



ICF Consulting / Laboratory Data Consultants

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MEMORANDUM

TO: Chris Lichens, Remedial Project Manager
Site Cleanup Section 4, SFD-7-4

THROUGH: Rose Fong, ESAT Task Order Manager (TOM)
Quality Assurance (QA) Program, MTS-3

FROM: Doug Lindelof, Data Review Task Manager
Region 9 Environmental Services Assistance Team (ESAT)

ESAT Contract No.: EP-W-06-041
Technical Direction Form No.: 00105001

DATE: July 6, 2006

SUBJECT: Review of Analytical Data, Tier 3

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Omega Chem OU2
Site Account No.:	09 BC LA02
CERCLIS ID No.:	CAD042245001
Case No.:	33916
SDG No.:	Y1QZ5
Laboratory:	Liberty Analytical Corp. (LIBRTY)
Analysis:	Volatiles
Samples:	20 Groundwater Samples (see Case Summary)
Collection Date:	February 28, 2005 through March 3, 2005
Reviewer:	Kendra DeSantolo, ESAT/Laboratory Data Consultants

This report has been reviewed by the EPA TOPO for the ESAT contract, whose signature appears above.

If there are any questions, please contact Rose Fong (QA Program/EPA) at (415) 972-3812.

Attachment

cc: Cynthia Gurley, CLP PO USEPA Region 4
Steve Remaley, CLP PO USEPA Region 9

CLP PO: Attention Action

SAMPLING ISSUES: Yes No

Data Validation Report

Case No.: 33916
SDG No.: Y1QZ5
Site: Omega Chem OU2
Laboratory: Liberty Analytical Corp. (LIBRTY)
Reviewer: Kendra DeSantolo, ESAT/LDC
Date: July 6, 2006

I. CASE SUMMARY

Sample Information

Samples: Y1QZ5 through Y1QZ9, Y1R00 through Y1R11,
Y1R13 through Y1R15
Concentration and Matrix: Low Concentration Water
Analysis: Volatiles
SOW: OLC03.2
Collection Date: February 28, 2005 through March 3, 2005
Sample Receipt Date: March 2, 3, and 4, 2005
Extraction Date: Not Applicable
Analysis Date: March 3, 4 and 5, 2005

Field QC

Field Blanks (FB): Y1QZ8 and Y1R15
Equipment Blanks (EB): Not Provided
Trip Blanks (TB): Not Provided
Background Samples (BG): Not Provided
Field Duplicates (D1): Y1QZ5 and Y1QZ9
Field Duplicates (D2): Y1R07 and Y1R08

Laboratory QC

Method Blanks & Associated Samples:
VBLKYA: Y1QZ6DL, Y1QZ7, Y1QZ8, Y1QZ9DL, and Y1R01
through Y1R06
VBLKYG: Y1QZ5, Y1QZ6, Y1QZ9, and Y1R00
VBLKYL: Y1QZ5DL, Y1R00DL, Y1R11, Y1R06DL, Y1R07,
Y1R07DL, Y1R08, Y1R08DL, Y1R09, Y1R10, and
Y1R13 through Y1R15
VBLKZK: Storage blank VHBLKZN

Tables

1A: Analytical Results with Qualifications
1B: Data Qualifier Definitions for Organic Data Review
2: Calibration Summary

CLP PO Action

None.

CLP PO Attention

1. Detected results for some analytes are qualified as nondetected and estimated (U,J) due to method blank and field blank contamination (see Comment B).

2. Results for some analytes are qualified as estimated (J) due to calibration problems (see Comments C and D).
3. Results for some analytes are qualified as estimated (J) due to deuterated monitoring compound (DMC) recovery problems (see Comment E).

Sampling Issues

1. Detected results for chloroform in samples Y1QZ7, Y1R01 through Y1R05, Y1R10, and Y1R14 are qualified as nondetected and estimated (U,J) due to field blank contamination (see Comment B).
2. The laboratory indicated in sample log-in sheets that the cooler temperature indicator bottle was absent in coolers received on March 3 and 4, 2005 (see p. 742 and 743 in data package). The cooler temperatures were recorded using an IR gun (see p. 28 and 30 in data package).

Additional Comments

Other than laboratory artifacts (approximate retention times of 4.1, 7.5, 12.0, and 14.6 minutes), tentatively identified compounds (TICs) were found in samples Y1QZ5, Y1QZ6, Y1ZQ9, and Y1R00 (see attached Alkane Narrative Report).

The laboratory performed manual integrations on calibrations and samples due to incorrect auto integration. Manual integrations were reviewed and found to be satisfactory and in compliance with proper integration techniques.

Standard preparation logs are not included in the data package and cannot be evaluated. This information was requested from the laboratory but has not been received to date. Data are not qualified in this report due to missing standard preparation logs. Refer to the attached telephone record log for details.

This report was prepared in accordance with the following documents:

- X ESAT Region 9 Standard Operating Procedure 901, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Volatile and Semivolatile Data Packages*;
- X USEPA Contract Laboratory Program Statement of Work for Analysis of Low Concentration Organic, OLC03.2, December 2000; and
- X USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review, June 2001.

II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

	<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1.	Holding Time/Preservation	Yes	
2.	GC/MS Tune/GC Performance	Yes	
3.	Initial Calibration	Yes	
4.	Continuing Calibration	No	C, D
5.	Laboratory Blanks	No	B
6.	Field Blanks	No	B
7.	Deuterated Monitoring Compounds	No	E
8.	Matrix Spike/Matrix Spike Duplicates	N/A	
9.	Laboratory Control Samples/Duplicates	N/A	
10.	Internal Standards	Yes	
11.	Compound Identification	Yes	
12.	Compound Quantitation	Yes	A, G, H
13.	System Performance	Yes	
14.	Field Duplicate Sample Analysis	No	F

N/A = Not Applicable

III. VALIDITY AND COMMENTS

A. The following results, denoted with an AL@ qualifier, are estimated and flagged AJ@ in Table 1A.

X All detected results below the contract required quantitation limits

Results below the contract required quantitation limits (CRQLs) are considered to be qualitatively acceptable, but quantitatively unreliable, due to the uncertainty in analytical precision near the limit of detection.

B. The following results are qualified as nondetected and estimated due to method blank and field blank contamination and are flagged AU,J@ in Table 1A.

X Chloroform in sample Y1QZ7, Y1R01 through Y1R05, and Y1R14

X cis-1,3-Dichloropropene in samples Y1R07, Y1R09 through Y1R11, and Y1R13 through Y1R15

X Methylene chloride in samples Y1QZ5, Y1QZ6, Y1QZ9, Y1R00, Y1R08 through Y1R11, and Y1R13 through Y1R15

Methylene chloride was found in method blanks VBLKYG and VBLKYL and field blank Y1QZ8, cis-1,3-dichloropropene was found in method blank VBLKYL, and chloroform was found in field blanks Y1QZ8 and Y1R15 (see Table 1A for concentrations). Results for the samples listed above are considered nondetected

and estimated (U,J) and quantitation limits have been raised according to blank qualification rules presented below.

No positive results are reported unless the concentration of the compound in the sample exceeds 10 times the amount in any associated blank for common laboratory contaminants or 5 times the amount for other compounds. If the sample result is greater than the CRQL, the quantitation limit is raised to the sample result and reported as nondetected. If the sample result is less than the CRQL, the result is reported as nondetected at the CRQL.

Chloroform results for samples Y1QZ5, Y1QZ6, Y1QZ9, and Y1R00 are not qualified as nondetected and estimated since their concentrations exceed 5 times the amount in the associated field blanks Y1QZ8 and Y1R15. Trichloroethene results for samples Y1R07 through Y1R10, Y1R13, and Y1R14 are not qualified as nondetected and estimated since their concentrations exceed 5 times the amount in the associated method blank VBLKYL.

The chloroform result for sample Y1R10 (4.2 Φ g/L) is not qualified as nondetected and estimated because a field blank was not collected on 3/2/05. Users should note that chloroform may be an artifact because it was found in field blanks Y1QZ8 and Y1R15.

A laboratory method blank is laboratory reagent water or baked sand analyzed with all reagents, deuterated monitoring compounds, and internal standards and carried through the same sample preparation and analytical procedures as the field samples. The laboratory method blank is used to determine the level of contamination introduced by the laboratory during analysis.

A field blank is clean water prepared as a sample in the field by the sampler and shipped to the laboratory with the samples. A field blank is intended to detect contaminants that may have been introduced in the field, although any laboratory introduced contamination will be present. Contaminants that are found in the field blank which are absent in the laboratory method blank could be indicative of a field QC problem, a deficiency in the bottle preparation procedure, a difference in preparation of the laboratory and field blanks, or other indeterminate error.

C. Results for the following analyte are qualified as estimated due to low relative response factors (RRFs) in continuing calibrations and are flagged "J" in Table 1A.

X Acetone in samples Y1QZ5 through Y1QZ9, and Y1R00 through Y1R06 and method blanks VBLKYA and VBLKYG

RRFs were below the 0.05 validation criterion for acetone in continuing calibrations (see Table 2). Since results are nondetected, false negatives may exist.

The RRF evaluates instrument sensitivity and is used in the quantitation of target analytes.

- D. Results for the following analytes are qualified as estimated due to large percent differences (%Ds) in continuing calibrations and are flagged AJ@ in Table 1A.
- X Methyl acetate in samples Y1QZ5, Y1QZ6, Y1QZ9, Y1R00, Y1R07 through Y1R11, and Y1R13 through Y1R15 and method blanks VBLKYG and VBLKYL
 - X Tetrachloroethene in samples Y1QZ6 through Y1QZ9 and Y1R01 through Y1R06 and method blank VBLKYA
 - X Carbon disulfide, methyl tert-butyl ether, and 1,1-dichloroethane in samples Y1R07 through Y1R11, and Y1R13 through Y1R15 and method blank VBLKYL

%Ds exceeded the $\leq 30.0\%$ validation criterion for the analytes listed above in the March 2, 3, and 4, 2005 continuing calibrations (see Table 2).

The DMC vinyl chloride-d3 also had a %D that exceeded the $\leq 30.0\%$ validation criterion in the 3/2/05 continuing calibration (see Table 2). Quantitation of the analytes associated with this DMC may have been affected by the high %D (see attached Table 9 from the Functional Guidelines).

The continuing calibration checks the instrument performance daily and produces the relative response factors (RRFs) for target analytes that are used for quantitation.

- E. Results for the following analytes are qualified as estimated due to DMC recoveries outside QC limits and are flagged AJ@ in Table 1A.

{Chloroethane-d5}

- X Dichlorodifluoromethane in sample Y1QZ9

{1,1-Dichloroethene-d2}

- X trans-1,2-Dichloroethene and cis-1,2-dichloroethene in sample Y1QZ5, Y1QZ6, Y1QZ7, Y1QZ9, Y1R00, and Y1R06

{Chloroform-d}

- X 1,1-Dichloroethane in samples Y1QZ5, Y1QZ6, Y1QZ9, and Y1R00
- X Chloroform in sample Y1R15

{1,2-Dichloroethane-d4}

- X 1,1-Dichloroethene, 1,1,2-trichloro-1,2,2-trifluoroethane, methyl tert-butyl ether, and carbon tetrachloride in sample Y1R14

The DMC recoveries outside QC limits are shown below.

<u>Sample</u>	<u>DMC</u>	<u>% Recovery</u>	<u>QC Limits</u>
Y1QZ9	Chloroethane-d5	136	60-126
Y1QZ5	1,1-Dichloroethene-d2	2200	65-130
Y1QZ5DL	1,1-Dichloroethene-d2	160	65-130
Y1QZ6	1,1-Dichloroethene-d2	1480	65-130
Y1QZ7	1,1-Dichloroethene-d2	63	65-130
Y1QZ9	1,1-Dichloroethene-d2	2600	65-130
Y1QZ9DL	1,1-Dichloroethene-d2	162	65-130
Y1R00	1,1-Dichloroethene-d2	3400	65-130
Y1R00DL	1,1-Dichloroethene-d2	135	65-130
Y1R06	1,1-Dichloroethene-d2	177	65-130
Y1R09	1,1-Dichloroethene-d2	138	65-130
Y1R05	2-Butanone-d5	186	42-171
Y1QZ5	Chloroform-d	124	80-123
Y1QZ6	Chloroform-d	134	80-123
Y1QZ9	Chloroform-d	148	80-123
Y1R00	Chloroform-d	220	80-123
Y1R15	Chloroform-d	124	80-123
Y1R14	1,2-Dichloroethane-d4	136	78-129
Y1R15	1,2-Dichloroethane-d4	132	78-129
Y1QZ6DL	1,2-Dichloropropane-d6	77	84-123

Detected results for affected analytes where DMC recoveries fell below QC limits may be biased low; where results are nondetected, false negatives may exist.

Detected results for affected analytes where DMC recoveries exceeded QC limits may be biased high. For DMC recoveries that exceeded QC limits, only detected results for associated analytes are qualified. Recoveries for DMCs 2-butanone-d5 in sample Y1R05 and 1,2-dichloroethane-d4 in sample Y1R15 exceeded QC limits but results were not qualified because they were nondetects. The samples were not reanalyzed.

Surrogates (e.g., deuterated monitoring compounds (DMCs)) are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. All samples are spiked with DMCs prior to purging. DMCs provide information about both the laboratory performance on individual samples and the possible effects of the sample matrix on the analytical results.

F. In the analysis of the field duplicate pairs, the following outliers were reported.

<u>Analyte</u>	<u>Y1QZ5 (D1) Conc., µg/L</u>	<u>Y1QZ9 (D1) Conc., µg/L</u>	<u>RPD (<25%)</u>
Trichlorofluoromethane	350	190	60
1,1-Dichloroethene	780	460	52
1,1,2-Trichloro-			

1,2,2-trifluoroethane	820	450	58
Chloroform	51	34	40
	Y1QZ5 (D1)	Y1QZ9 (D1)	
<u>Analyte</u>	<u>Conc., µg/L</u>	<u>Conc., µg/L</u>	<u>RPD (<25%)</u>
Trichloroethene	190	130	38
Tetrachloroethene	620	400	43
	Y1R07 (D2)	Y1R08 (D2)	
<u>Analyte</u>	<u>Conc., µg/L</u>	<u>Conc., µg/L</u>	<u>RPD (<25%)</u>
cis-1,2-Dichloroethene	43	31	32

The effect on data quality is not known.

The analysis of field duplicate samples is a measure of both field and analytical precision. The imprecision in the results of the analysis of the field duplicate pair may be due to the sample matrix or poor sampling or laboratory technique.

- G. Due to high levels of target analytes, samples Y1QZ7, Y1R01, Y1R02, Y1R04, and Y1R09 were analyzed at 12.5-, 16.7-, 2.5-, 12.5-, and 41.6- dilutions, respectively, and samples Y1R06 through Y1R08, Y1R10, and Y1R13 were analyzed at 25-fold dilutions. The CRQLs listed for these samples in Table 1A have been multiplied by the dilution factor.
- H. Samples Y1QZ5, Y1QZ6, Y1QZ9, and Y1R00 were reanalyzed at 50-, 25-, 25-, and 125-fold dilutions, respectively, due to high levels of chloroform, trichlorofluoromethane, 1,1-dichloroethene, 1, 1, 2-trichloro-1, 2, 2-trifluoroethane, trichloroethene, and tetrachloroethene that exceeded the calibration range. Results for these analytes are reported from the diluted analyses in Table 1A; results for other analytes are reported from the undiluted analyses.

Sample Y1R06 was reanalyzed at a 125-fold dilution due to high levels of 1, 1, 2-trichloro-1, 2, 2-trifluoroethane, trichloroethene, and tetrachloroethene that exceeded the calibration range. Results for these analytes are reported from the 125-fold diluted analysis in Table 1A; results for other analytes are reported from the 25-fold diluted analysis.

Samples Y1R07 and Y1R08 were reanalyzed at 41.6-fold dilution due to high levels of trichloroethene that exceeded the calibration range. Results for trichloroethene are reported from the 41.6-fold diluted analyses in Table 1A; results for other analytes are reported from the 25-fold diluted analyses.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR ORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review," January 2005.

- U The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method.
- L Indicates results which fall below the Contract Required Quantitation Limit. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).
- NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.
- R The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.

Table 2
Calibration Summary

Case No.: 33916
 SDG No.: Y1QZ5
 Site: Omega Chem OU2
 Laboratory: Liberty Analytical Corp. (LIBRTY)
 Reviewer: Kendra DeSantolo, ESAT/LDC
 Date: July 6, 2006

RELATIVE RESPONSE FACTORS (RRF)

	<u>RRF</u>	<u>RRF</u>
Analysis date:	3/02/05	3/03/05
Analysis time:	23:09	22:28
GC/MS I.D.:	5972HP71	5972HP71
<u>Analyte</u>	<u>Cont.</u>	<u>Cont.</u>
Acetone	0.039	0.048

PERCENT DIFFERENCES (%D)

	<u>%D</u>	<u>%D</u>	<u>%D</u>
Analysis Date:	3/02/05	3/03/05	3/04/05
Analysis Time:	23:09	22:28	15:34
GC/MS I.D.:	5972HP71	5972HP71	5972HP71
<u>Analyte</u>	<u>Cont.</u>	<u>Cont.</u>	<u>Cont.</u>
Carbon disulfide	-----	-----	-35.0
1,1-Dichloroethane	-----	-----	-32.0
Methyl acetate	-32.3	-----	-38.6
Methyl tert-butyl ether	-----	-----	-34.1
Tetrachloroethene	-----	+33.0	-----
Vinyl chloride-d3	-36.4	-----	-----

- = RRF biased low; + = RRF biased high.

ASSOCIATED SAMPLES AND METHOD BLANKS

Initial 2/23/05: All samples, all method blanks, storage blank VBLKZN
 Cont., 3/02/05 (23:09): Samples Y1QZ5, Y1QZ6, Y1QZ9, Y1R00, method blank VBLKYG
 Cont., 3/03/05 (22:28): Samples Y1QZ6DL, Y1QZ7, Y1QZ8, Y1QZ9DL, Y1R01 through
 Y1R06, method blank VBLKYA
 Cont., 3/04/05 (15:34): Samples Y1QZ5DL, Y1R00DL, Y1R06DL, Y1R07, Y1R07DL,
 Y1R08, Y1R08DL, Y1R09, Y1R10, Y1R11, Y1R13 through Y1R15,
 method blank VBLKYL
 Cont., 3/05/05 (22:16): Storage blank VHBLKZN, method blank VBLKZK.