations produce narcosis and hypoglycemia. Emergency first aid - Inhalation: immediately

remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer CPR. Contact physician immediately. Eye contact: flush with water continuously for 15 minutes. Get emergency medical assistance. Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothes and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance. Ingestion: call local poison control center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must either be capped or have a column connected at all times.

4.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Pollution Prevention Plan* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the volume of any reagent or standard described in Sections 7.2 or 7.3 so long as good laboratory practices are adhered to regarding the accuracy and precision of the glassware, syringes, and/or analytical balances used to prepare the solution. Reducing the concentration of a reagent is not allowed under this procedure because the impact of such a change on the chemistry of the procedure must be assessed prior to implementation.

Reduce the toxicity of waste by purchasing lower concentration stock standards, lower concentration stock reagents, and solutions to replace neat chemicals whenever

possible. However, do not change the concentrations of standards and reagents specifically designated in this SOP

4.4 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedure* and City of Richmond Discharge Permit. Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or their designees.

This procedure produces the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-regulated Waste	Not applicable
Sample Extracts	Hazardous Waste	See solvent, diesel fuel and motor oil MSDs

5 SAMPLE HANDLING AND PRESERVATION

5.1 Internal Chain-of-Custody

- Sample extracts for GC analysis are received from the extraction lab personnel and custody transferred to the GC laboratory staff by signing the appropriate sections in the extraction logbook. Copies of tracking sheets, chain-of-custody records, extraction logbook pages, and moisture determination records should accompany the sample extracts.
- The extracts are marked with Region 9 Laboratory numbers and checked against the tracking sheets and chain-of-custody record to determine the client sample number, case number, and Sample Delivery Group (SDG) number.

5.2 Sample Extract Storage

- Store sample extracts in the refrigerator in Room 400 maintained at > 0°C to 6°C

prior to analysis. Sample extracts must be analyzed within 40 days of extraction. Maintain a refrigerator temperature log daily. Report deviations following U.S. Environmental Protection Agency Region 9 SOP 805, *Refrigerator Temperature Monitoring*.

- Following analysis and reporting, the extracts must be stored under refrigeration for an additional 60 days before segregating for disposal. The sample results and preparation information are used to determine proper disposal.

6 INTERFERENCES

Chromatographic interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to anomalous peaks or elevated baselines in chromatograms, or by carryover when low concentration extracts are analyzed after high concentration extracts.

6.1 Extract contaminants

- Phthalate esters are commonly used as plasticizers and are easily extracted from plastic materials. Avoid contacting samples, solvents, reagents, glassware, extracts, or other sample processing apparatus with plastic materials.

6.2 Carryover

- Interfering contamination may occur when a sample containing low analyte concentrations is analyzed immediately after a sample containing relatively high analyte concentrations. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed. After analysis of a sample containing high analyte concentrations, a laboratory instrument blank should be analyzed to ensure that accurate values are obtained for the next sample.
- Interfering contamination may occur when a sample containing oil range hydrocarbons, especially with carbon numbers exceeding C₄₀, is analyzed. After analysis of a sample containing oil range hydrocarbons, a laboratory instrument blank should be analyzed to ensure that accurate values are obtained for the next sample. The column may need to be heated to an elevated temperature, not exceeding the column limit, until the baseline returns to previous levels. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis.

Minor deviations may be made in specific apparatus and materials provided that they are documented and equivalency is maintained.

7.1 Instrumentation

- Gas chromatograph with FID detector and splitless injection port (Agilent 6890, or equivalent).
- Fused Silica Capillary Gas Chromatography Column -- Any capillary column with a phase ratio (β) of about 265 that provides adequate resolution and capacity may be used. The column used for method validation was 15M x 0.32 mm x 0.1 μ m Rtx-1.
- Data Acquisition and Processing System -- Able to control the GC and to acquire, store, and process gas chromatographic data. The software must be able to calculate calibration factors and the concentrations of analytes in samples. Agilent Technologies EnviroQuant ChemStation software and data acquisition computers (or equivalent).

7.2 Reagents

- Acetone - capillary GC/GC-MS solvent grade.
Caution: Acetone liquid and vapors are highly flammable. See Section 4.1.1 for precautions.
- Dichloromethane - recycled or capillary GC/GC-MS solvent grade.

Caution: Dichloromethane is a suspected carcinogen. See Section 4.1.2 for precautions.

7.3 Standards

All standards must be entered into the Region 9 laboratory information management system (LIMS).

- Surrogate Spiking Solution - Solution of n-hexacosane ($n\text{-C}_{26}\text{H}_{54}$) in dichloromethane:acetone 2:1 v/v at 2,500 μ g/mL. Prepare from neat n-hexacosane by weighing 125 mg n-hexacosane into a 50 mL volumetric flask, dissolving it in 33 mL of dichloromethane (may require sonication or warming) and diluting to volume with acetone.

- Instrument Blank - Solution of n-hexacosane in dichloromethane at 50 µg/mL. Prepare from the surrogate spiking solution by diluting 1 mL to 50 mL in dichloromethane.
- Stock Standard Solutions - Individual solutions of analytes purchased from commercial suppliers, such as Restek #31258 (XHc Diesel Fuel #2 Composite Standard), or equivalent, or Restek #31256 (XHc Kerosene Composite standard), or equivalent, or Restek #31464 (Motor Oil Composite Standard), or equivalent, or a homologous n-alkane series covering the carbon number range of interest. These solutions are diluted with dichloromethane to make the calibration solutions.

Note: Whenever possible, the instrument should be calibrated using a sample of the fuel or oil that is contaminating the site. The calibration standard should be selected prior to the start of the project in conjunction with the client. A different calibration standard may be required if the fuel type in the sample does not match the calibration standard.

- TPH Matrix Spiking Solution - A solution of the fuel of interest at a concentration of 2,500 µg/mL in acetone. This solution is valid for six months from the date of preparation, or until ongoing QC indicates a problem exists, whichever is sooner.
- Calibration Verification Solution - Equivalent to the mid-point initial calibration solution.
- Quantitation Limit Standard (QLS) - Equivalent to the lowest level calibration standard. The QLS is used to verify instrument response at the quantitation limit.
- Second Source Verification (SCV) - Equivalent to the mid-point initial calibration solution but prepared from a source different from the source of calibration standards. The SCV is used to check the accuracy of the initial calibration solutions.

7.3.1 Calibration Solutions

Prepare TPH-diesel and TPH-motor oil calibration solutions at five concentrations in dichloromethane from stock standard solutions at concentrations of 50,000 µg/mL and surrogate spiking solutions at concentrations of 2,500 µg/mL as shown in the tables below. All solutions are valid for six months from the date of preparation, or until ongoing QC indicates a problem. A standard can also be prepared from a homologous n-alkane series covering the expected carbon number range.

TPH-Diesel Solution	Volume Used, μL	Final Volume, mL	Final Concentration, $\mu\text{g/mL}$
Stock Standard	10	10	50
Surrogate Spike	40	10	10
Stock Standard	30	10	150
Surrogate Spike	100	10	25
Stock Standard	100	10	500
Surrogate Spike	200	10	50
Stock Standard	250	10	1,250
Surrogate Spike	300	10	75
Stock Standard	800	10	4,000
Surrogate Spike	400	10	100

TPH-Motor Oil Solution	Volume Used, μL	Final Volume, μL	Final Concentration, $\mu\text{g/mL}$
Stock Standard	40	10	200
Surrogate Spike	200	10	50
Stock Standard	80	10	400
Surrogate Spike	200	10	50
Stock Standard	200	10	1,000
Surrogate Spike	200	10	50
Stock Standard	800	10	4,000
Surrogate Spike	200	10	50
Stock Standard	2000	10	10,000
Surrogate Spike	200	10	50

As an alternative to purchasing commercially available calibration solutions, standards may be prepared from neat fuels or oils as follows: Determine the density of the hydrocarbon fuel mixture by filling a tared 10 mL volumetric flask to volume with neat fuel at room temperature; record the weight in grams to the nearest 0.1mg. Divide the net weight by 10 to obtain the density in g/mL. Use the experimentally determined density in the following calculations.

Prepare a 4,000 mg/L (nominal) range standard by injecting 5 μL of neat standard per mL of dichloromethane. The actual concentration, in mg/L, will be

5,000 times the density of the neat standard in g/mL. For example, injecting 250 μ L of kerosene into about 49 mL of solvent in a 50 mL volumetric flask, then adding additional solvent to volume, would result in a 3,910 mg/L standard assuming a density of 0.782 g/mL for kerosene.

If the neat standard, such as motor oil, is too viscous to measure with a micro liter syringe, weigh out about 200 mg (0.2 g) using an analytical balance and dilute to 50 mL with dichloromethane.

Prepare the other calibration solutions by serially diluting the 4,000 mg/L standard.

7.3.2 Storage of Standard Solutions

Store the unopened ampulated stock standard solutions at $> 0^{\circ}\text{C}$ to 6°C . Store all other working standard solutions in glass bottles or vials with Teflon lined screw caps at $\leq -10^{\circ}\text{C}$ and protect all standards from light. Fresh standards should be prepared every six months, or sooner if comparison with check-standards indicates a problem. The standard solution must be checked frequently for stability. Replace all working standard solutions after six months, or sooner if comparison with SCV samples indicates a problem. CAUTION: Analysts must allow all standard solutions to equilibrate to room temperature before use. Hexacosane has poor solubility at low temperatures. Solutions containing hexacosane must be sonicated before use.

7.4 Supplies

- Volumetric flasks, type A, 100-mL, 50-mL, 25-mL, and 10-mL.
- Microliter syringes (10- μ L, 25- μ L, 50- μ L, 100- μ L, 250- μ L, 500- μ L, and 1-mL).

8 ANALYTICAL PROCEDURES

8.1 Instrument Operation

Set up the instrument operating parameters provided in Appendix D. Adjust as needed to meet method and SOP requirements and chromatographic practice.

Enter data into ChemStation using file naming conventions provided in Appendix E.

Perform a blank column compensation run if necessary after the GC system stabilizes to establish the column bleed background which will be subtracted from all subsequent GC runs. Whenever conditions change or the system becomes contaminated it may be necessary to repeat this step to ensure a flat baseline for reliable integration.

Prior to analyzing calibration, QC, or field samples make a LIMS batch and sequence as required to obtain LIMS assigned IDs for the calibration and QC samples.

8.2 Calibration and Standardization

8.2.1 Initial Calibration

Perform an initial calibration using a minimum of five calibration standards to establish an external standard linear calibration using the average calibration factor. See Section 9.2.1 of this SOP for required frequency and QC limits. Prepare calibration solutions according to Section 7.3.1.

Analyze each of the initial calibration standards and an instrument blank as described in Section 8.3.2. Using the chromatography software, calculate the average calibration factors and %RSD. See 8.3.3 for integration procedures.

8.2.2 Retention Time Windows

Calculate retention time windows when a new GC column is installed or when a new DOC is required for the surrogate on each chromatographic column and instrument. Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. See Section 9.2 for retention time window criteria.

- Record the retention time to three decimal places (e.g., 9.007) for the surrogate from three injections over the course of a 72 hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.
- Calculate the mean and standard deviation of the three absolute retention times for the surrogate using Equation 4. If the standard deviation of the retention times for a target compound is less than 0.01 minutes then use a default standard deviation of 0.01 minutes.
- The width of the retention time window for the surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation is employed, the width of the window will be ± 0.03 minutes.

8.2.3 Secondary Calibration Verification

- Analyze a SCV standard immediately after each initial calibration. See Section 9.2.3 of this SOP for frequency and QC limits.

Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

8.2.4 Calibration Verification

Analyze a calibration verification standard in every 12-hour analytical time period prior to an instrument blank analysis. The calibration verification standard is used to validate the initial calibration standard for the samples run during the associated 12-hour time period. The calibration verification standard concentrations are 500 µg/mL for TPH-diesel and 1,000 µg/mL for TPH-motor oil. See Section 9.2.4 for calibration verification requirements and Appendix C for QC limits.

8.2.5 Quantitation Limit Standard

- Analyze a quantitation limit standard (QLS) each day when analyses of field or QC samples are performed. The QLS is used to verify analytical system response at the quantitation limit. The QLS is 50 µg/mL for TPH-diesel and 200 µg/mL for TPH-motor oil. See Section 9.2.5 for QLS requirements and Appendix C for QC limits.
- If the initial calibration, the SCV, and the IB meet all the criteria specified in Appendix C, the remainder of the 12-hour analytical period may be used for the analysis of field and QC samples using the average CF from the initial calibration to quantitate the data.

8.3 Sample Analysis

8.3.1 Sample Preparation

Samples can be analyzed only after the initial calibration or calibration verification, QLS, MB, and IB meet all of the appropriate criteria specified in Appendix C.

Generate a LIMS batch and sequence as required prior to analyzing QC or field samples to obtain LIMS assigned IDs for the calibration and QC samples.

8.3.2 Analytical Sequence and Analysis

Set up a data acquisition sequence from the LIMS sequence using the GC operating parameters in Appendix D. Identify samples by laboratory sample ID.

Additional header information shall include the dilution factor, instrument ID, and the analyst's initials. Enter this sequence in the instrument run log, if used.

Include all QC sample extracts. See Section 9.3 for batch QC frequency and corrective action requirements. It is highly recommended that the MB, LCS, and MS/MSD extracts be analyzed as early as possible in the analysis of a batch.

8.3.3 Analyte Identification and Quantitation

After completion of analysis, review the chromatogram to identify the fuel in the sample. Compare the chromatographic pattern generated by analysis of the sample to the chromatographic pattern of fuels analyzed under the same conditions as the sample by visually comparing the printed chromatograms or by electronically overlaying the chromatograms, if needed. The fuel and oil ranges contain large number of chemical components which overlap. Use the following table in reporting the fuel and oil ranges:

Report	Chromatogram indicates the presence of:			
	TPH-Diesel Only	TPH-Motor Oil Only	Both	Other components
TPH-Diesel	Quantitate against the TPH-diesel standard and report.	Quantitate the overlap area against the TPH-diesel standard and report the value as "non-detect"*	Manually drop a vertical line from the valley separating the components and report both components.	Quantitate, flag as estimated, and indicate findings in the narrative.
TPH-Motor Oil	Quantitate the overlap area against the oil standard and report the value as "non-detect"*	Quantitate against the oil standard and report.	Manually drop a vertical line from the valley separating the components and report both components.	Quantitate, flag as estimated, and indicate findings in the narrative.

* If the sample concentration is greater than the QL, raise the QL to the value found. If the sample concentration is less than the QL, report as non-detect at the QL.

Review the baseline drawn by the data system integrator to verify that it accurately reflects the area response of the fuel in the sample. If in the judgment of the analyst, it does not then draw a manual baseline from the point where the baseline starts to deviate from the trend to a second inflection point in the chromatogram, or to the end of the chromatogram if there is no second inflection point. See Appendix G for examples. Document any manual integrations following the procedure described in U.S. Environmental

Protection Agency Region 9 SOP 835, *Chromatographic Integration Procedures*.

If only TPH-diesel is present, integrate the retention time over which it elutes and report it. Quantitate the overlap area against the TPH-motor oil standard and report this value as non detected (U). If only TPH-motor oil is present, quantitate and report in like manner, reporting the TPH-motor oil value as calculated and the TPH-diesel overlap as non detected (U).

If both TPH-diesel and TPH-motor oil range components are present manually drop a vertical line from the valley or inflection point separating the two components. Use this retention time as the end RT for TPH-diesel and the beginning RT for TPH-motor oil and quantitate and report both components. If there is no valley or inflection point separating the two components, determine the RT for separating the ranges by overlaying the chromatograms for the TPH-diesel and TPH-motor oil CV standards.

Quantitate the chromatogram using the appropriate initial calibration mean CFs for the identified fuel. If applicable, indicate degree of similarity of sample chromatogram to the fuel to which it is being compared. Print out quantitation reports and chromatograms for each field and QC sample.

- Water calculations

Calculate results for target analytes using Equation 1:

Equation 1:

$$\text{Conc. ug / L} = \frac{A_x \times V_t \times DF}{CF \times V_o}$$

Where:

A_x = area sum response of the sample
 DF = dilution factor
 CF = mean calibration factor from the initial calibration
 V_o = volume of water extracted in Liters
 V_t = volume of concentrated extract in mL

- Soil calculations

Calculate results for target analytes using Equation 2:

Equation 2:

$$\text{Conc. mg/kg (dry weight basis)} = \frac{A_x \times V_t \times DF}{CF \times W \times D}$$

Where:

- A_x = area sum response of the sample
- D = dry weight factor (Percent solids/100)
- W = weight of sample in grams
- CF = mean calibration factor from the initial calibration
- V_t = volume of concentrated extract in mL
- DF = dilution factor

Yields concentration units of $\mu\text{g/g} = \text{mg/kg}$

- Check surrogate recovery for each sample with criteria in Appendix C.
- Dilute and inject a new aliquot of the extract if the on-column concentration of the fuel of interest in any sample exceeds the initial calibration range. Use the following criteria in performing dilutions:

1. Use the results of the original analysis to determine the approximate dilution factor required to get the fuel of interest within the initial calibration range.
2. Do not dilute MS/MSD samples to get either the spiked or non-spiked target compounds within the initial calibration range. If the sample from which the spike aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the report narrative.
3. In the case of extremely contaminated samples several dilutions may be required.
4. Distinguish between the undiluted and diluted analysis by adding a "RE[X]" suffix to the laboratory sample ID on the diluted analysis, where X is a sequential number that identifies the reanalysis.
5. Demonstrate that there is no carryover to subsequent analyses after a sample is analyzed that contains compounds at a level exceeding the initial calibration range of the system. This can be done by analyzing an instrument blank.

Review the results for the sample analyzed immediately after a contaminated sample for all compounds that were in the contaminated

sample that exceeded the limits above. The sample should not contain a concentration above the QL for the target compound that exceeded the limits in the contaminated sample.

6. The most common cause of carryover is hydrocarbon in the oil/asphalt range. This may require cleaning the injection port and baking out the column.

8.3.4 QC Review

As soon as possible after analysis (typically prior to entry into LIMS), inspect sample and QC data for compliance with QC limits in Appendix C. If no significant problems are found, review the following QC data for compliance with SOP requirements:

- Target analyte results must be within range of initial calibration.
- Process and review results of instrument QC (CV, QLS) immediately after their analysis to verify that the results are within QC limits. If the instrument QC results are not within QC limits, stop the sequence and take corrective action before resuming the sequence. See Section 9.2 for instrument QC requirements.
- Process and review the results for the MB, LCS, and MS/MSD batch QC samples and verify that the results are within QC limits. See Section 9.3 for batch QC requirements.
- Check that surrogate compound retention times are within the window specified in Section 9.4. Determine if surrogate recoveries for field and QC samples are within QC limits.
- Review all sample results to determine if any samples need to be re-analyzed at a dilution.
- If a run is rejected for any reason, mark the raw data “Not Used” in large print and document the reason on the quantitation report.

8.3.5 Data Export and LIMS Entry

- Generate epatemp.txt files for field and QC samples by also printing the report to the screen; these files are used by the LIMS DataTool module to import the instrument results into the Data Entry/Review table.
- Copy sample data files from the local drive to the appropriate instrument

data subdirectory on the Region 9 LAN to make them available to LIMS and to archive them.

- Create an empty upload file containing the samples analyzed in the LIMS batch or sequence. Import and merge the data files using the LIMS DataTool module. Load the resulting merged data file into the LIMS Data Entry/Review table. See LIMS manual for detailed procedure.
- Edit dilutions in DataTool or LIMS entry table as needed.
- Review results in the LIMS. Qualify and flag results in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

8.3.6 Instrument Maintenance

The following are suggested remedial actions that may improve method performance; re-calibration may be necessary after most of these actions:

- Check and adjust GC operating conditions and temperature programming parameters.
- Clean or replace the splitless injector liner with a new, silanized liner.
- Break off a short portion of the GC column from the end near the injector, or replace the column. Breaking off a portion of the column will somewhat shorten the analyte retention times.
- Prepare fresh calibration solutions and repeat the initial calibration.
- Replace any components in the GC that permit analytes to come in contact with hot metal surfaces.

The analyst should observe trends in the data such as declining response, erratic relative response, loss of classes of compounds, etc., which may signal the need for instrument maintenance. Document all routine maintenance or corrective actions taken in the maintenance logbook. Preventative maintenance procedures are listed in Appendix E.

The following sections describe possible causes and corrective actions for common problems. Refer to Appendix E for routine preventative maintenance procedures and schedule.

Symptom

- Carryover
Possible causes: Analyzing a sample containing high mole weight components or analyzing high-level and low-level samples sequentially.
Corrective action: As necessary, replace inlet liner, clean inlet, bake out

inlet, bake out column, clip column, replace septum, replace column.

- Shorter retention time.
Possible cause: column flow rate problem.
Corrective action: check flow rate and adjust as necessary.
- Longer retention time and or smaller peaks.
Possible causes: column flow rate problem, injection port leak, or column contamination.
Corrective action: As necessary, check for leaks, replace septum, replace the liner, replace the lower injection port seal, and cut the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.
- Loss of resolution.
Possible causes: column flow rate problem, injection port leak, or column contamination.
Corrective action: Check for leaks, replace septum, replace the liner, replace inlet seal, and clip the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

9 QUALITY CONTROL

9.1 Demonstration of Capability

The EPA Region 9 Laboratory operates a formal quality control program. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated. A summary of the QC Criteria is provided in Appendix C.

A Demonstration of Capability must be in place prior to using an analytical procedure and repeated if there is a change in instrument type, personnel, or method. Follow procedures described in U.S. Environmental Protection Agency Region 9 Laboratory SOP 880 *Demonstration of Laboratory Capability and Analyst Proficiency* for more details.

9.2 Instrument QC

9.2.1 Initial Calibration

Demonstration and documentation of an acceptable initial calibration are

required before any samples are analyzed. The calibration is a five level external standard calibration method.

The GC system must be calibrated whenever corrective action changes instrument response (e.g., detector gas adjustment, column replacement, etc.) is performed or if the calibration verification criteria cannot be met.

- Analyze the initial calibration standards according to Section 8.2.1.
- Obtain area sums for each fuel mixture or homologous n-alkane series over the retention time range during which at least 90% of the material elutes.
- Draw a manual baseline if the baseline drawn by the data system integrator does not accurately reflect the total area response, including the unresolved area that lies below the individual peaks, of the fuel. Draw a manual baseline from the point where the baseline starts to deviate from the trend to a second inflection point in the chromatogram, or to the end of the chromatogram if there is no second inflection point. Manual integrations must conform to U.S. Environmental Protection Agency Region 9 SOP 835, *Chromatographic Integration Procedures*. See Appendix G for example chromatograms.
- The data system calculates the calibration factor (CF) for the target fuel or n-alkane mixture from its area sum response and for the surrogate for all five calibration standards using Equation 3.

Equation 3

$$CF = (A_x) / (C_x)$$

Where

A_x = Area of compound x

C_x = Concentration of the standard injected (μg/mL)

- Calculate the average CF for all analytes.
- Calculate the percent relative standard deviation (%RSD) of the CF values for each compound using Equation 4.

Equation 4

$$\%RSD = (SD / CF_{avg}) \times 100$$

Where SD is calculated as:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - CF_{ave})^2}{n-1}}$$

- Verify that the %RSD of both the target fuel(s) and surrogate are within QC limits immediately after the initial calibration is finished. See Appendix C for QC limits.
- If an ICAL fails because of one standard, a fresh solution of that standard may be re-analyzed and substituted for the failed one in the ICAL. If more than one standard fails, corrective action is required.

9.2.2 Retention time windows

Retention time windows must be established when a new GC column is installed or when a new DOC is required.

- All surrogates in the field and QC samples must fall within the established retention time windows.
- If the surrogate retention time does not fall within the retention time window, evaluate the chromatogram and take corrective action to restore the system if necessary. If repairs to the system are required then a new initial calibration must be performed.

9.2.3 SCV Analysis

Analyze an SCV sample immediately after each initial calibration. See Appendix C for QC limits. If the SCV sample fails it may be repeated once. If the second SCV fails, the cause for failure must be determined and corrected before analysis of samples can proceed.

Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

9.2.4 Calibration Verification

- Analyze a calibration verification standard at the beginning of each 12-hour analytical period and at the end of the 12-hour analytical period. The 12-hour analytical period begins with the injection of the calibration

verification standard and ends with the completion of analysis of the last sample that can be injected within 12 hours of the beginning of the period. Analysis of calibration verification standards, bracketed by instrument blanks, after every ten samples is recommended. The calibration verification standard is used to validate the initial calibration for the samples run during the associated 12-hour time period.

- Analyze the calibration verification standard according to Section 8.2.4.
- Calculate the calibration factor (CF) for the target fuel from its area sum response and for the surrogate compound using Equation 3.
- Calculate the percent difference (%D) between the calibration verification CF and the initial calibration average CF for the target fuel and the surrogate using Equation 5.

Equation 5.

$$\%D = \frac{CF_c - CF_{avg}}{CF_{avg}} \times 100$$

Where:

CF_c = calibration verification CF

CF_{avg} = initial calibration average CF

- The %D must be within QC limits. See Appendix C for QC. If an analyte fails this criterion a second calibration verification may be analyzed. Repeated failure requires that corrective action be taken to restore the system before any additional samples are analyzed. All affected samples must be re-analyzed.

If repairs to the system are required then a new initial calibration must be performed. The analyst should observe trends in the data such as declining response, erratic response, etc., which may signal the need for instrument maintenance.

- Acceptable sample analyses must be bracketed by the analyses of calibration verification standards that meet QC limits.

9.2.5 Quantitation Limit Standard (QLS)

- Analyze a quantitation limit standard (QLS) each day when analyses of field or QC samples are performed. The QLS is used to verify analytical system response at the quantitation limit. The QLS is analyzed at 50 µg/mL of

TPH-diesel and 200 µg/mL for TPH-motor oil.

- Analyze a standard of the fuel of interest at the concentration of the lowest initial calibration level according to Section 8.2.1 of this SOP.
- Calculate the concentration of the target fuel.
- Calculate the percent of true value for the target fuel using Equation 6.

Equation 6:

$$\% \text{ True Value} = (C_d / T_v) \times 100$$

Where:

C_d = Concentration determined by analysis

T_v = True value of standard

- If the %D is not within the QC limits in Appendix C, a second QLS sample may be analyzed. Repeated failure requires that the cause be determined and corrected before analysis of samples can begin. If repairs to the system are required then a new initial calibration must be performed.

9.2.6 Instrument Blank (IB)

- Analyze an instrument blank after the initial calibration or calibration verification is performed and before samples are analyzed. The instrument blank chromatogram and quantitation report must be checked to insure it is within QC limits in Appendix C. It is also important to monitor the chromatographic baseline to insure there are no humps or disruptions which could be integrated as peak area when sample constituents elute on top of them. Surrogate recovery is not evaluated for IB samples. If the instrument blank meets these requirements sample analysis may proceed.

9.3 Batch QC

9.3.1 Method Blank

- A method blank (MB) is extracted and analyzed with each extraction batch or every 20 samples, whichever is more frequent, to demonstrate that the entire analytical system - from extraction through GC analysis - is free of contamination.
- Analyze the MB according to Section 8.
- Evaluate the MB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- Corrective action - If the MB is not acceptable, the source of the contamination must be found and eliminated and the problem documented before analysis can proceed. If re-analysis does not solve the problem, the

batch may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case by case basis.

- If the surrogate recovery does not meet acceptance criteria, re-analyze the extract. If the surrogate recovery still does not meet acceptance criteria, the batch may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case by case basis.

9.3.2 Laboratory Control Sample

- Analyze a laboratory control sample (LCS) to demonstrate that the analytical system is in control. A LCS is extracted and analyzed once per extraction batch or every 20 samples, whichever is more frequent. The LCS is an MB spiked with laboratory fortified matrix solution.
- Analyze a LCS containing the target fuel at a concentration of 2,500 µg/L for water or 50 mg/kg for soil according to Section 8 of this SOP.
- Calculate the percent recovery (%R) using Equation 7.
- The %R must be within the QC limits in Appendix C. If acceptable accuracy cannot be achieved, the problem must be located and corrected prior to reporting any sample data and before additional samples are analyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate

- Laboratory fortified matrix (MS) and duplicate (MSD) samples are extracted and analyzed for each SDG, which typically contain twenty or fewer samples. Matrix QC samples are usually designated in the field. In the event that a sample was not designated as the laboratory fortified matrix spike sample and adequate sample volume exists, the analyst will choose one representative sample from the SDG for QC analysis. The analyst shall not designate any obvious field blanks as the QC sample.
- Analyze the MS/MSD extracts according to Section 8 of this SOP as soon as possible following the analysis of the sample designated as the laboratory fortified matrix sample.
- Calculate the recovery of each compound using Equation 7.

Equation 7:

$$\% \text{ Rec} = ((\text{SSR} - \text{SR})/\text{SA}) \times 100$$

Where,

SSR = Spiked sample result

SR = Sample result

SA = Spike added

- Calculate the relative percent differences (RPD) of the recoveries of each

compound in the MS and MSD using Equation 8.

Equation 8:

$$RPD = \frac{(MSC - MSDC)}{(MSC + MSDC) / 2} \times 100$$

Where,

MSC = Measured concentration of analyte in MS

MSDC = Measured concentration of analyte in MSD

- See Appendix C for QC limits.

The MS/MSD recovery limits are advisory limits only. If the limits are not met, then no further action is required, as long as the LCS is within limits, since the purpose of these analyses is to determine matrix effects on compound recovery. However, frequent failure to meet the recovery or RPD criteria should alert the analyst that a problem may exist and must be investigated. The analyst should analyze the matrix spike solution and check the recoveries of the spike compounds. A new solution should be prepared if the recoveries are not within 20% of expected.

- The table below lists the action to be taken based on the LCS and MS/MSD results.

QC ACCEPTANCE MATRIX+ = PASS - = FAIL								
CASE	1	2	3	4	5	6	7	8
BS - % REC	+	+	+	+	-	-	-	-
MS/MSD -% REC	+	-	+	-	+	-	+	-
MS/LMSD - RPD	+	+	-	-	+	+	-	-

- Case 1: Extraction batch acceptable.
 Case 2: Extraction batch acceptable; matrix effect confirmed.
 Cases 3 & 4: Extraction batch is unsatisfactory. Investigate MS/MSD problem and document findings in report narrative.
 Case 5: Extraction batch rejected. Batch may have to be re-extracted unless LCS problem is determined and documented.
 Cases 6, 7 & 8: Extraction batch rejected. Re-extract batch.

9.4 Sample QC

9.4.1 Surrogate Recovery

- Calculate the surrogate recovery in all field and QC samples immediately after analysis using the following formula:

Equation 9:

$$\%R = (\text{Amount Found}/\text{Amount Spiked}) \times 100.$$

- The surrogate recovery must be within QC limits. See Appendix C for QC limits.
- Take the following steps if surrogate recovery is not within the limits:
 1. Ensure that there are no calculation errors, and check the system performance.
 2. Re-analyze the extract if a system performance problem or calculation error is not evident. The extract may be diluted for re-analysis if examination of the chromatogram so indicates.
 3. If re-analysis of the extract does not solve the problem, the sample may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case by case basis.
- Do not re-extract undiluted samples with surrogate recoveries outside the limits if the diluted analysis with acceptable surrogate recoveries is being submitted. Report the event in the run log.
- Do not re-analyze the MS or MSD samples, even if surrogate recoveries are outside the limits.
- If the sample associated with the MS/MSD analyses does not meet the surrogate recovery criteria, it should be re-analyzed only if the matrix spike and duplicate surrogate recoveries are within the limits. If the sample and spikes show the same pattern (i.e., outside the limits), then the sample does not need re-analysis. The similarity in surrogates recoveries in the sample and spike analyses must be discussed in the report narrative
- If the surrogate recoveries of the re-analysis of the extract are within limits, then:
 1. If the re-analysis was undiluted, the problem was within the laboratory's control. Report the results from the re-analysis and submit the data from both analyses. Distinguish between the analysis and re-analysis by adding a "RE[X]" suffix to the laboratory sample ID on the re-analysis. The problem must be documented in the report narrative.

2. If the re-analysis was diluted, the problem was a matrix effect. Report the results from the re-analysis and submit the data from both analyses and discuss the result in the report narrative. Distinguish between the undiluted and diluted analysis by adding a "RE[X]" suffix to the laboratory sample ID on the diluted analysis. The problem must be documented in the report narrative.
 3. If the surrogate recoveries of the re-extraction are within limits, then the problem was within the laboratory's control. Report the results from the re-extraction. Distinguish between the original analysis and the re-analysis by adding the "RE[X]" suffix to the laboratory sample ID in the re-analysis. The problem must be documented in the report narrative.
- If the re-extraction does not solve the problem, report the results from the first analysis and submit the data from both analyses. Distinguish between the original analysis and the re-analysis by adding the "RE[X]" suffix to the laboratory sample ID in the re-analysis. The problem must be documented in the report narrative.

9.5 Method Performance

Region 9 Laboratory performance for this procedure from January 1, 2003 to February 28, 2005 is summarized in the following table.

Method Performance

Analyte	Matrix	QC Type	Number of Measurements	Mean Recovery, %	95% Confidence Interval (2σ)
TPH-d	water	LCS	46	89.8	70.7-109
TPH-d	solid	LCS	29	87.9	73.9-102
TPH-mo	water	LCS	Insufficient data	-	-
TPH-mo	solid	LCS	Insufficient data	-	-

The following functional areas of the SOP may be significant sources of analytical error:

- Poor extraction efficiency due to specific analyte characteristics or other problems.
- Standard degradation.
- Chromatographic separation and peak integration.

10 DOCUMENTATION

10.1 Standards

All standards (ICAL, ICV/CCV, QL, MS/MSD, and LCS) are recorded in the Element database. A copy of each Analytical Standard Record associated with sample analysis must be included in the data package.

10.2 Analytical sequence

Document the analytical sequence in the Element database and the instrument Run Log.

Record the instrument ID and the LIMS calibration ID for each sequence. Record the Lab number, analysis, container, position, LIMS standard ID, LIMS IS ID as applicable for each field and QC sample in the Element analysis sequence.

Record the Case and SDG number and other run log header information as applicable. Record the data file name, date and time of analysis, analyst initials, laboratory sample IDs, client sample IDs, dilution factors and comments, if any for each field and QC sample in the run log.

10.3 Analytical Report and Data Package

Analytical reports are produced using the Element database. The data package is produced from Element database and manual log records. Appendix F provides the typical format for data package deliverables.

10.4 Maintenance Logbook

Maintain a maintenance logbook for each instrument. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control. Document all preventive or routine maintenance performed, as well as repairs or corrective or remedial actions in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control*.

10.5 SOP Distribution and Acknowledgement

Distribute the approved SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. Document using the SOP Distribution and Acknowledgement List as shown in Appendix H.

11 REFERENCES

Agilent Technologies 6890 Gas Chromatograph Users Manual

Agilent Technologies EnviroQuant ChemStation User's Guide

U.S. Environmental Protection Agency, *Method 8000B, Determinative Chromatographic Separations, Revision 2, December, 1996.*

U.S. Environmental Protection Agency, *Method 8015B, Nonhalogenated Organics Using GC/FID, Revision 2, Dec. 1996.*

U.S. Environmental Protection Agency Region 9 SOP 125, *Disposal Procedures for Unused Aqueous Environmental Samples.*

U.S. Environmental Protection Agency Region 9 SOP 275, *Extraction of Water Samples by Continuous Liquid-Liquid Extraction.*

U.S. Environmental Protection Agency Region 9 SOP 290, *Extraction of Soil Samples Using Pressurized Fluid Extraction.*

U.S. Environmental Protection Agency Region 9 SOP 706, *Laboratory Waste Management Procedures.*

U.S. Environmental Protection Agency Region 9 SOP 805, *Refrigerator Temperature Monitoring.*

U.S. Environmental Protection Agency Region 9 SOP 835, *Chromatographic Integration Procedures.*

U.S. Environmental Protection Agency Region 9 SOP 840, *Notebook Documentation and Control.*

U.S. Environmental Protection Agency Region 9 Laboratory SOP 880, *Demonstration of Capability.*

APPENDIX A.
DEVIATIONS FROM THE REFERENCE METHOD 8015B

1. The reference method reports petroleum hydrocarbons as diesel range organics (DRO) while this SOP reports TPH as diesel. In the SOP, the retention time range for TPH-diesel is established from diesel fuel standards, not the retention time of C₁₀ and C₂₈ alkanes as specified in the reference method. In addition, the SOP extends the chromatographic range of the method to include TPH as motor oil as an analyte.
2. The CF is area/concentration unit (µg/mL) not area/mass (ng) as in the reference method. The formulas for determining sample analyte concentrations have been modified to reflect this change.
3. Control limits for surrogate, LCS, and MS/MSD recoveries are specified in the SOP, not from evaluation of laboratory data.

APPENDIX B.
ANALYTES AND QUANTITATION LIMITS

Hydrocarbon Fuel	QL, on column, $\mu\text{g/mL}$	QL, Solid, mg/kg (30g sample)	QL, Water, $\mu\text{g/L}$ (1 L sample)
Diesel	50	5	250
Oil range	200	20	1,000

**APPENDIX C.
CONTROL MEASURES AND CRITERIA**

QC MEASURE	CRITERIA
Initial Calibration (ICAL)	RSD < 20
Second Source Verification (SCV)	Analyze after ICAL. CF within 30% of mean ICAL CF
Calibration Verification (CCV)	Analyze before QC or field samples and every 12hrs, or more frequently, thereafter. Results: %D ≤ ±15
Quantitation Limit Standard (QLS)	Analyze each day that field or QC samples are analyzed. Result: ± 40% of true value
Method Blank (MB)	Extracted once per extraction batch or every 20 samples, whichever is more frequent. Results must be < ½ QL of target analytes.
Instrument Blank (IB)	< ½ QL of target analytes
Laboratory Control Sample (LCS) fortified with Diesel	Extracted once per extraction batch or every 20 samples, whichever is more frequent. Result: %R between 70 – 130
MS/MSD fortified with Diesel	Extracted once per SDG or every 20 samples, whichever is more frequent. Result: %R between 70 - 130 and RPD ≤ 25
Surrogate Recovery of QC and field samples (except IB)	%R between 70 - 130

APPENDIX D.
RECOMMENDED INSTRUMENT OPERATING PARAMETERS

Instrument: Agilent 6890

Chromatographic column: 15m x 0.32mm ID, 0.1µm film (Restek Rtx-1)

OVEN

Maximum temperature: 350°C
 Equilibration time: 0.50 min.
 Initial temperature: 50°C
 Initial time: 2.00 min.

Ramp:
 Rate 1: 15.00°C/min
 Final temperature 1: 325
 Final time 1: 14.00 min.

INLET

Mode: Pulsed splitless
 Temperature: 320°C
 Pressure: 3.00 psig
 Pulse pressure: 10.0 psig
 Pulse time: 0.30 min
 Purge flow: 60 mL/min.
 Purge time: 0.30 min.
 Gas saver: On
 Gas saver flow: 20.0 mL/min
 Gas saver time: 2.00 min.
 Carrier gas: Helium

COLUMN

Mode: Ramped pressure
 Initial pressure: 3.00 psig
 Initial time: 2.00 min.
 Rate 1: 0.61 psig/min.
 Final pressure 1: 20.00 psig
 Final time 1: 0.13 min.
 Rate 2: 20.00 psig/min.
 Final pressure 2: 30.00 psig
 Final time 2: 5.00 min.
 Nominal initial flow: 1.1 mL/min.
 Average velocity: 21 cm/sec

DETECTOR (FID)

Temperature: 350°C
 Hydrogen flow: 40 mL/min.
 Air flow: 440 mL/min.
 Mode: Constant makeup
 flow
 Makeup flow: 49.0 mL/min
 Makeup gas: Nitrogen

SIGNAL

Signal: Signal - Col Comp
 Data rate: 50 Hz
 Start save time: 1.80 min.
 Stop save time: 30.00 min.
 Column Comp: On

INJECTOR (7673)

Sample washes: 1
 Sample pumps: 3
 Injection volume: 2.0 microliters
 Syringe size: 10 microliters
 PostInj Solvent A washes: 3
 PostInj Solvent B washes: 3
 Viscosity delay: 0 seconds
 Plunger speed: Fast
 Pre Injection dwell: 0.00 min.
 Post Injection dwell: 0.00 min.

APPENDIX E.
CHEMSTATION FILE NAMING CONVENTIONS

ChemStation File Naming Convention

File data, methods, and sequences on ChemStation computers and the LAN using the following naming conventions:

Directories

On the Workstation:

Data: C:\HPCHEM\1\Data\MDDY or D:\HPCHEM\1\Data\MDDYS

Methods: C:\HPCHEM\1\Methods or D:\HPCHEM\1\Methods

Sequences: C:\HPCHEM\1\Sequence or D:\HPCHEM\1\Sequence

For system controlling multiple instruments, 1 may be changed to reflect the instrument number

System running ChemStation versions C & D HPCHEM is named as MSDCHEM

On the LAN:

Data: I:\Room Number\Instrument\Year\MDDYS

Methods: I:\Room Number\Instrument\Methods

Sequences: I:\Room Number\Instrument\Sequence

Methods

MDDYITA

Sequence

MDDYS

Data Files

For GC:

MDDYICSS

For GC/MS

MDDYIQSS

Variables

A: Enter analysis, as follow:

1,4-Dioxane X

504 E

TO15 A

BNA B

BNA-L (SIM) L

Congeners C

P/P P

PCB	P
RSK175	R
Soil Gas	A
TPH-G	G
TPH-D	D
VOA	V

C: Channel: A = front
B = back (if applicable)

DD: Day

I: Instrument
6890 series GCs by last number in name: e.g. 6890-1 = 1 except 580-2 = A
All GC/MSs by last letter in name: e.g. 5973L = L

M: Month 1-9, A: October, B: November, C: December

Q: QC type

BFB	F
Blank	B
CV	C
Degradation	P
DFTPP	D
IB	Z
IC	I
LCS	L
LCV	Q
Second Source	S
MS/MSD	M

S: Sequential number 1,2 3,

T: Matrix Type (if applicable)
Water W
Solid S
Air A
Oil O
Other X

Y: Year i.e. 5 for 2005

**APPENDIX F.
PREVENTIVE MAINTENANCE REQUIREMENTS**

Item	Frequency	Actions/Comments
Gas purifiers (carrier gas & detector gas)	Annually	Replacement schedule is based on capacity and grade of gases. In general, replace non-indicating traps every 6-12 months or when indicating traps start to change color. Replace indicating traps when indicating material is spent.
Split vent trap	Annually	Replace.
Flowmeter calibration	2 years	Manual flowmeters only.
Syringes and/or syringe needles	As Needed	Replace syringe if dirt is noticeable in the syringe, if it cannot be cleaned, if the plunger doesn't slide easily, or if clogged. Replace needle if septa wear is abnormal or the needle becomes clogged.
Inlet liner	With each ICAL	Check often. Replace when dirt is visible in the liner or if chromatography is degraded.
Liner O-rings	With each ICAL	Replace with liner or with signs of wear.
Inlet septum	Daily (when analyzing samples)	Check often. Replace when signs of deterioration are visible (gaping holes, fragments in inlet liner, poor chromatography, low column pressure, etc.).
Inlet Hardware	Annually	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Column Maintenance	With each ICAL	Remove 1/2-1 meter from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.).
Solvent rinse	As needed	When chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Replacement	As needed	When trimming and/or solvent rinsing no longer return chromatographic performance.
Ferrules		Replace ferrules when changing columns and inlet/detector parts.
FID Jets & Collector	As needed	Clean when deposits are present. Replace when they become scratched, bent, or damaged, or when having difficulty lighting FID or keeping flame lit.

**APPENDIX G.
TYPICAL DATA PACKAGE FORMAT**

Data package contents, in order. Optional sections are shown in *italic text*. Separator pages are underlined.

Draft Report (from LIMS)

Data Package Cover [First numbered page in the data package]

Review Forms

Daily folder review forms or checklists
Other review forms as applicable

Tracking Forms

Work Order(s)
COC(s)

Sample Preparation (for projects that require extraction or digestion)

Bench Sheets (and extraction logs, where used)
Sample cleanup data and records (e.g., GPC logs)
Moisture data as applicable
Analysis matrix (for organics)

[Analysis Method] Data (For each method where multiple methods in package)

Bench sheet(s) where not used in Sample Preparation section
Sequence logs and instrument or other data as applicable, in run order and grouped by day.

Alternatively, separate calibration and sample data as:

Initial Calibration Data

Sample Data

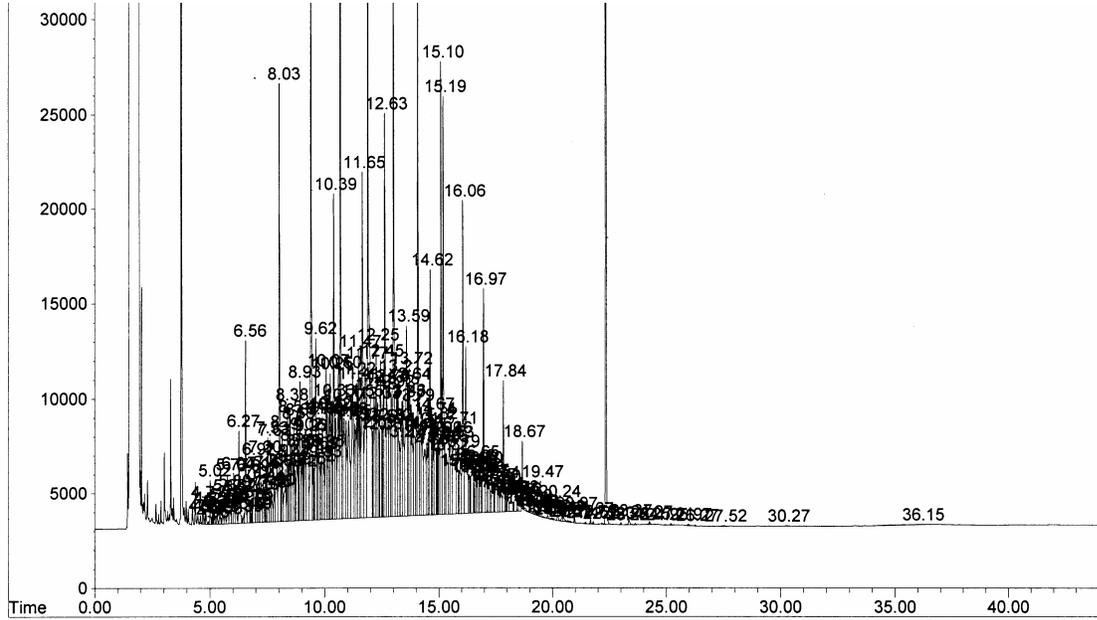
Miscellaneous Data

Other data as applicable (e.g., conductivity for perchlorate)

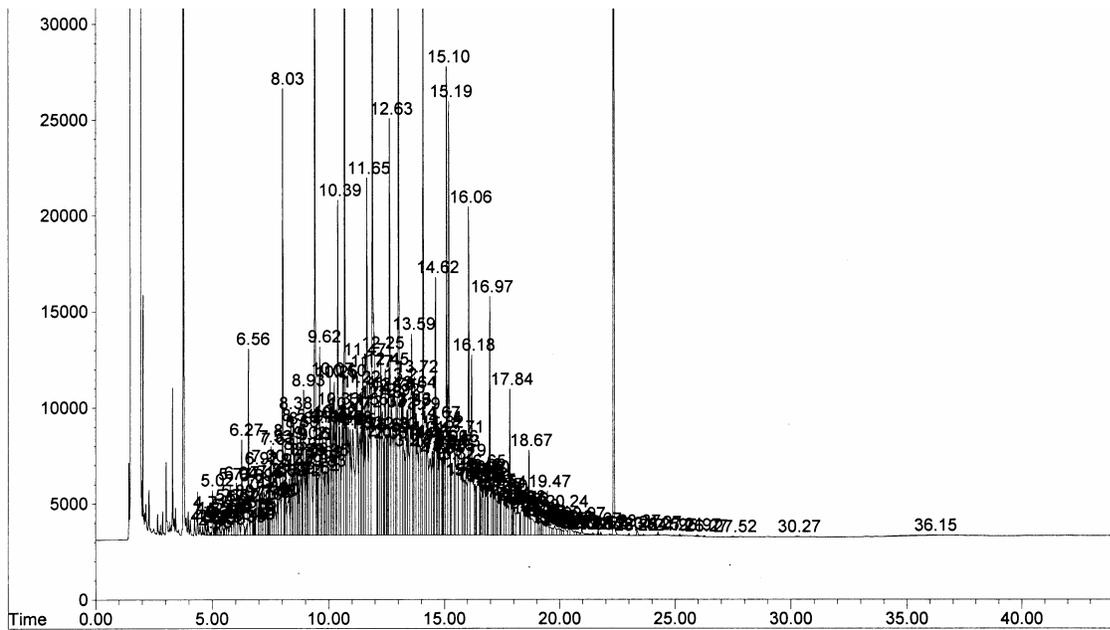
Standard Records

Standards records from LIMS (and logbook pages as needed)

APPENDIX H. INTEGRATION EXAMPLES



INCORRECT BASELINE INTEGRATION



CORRECT BASELINE INTEGRATION

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
TPH - Extractable in Solid (8015B/SOP385)								
Preservation: Store cool at 4°C								
Container: Metal Core Sleeve								
Amount Required: 100 g								
Hold Time: 14 days								
TPH as Diesel	2.5	5.0 mg/kg			70 - 130	25	70 - 130	
TPH as Motor Oil	10	20 mg/kg						
surr: Hexacosane				70 - 130				

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
TPH - Purgeable in Solid (8015B/SOP380)								
Preservation: Store cool at 4°C								
Container: EnCore (5g)								
Amount Required: 5g Encore								
Hold Time: 2 days								
tert-Butyl methyl ether (MTBE)	0.00025	0.00050 mg/kg			65 - 135	25	70 - 130	
Benzene	0.00025	0.00050 mg/kg			65 - 135	25	70 - 130	
Toluene	0.00025	0.00050 mg/kg			65 - 135	25	70 - 130	
Ethylbenzene	0.00025	0.00050 mg/kg			65 - 135	25	70 - 130	
m&p-Xylene	0.00050	0.0010 mg/kg			65 - 135	25	70 - 130	
o-Xylene	0.00025	0.00050 mg/kg			65 - 135	25	70 - 130	
TPH as Gasoline	0.025	0.050 mg/kg			65 - 135	25	70 - 130	
surr: a,a,a-Trifluorotoluene				70 - 130				