



ICF International / Laboratory Data Consultants

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MEMORANDUM

TO: Lisa Hanusiak, Remedial Project Manager
Site Cleanup Section 3, SFD-7-3

THROUGH: Rose Fong, ESAT Task Order Manager (TOM)
Quality Assurance (QA) Program, PMD-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.: 00105052

DATE: April 4, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Alhambra
Site Account No.:	09 ES LA01
CERCLIS ID No.:	CAD980818579
Case No.:	35903
SDG No.:	Y30D8
Laboratory:	A4 Scientific, Inc. (A4)
Analysis:	Trace Volatiles
Samples:	5 Ground Water Samples (see Case Summary)
Collection Date:	November 20, 2006
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: Ray Flores, CLP PO USEPA Region 6
Steve Remaley, CLP PO USEPA Region 9

CLP PO: Attention Action

SAMPLING ISSUES: Yes No

00105052-7751/35903/Y30D8-V

Data Validation Report

Case No.: 35903
SDG No.: Y30D8
Site: Alhambra
Laboratory: A4 Scientific, Inc.
Reviewer: April Martinez, ESAT/LDC
Date: April 4, 2007

I. CASE SUMMARY

Sample Information

Samples: Y30D8 through Y30E2
Concentration and Matrix: Low Concentration Water
Analysis: Trace Volatiles
SOW: SOM01.1
Collection Date: November 20, 2006
Sample Receipt Date: November 22, 2006
Extraction Date: Not Applicable
Analysis Date: December 2, 2006

Field QC

Field Blanks (FB): Not Provided
Equipment Blanks (EB): Y30E2
Trip Blanks (TB): Not Provided
Background Samples (BG): Not Provided
Field Duplicates (D1): Y30D9 and Y30E0

Laboratory QC

Method Blanks & Associated Samples:
VBLK06: Y30D8 through Y30E2, Y30D8MS, Y30D8MSD
VBLK15: Storage blank VHBLK01

Tables

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

CLP PO Action

Nondetected results for 1,4-dioxane in all samples, all method blanks, and storage blank VHBLK01 are qualified as rejected (R) due to very low response factors (<0.01) in initial and continuing calibrations (see Comment A).

CLP PO Attention

1. Detected results for some analytes are qualified as nondetected and estimated (U,J) due to method blank and equipment blank contamination (see Comment C).
2. Results for some analytes are qualified as estimated (J) due to calibration problems (see Comments 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

1. Detected results for toluene, ethylbenzene, and m,p-xylene are qualified as nondetected and estimated (U,J) due to equipment blank contamination (see Comment C).
2. The laboratory indicated in the sample log-in sheet that the custody seal and cooler temperature indicator bottle was absent from the cooler (see p. 320 in data package). The cooler temperatures were recorded using a thermometer (see SDG Narrative, p. 1 in data package).
3. No sample was designated for Alaboratory QC@ on the traffic report & chain of custody record. The laboratory performed matrix spike/matrix spike duplicate (MS/MSD) analysis on sample Y30D8.

Additional Comments

Other than laboratory and field artifacts (approximate retention times of 3.1, 4.0, 4.5, 5.0, 8.1, 9.2, 12.5, and 14.2 minutes), tentatively identified compounds (TICs) were found in samples Y30E1 and Y30E2 (see attached Form 1Js).

The laboratory performed manual integrations on calibrations 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 Volatile and Semivolatile Data Packages*;
- X USEPA Contract Laboratory Program Statement of Work for Organics Analysis, *Multi-Media, Multi-Concentration*, SOM01.1, May 2005; 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	A, D, E
4.	Continuing Calibration	No	A, D
5.	Laboratory Blanks	No	C
6.	Field Blanks	No	C
7.	Deuterated Monitoring Compounds	No	F
8.	Matrix Spike/Matrix Spike Duplicates	No	G
9.	Laboratory Control Samples/Duplicates	N/A	
10.	Internal Standards	Yes	
11.	Compound Identification	Yes	
12.	Compound Quantitation	Yes	B
13.	System Performance	Yes	
14.	Field Duplicate Sample Analysis	Yes	

N/A = Not Applicable

III. VALIDITY AND COMMENTS

- A. Nondetected results for the following analyte are qualified as rejected due to very low relative response factors (RRFs) in initial and continuing calibrations and are flagged "R" in Table 1A.

X 1,4-Dioxane in all samples, all method blanks, and storage blank VHBLK01

RRFs at or below 0.01 were reported for 1,4-dioxane in initial and continuing calibrations (see Table 2). Since results are nondetected, false negatives may exist.

The DMC 1,4-dioxane-d8 also had RRFs at or below 0.01 in initial and continuing calibrations (see Table 2).

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

- B. 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.

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

X Methylene chloride in all samples and storage blank VHBLK01

- X 1,3-Dichlorobenzene in samples Y30D9, Y30E1, and Y30E2
- X 1,2,4-Trichlorobenzene and 1,2,3-Trichlorobenzene in sample Y30D8
- X Toluene and m,p-xylene in samples Y30E0 and Y30E1
- X Ethylbenzene in sample Y30E1

Methylene chloride, 1,2,4-trichlorobenzene, and 1,2,3-trichlorobenzene were found in all method blanks; 1,3-dichlorobenzene was found in method blank VBLK06; toluene, ethylbenzene, and m,p-xylene were found in equipment blank Y30E2 (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 the 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.

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.

An equipment blank is clean water that has been collected as a sample using decontaminated sampling equipment. The intent of an equipment blank is to monitor for contamination introduced by the sampling activity, although any laboratory introduced contamination will also be present.

- D. Results for the following analyte are qualified as estimated due to low RRFs in initial and continuing calibrations and are flagged "J" in Table 1A.

- X tert-Butyl alcohol in all samples, all method blanks, and storage blank VHBLK01

Average RRFs below 0.05 were reported for tert-butyl alcohol in initial calibrations (see Table 2). RRFs were below the 0.05 validation criterion for tert-butyl alcohol in continuing calibrations (see Table 2). Since results are nondetected, false negatives may exist.

- E. Results for the following analyte are qualified as estimated due to a large percent relative standard deviation (%RSD) in the initial calibration and are flagged "J" in Table 1A.

X Bromomethane in method blank VBLK15 and storage blank VHBLK01

A %RSD of 33.0% was reported for bromomethane in the 12/07/06 initial calibration. This value exceeded the #30.0% 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.

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

{Benzene-d6}

X Benzene in sample Y30E2

{1,2-Dichloropropane-d6}

X Cyclohexane, methylcyclohexane, 1,2-dichloropropane, and bromodichloromethane in samples Y30E1 and Y30E2

DMC recoveries outside QC limits are shown below.

<u>Sample</u>	<u>DMC</u>	<u>% Recovery</u>	<u>QC Limits</u>
Y30D8	Chloroethane-d5	140	71-131
Y30D9	Chloroethane-d5	154	71-131
Y30E0	Chloroethane-d5	150	71-131
Y30D8MS	Benzene-d6	74	77-124
Y30D8MSD	Benzene-d6	73	77-124
Y30E2	Benzene-d6	73	77-124
Y30D8MS	1,2-Dichloropropane-d6	77	79-124
Y30D8MSD	1,2-Dichloropropane-d6	77	79-124
Y30E1	1,2-Dichloropropane-d6	78	79-124
Y30E2	1,2-Dichloropropane-d6	74	79-124

Detected results for affected analytes where DMC recoveries fell below QC limits may be biased low; where results are nondetected, false negatives may exist. Recoveries for the DMC chloroethane-d5 exceeded the QC limit but associated sample 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.

- G. MS/MSD recoveries for benzene, trichloroethene, and toluene in QC samples Y30D8MS and Y30D8MSD did not meet the criteria for accuracy specified in the SOW, as shown below.

<u>Analyte</u>	<u>Y30D8MS % Recovery</u>	<u>Y30D8MSD % Recovery</u>	<u>QC limits % Recovery</u>
Benzene	67	66	76-127
Trichloroethene	69	67	71-120
Toluene	72	70	76-125

Results obtained may indicate poor laboratory technique or matrix effects which may interfere with analysis. Detected results for benzene, trichloroethene, and toluene may be biased low; where results are nondetected, false negatives may exist.

Matrix spike sample analysis provides information about the effect of the sample matrix on sample preparation and measurement.

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.: 35903
 SDG No.: Y30D8
 Site: Alhambra
 Laboratory: A4 Scientific, Inc.
 Reviewer: April Martinez, ESAT/LDC
 Date: April 4, 2007

RELATIVE RESPONSE FACTORS

	<u>RRF</u>	<u>RRF</u>	<u>RRF</u>
Analysis date:	11/29/06	12/02/06	12/02/06
Analysis time:	08:52-	10:25	18:17
GC/MS I.D.:	C-5973	C-5973	C-5973
<u>Analyte</u>	<u>Init.</u>	<u>Cont.</u>	<u>Cont.</u>
1,4-Dioxane	0.008	0.010	0.011
tert-Butyl Alcohol	0.043	0.047	-----
1,4-Dioxane-d8	0.009	0.010	0.012

	<u>RRF</u>	<u>RRF</u>	<u>RRF</u>
Analysis date:	12/7/06	12/07/06	12/07/06
Analysis time:	10:32-	10:58	17:50
GC/MS I.D.:	C-5973	C-5973	C-5973
<u>Analyte</u>	<u>Init.</u>	<u>Cont.</u>	<u>Cont.</u>
1,4-Dioxane	0.011	0.011	0.009
tert-Butyl Alcohol	0.038	0.038	0.033
1,4-Dioxane-d8	0.011	0.008	0.007

ASSOCIATED SAMPLES AND METHOD BLANKS

Initial 11/29/06: All samples, method blank VBLK06
 Cont., 12/02/06 (10:25): All samples, method blank VBLK06
 Cont., 12/02/06 (18:17): All samples, method blank VBLK06

Initial 12/7/06: Storage blank VHBLK01, method blank VBLK15
 Cont., 12/07/06 (10:58): Storage blank VHBLK01, method blank VBLK15
 Cont., 12/07/06 (17:50): Storage blank VHBLK01, method blank VBLK15.