



ICF Consulting / Laboratory Data Consultants

Environmental Services Assistance Team, Region 9
1337 South 46th Street, Building 201, Richmond, CA 94804-4698
Phone: (510) 412-2300 Fax: (510) 412-2304

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 7, 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.:	33335
SDG No.:	Y1FR9
Laboratory:	Shealy Environmental Services, Inc. (SHEALY)
Analysis:	Semivolatiles
Samples:	18 Groundwater Samples (see Case Summary)
Collection Date:	September 13 through 16, 2004
Reviewer:	April Martinez, ESAT/Laboratory Data Consultants

This report has been reviewed by the EPA TOM 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.: 33335
SDG No.: Y1FR9
Site: Omega Chem OU2
Laboratory: Shealy Environmental Services, Inc. (SHEALY)
Reviewer: April Martinez, ESAT/LDC
Date: July 7, 2006

I. CASE SUMMARY

Sample Information

Samples: Y1FS0 through Y1FS9 and Y1FT1 through Y1FT8
Concentration and Matrix: Low Concentration Water
Analysis: Semivolatiles
SOW: OLC03.2
Collection Date: September 13 through 16, 2004
Sample Receipt Date: September 15 through 17, 2004
Extraction Date: September 16 and 22, 2004
Analysis Date: September 22 and 28, 2004

Field QC

Field Blanks (FB): Not Provided
Equipment Blanks (EB): Not Provided
Background Samples (BG): Not Provided
Field Duplicates (D1): Y1FS1 and Y1FS2
Field Duplicates (D2): Y1FT5 and Y1FT6

Laboratory QC

Method Blanks & Associated Samples:
SBLK71: Y1FT5 through Y1FT8
SBLK89: Y1FS0 through Y1FS9 and Y1FT1 through Y1FT4

Tables

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

CLP PO Action

None.

CLP PO Attention

1. Detected results for di-n-butylphthalate are qualified as nondetected and estimated (U,J) due to method blank contamination (see Comment B).
2. Results for some analytes are qualified as estimated (J) due to calibration problems (see Comments C, D, and E).
3. Results for some analytes are qualified as estimated (J) due to deuterated monitoring compound (DMC) recovery problems (see Comment F).

Sampling Issues

Samples Y1FS9 and Y1FT1 through Y1FT3 were received by the laboratory with a cooler temperature of 6.8EC, which exceeds the 4±2EC sample preservation criterion. Since the cooler temperature is below 10EC, no adverse effect on data quality is expected.

Additional Comments

Other than laboratory artifacts (approximate retention times of 5.0, 8.7, and 12.9 minutes), tentatively identified compounds (TICs) were found in the samples Y1FS0 through Y1FS3, Y1FS5 through Y1FS8, Y1FT1, Y1FT2, Y1FT3, and Y1FT7 (see attached Form 1LCGs).

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.

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 for Organic Analysis, OLC03.2, May 1999; 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	No	C, D
4.	Continuing Calibration	No	C, E
5.	Laboratory Blanks	No	B
6.	Field Blanks	N/A	
7.	Deuterated Monitoring Compounds	No	F
8.	Matrix Spike/Matrix Spike Duplicates	N/A	
9.	Laboratory Control Samples/Duplicates	N/A	
10.	Internal Standards	Yes	
11.	Compound Identification	No	H
12.	Compound Quantitation	No	A, H
13.	System Performance	Yes	
14.	Field Duplicate Sample Analysis	No	G

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 contamination and are flagged AU,J@ in Table 1A.

X Di-n-butylphthalate in samples Y1FS0 through Y1FS4, Y1FS6, Y1FS9, Y1FT2, Y1FT3, Y1FT4, and Y1FT7

Di-n-butylphthalate was found in method blank SBLK89 (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.

Users should note that bis(2-ethylhexyl)phthalate found in samples may be an artifact because it is a common laboratory contaminant.

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.

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

X Atrazine in samples Y1FS0 through Y1FS9 and Y1FT1 through Y1FT4 and method blank SBLK89

An average RRF of 0.022 was reported for atrazine in the 9/18/04 initial calibration. An RRF of 0.025 was reported for atrazine in the 9/22/04 continuing calibration. These values are below the 0.05 validation criterion.

Since atrazine 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 relative standard deviation (%RSD) in initial calibrations and are flagged "J" in Table 1A.

X Benzaldehyde, 2,4-dinitrophenol, and atrazine in samples Y1FS0 through Y1FS9 and Y1FT1 through Y1FT4 and method blank SBLK89

%RSDs of 88.3%, 51.8%, and 28.1% were reported for benzaldehyde, 2,4-dinitrophenol, and atrazine, respectively, in the 09/18/04 initial calibration. These values exceed the #20.5%/50.0% validation criterion.

The initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

- E. Results for the following analyte are qualified as estimated due to a large percent difference (%D) in the continuing calibration and are flagged AJ@ in Table 1A.

X Benzaldehyde in samples Y1FS0 through Y1FS9 and Y1FT1 through Y1FT4 and method blank SBLK89

%D of -59.0% was reported for benzaldehyde in the 09/22/04 continuing calibration. This value exceeds the ∇ 50.0% validation criterion.

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

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

{Dimethylphthalate-d6}

X Caprolactam, 1,1'-biphenyl, dimethylphthalate, diethylphthalate, di-n-butylphthalate, butylbenzylphthalate, bis(2-ethylhexyl)phthalate, and di-n-octylphthalate in samples Y1FS9 and Y1FT5 through Y1FT8

{4,6-Dinitro-2-methylphenol-d2}

X 4,6-Dinitro-2-methylphenol in samples Y1FS0 through Y1FS9, Y1FT1 through Y1FT4, Y1FT7, and Y1FT8 and method blank SBLK89

{Acenaphthylene-d8}

X Naphthalene, 2-methylnaphthalene, 2-chloronaphthalene, acenaphthylene, and acenaphthene in sample Y1FT7

The DMC recoveries outside QC limits are shown below.

<u>Sample</u>	<u>DMC</u>	<u>% Recovery</u>	<u>QC Limits</u>
Y1FS9	Dimethylphthalate-d6	58%	62-102
Y1FT5	Dimethylphthalate-d6	50%	62-102
Y1FT6	Dimethylphthalate-d6	58%	62-102
Y1FT7	Dimethylphthalate-d6	48%	62-102
Y1FT8	Dimethylphthalate-d6	60%	62-102
Y1FT7	Acenaphthylene-d8	48%	49-98
Y1FS0	4,6-Dinitro-2-methylphenol-d2	45%	53-153
Y1FS1	4,6-Dinitro-2-methylphenol-d2	45%	53-153
Y1FS2	4,6-Dinitro-2-methylphenol-d2	40%	53-153
Y1FS3	4,6-Dinitro-2-methylphenol-d2	50%	53-153
Y1FS4	4,6-Dinitro-2-methylphenol-d2	48%	53-153
Y1FS5	4,6-Dinitro-2-methylphenol-d2	43%	53-153
Y1FS6	4,6-Dinitro-2-methylphenol-d2	45%	53-153
Y1FS7	4,6-Dinitro-2-methylphenol-d2	50%	53-153
Y1FS8	4,6-Dinitro-2-methylphenol-d2	43%	53-153
Y1FS9	4,6-Dinitro-2-methylphenol-d2	35%	53-153
Y1FT1	4,6-Dinitro-2-methylphenol-d2	48%	53-153

Y1FT2	4,6-Dinitro-2-methylphenol-d2	43%	53-153
Y1FT3	4,6-Dinitro-2-methylphenol-d2	43%	53-153
Y1FT4	4,6-Dinitro-2-methylphenol-d2	45%	53-153
Y1FT7	4,6-Dinitro-2-methylphenol-d2	48%	53-153
Y1FT8	4,6-Dinitro-2-methylphenol-d2	48%	53-153
SBLK89	4,6-Dinitro-2-methylphenol-d2	48%	53-153
SBLK89RE	4,6-Dinitro-2-methylphenol-d2	45%	53-153

Detected results for affected analytes may be biased low; where results are nondetected, false negatives may exist. 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.

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

<u>Analyte</u>	<u>Y1FS1 (D1) Conc., µg/L</u>	<u>Y1FS2 (D1) Conc., µg/L</u>	<u>RPD (<25%)</u>
bis(2-Ethylhexyl)phthalate	5.6	4.2	29

<u>Analyte</u>	<u>Y1FT5 (D2) Conc., µg/L</u>	<u>Y1FT6 (D2) Conc., µg/L</u>	<u>RPD (<25%)</u>
bis(2-Ethylhexyl)phthalate	32	17	61

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.

H. The laboratory reported detected results for 4-nitrophenol in samples Y1FS0 and Y1FS1 of 1.6 Φg/L (below the CRQL of 5.0 Φg/L). However, the mass spectra do not meet the specified National Functional Guidelines criteria. In the reviewer's professional judgment, 4-nitrophenol in samples Y1FS0 and Y1FS1 should not be reported as detects because the ion m/z 139 is missing in the sample mass spectra (attached, p. 883 and 895 in data package). The peak at retention time of 11.4 minutes is the DMC 4,6-dinitro-2-methylphenol-d2. Results for 4-nitrophenol in samples Y1FS0 and Y1FS1 are reported in Table 1A as nondetected (5.0U).

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 Low Concentration Organic Data Review," June 2001.

- U The analyte was analyzed for, but was not detected above 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; the associated numerical value is the approximate concentration of the analyte in the sample.

- 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 above the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.

- R The sample results are unusable. The analyte may or may not be present in the sample.

