



**ICF Consulting / 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 Project Officer (TOPO)  
Quality Assurance (QA) Program, PMD-3

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

ESAT Contract No.: 68-W-01-028  
Technical Direction Form No.: 00905073

DATE: February 13, 2006

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.:	34815
SDG No.:	Y2916
Laboratory:	A4 Scientific, Inc. (A4)
Analysis:	Semivolatiles
Samples:	13 Water Samples (see Case Summary)
Collection Date:	November 17, 18, 21, and 22, 2005
Reviewer:	Santiago Lee, ESAT/Laboratory Data Consultants (LDC)

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: Ray Flores, CLP PO USEPA Region 6  
Steve Remaley, CLP PO USEPA Region 9

CLP PO:  Attention  Action

SAMPLING ISSUES:  Yes  No



## Data Validation Report

Case No.: 34815  
SDG No.: Y2916  
Site: Alhambra  
Laboratory: A4 Scientific, Inc. (A4)  
Reviewer: Santiago Lee, ESAT/LDC  
Date: February 13, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: Y2915 through Y2927  
Concentration and Matrix: Low Concentration Water  
Analysis: Semivolatiles  
SOW: OLC03.2  
Collection Date: November 17, 18, 21, and 22, 2005  
Sample Receipt Date: November 19, 22, and 23, 2005  
Extraction Date: November  
Analysis Date: December 7 and 8, 2005

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Y2919, Y2921, Y2923, and Y2926  
Background Samples (BG): Not Provided  
Field Duplicates (D1): Y2916 and Y2917

#### Laboratory QC

Method Blanks & Associated Samples:  
SBLK3Y: All samples, Y2926MS, and Y2926MSD

#### Tables

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

#### CLP PO Action

Nondetected results for 4-chloroaniline, hexachlorocyclopentadiene, and 3,3'-dichlorobenzidine in samples Y2918 and Y2927 are qualified as rejected (R) due to very low deuterated monitoring compound (DMC) recoveries (see Comment A).

#### CLP PO Attention

1. Results for di-n-octylphthalate in all samples are qualified as estimated (J) due to a calibration problem (see Comment C).
2. Results for some analytes are qualified as estimated (J) due to DMC recovery problems (see Comment D).
3. The equipment blank Y2926 was used for matrix spike/matrix spike duplicate (MS/MSD) analysis (see Sampling Issues section).

### Sampling Issues

No sample was designated for Alaboratory QC@ on the traffic report & chain of custody records (TR/COCs). The laboratory performed MS/MSD analysis on sample Y2926 (see attached electronic mail dated November 29, 2005; p. 336 in data package). However, Y2926 is an equipment blank, not a field sample. Consequently, MS/MSD results are not meaningful.

### Additional Comments

Other than laboratory and field artifacts (approximate retention times of 3.0, 3.1, 3.2, 3.3, 3.4, 4.0, and 6.7 minutes), semivolatile tentatively identified compounds (TICs) were found in samples Y2918 and Y2920.

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 (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	No	C
4.	Continuing Calibration	Yes	
5.	Laboratory Blanks	Yes	
6.	Field Blanks	Yes	
7.	Deuterated Monitoring Compounds	No	A, D
8.	Matrix Spike/Matrix Spike Duplicates	N/A	E
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 analytes are qualified as rejected due to very low DMC recoveries and are flagged AR@ in Table 1A.

{4-Chloroaniline-d4}

X 4-Chloroaniline, hexachlorocyclopentadiene, and 3,3'-dichlorobenzidine in samples Y2918 and Y2927

Recoveries of 0% and 1% were reported for the DMC 4-chloroaniline-d4 in samples Y2918 and Y2927, respectively, which are well below the QC limit of 8-70%. Since 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.*

- 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. 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 Di-n-octylphthalate in all samples and method blank SBLK3Y

A %RSD of 30.8% was reported for d-n-octylphthalate in the initial calibration. This value exceeds 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.*

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

{4-Methylphenol-d8}

X 2-Methylphenol, 4-methylphenol, and 2,4-dimethylphenol in samples Y2916 and Y2927

{Dimethylphthalate-d6}

X Caprolactam, 1,1'-biphenyl, dimethylphthalate, diethylphthalate, di-n-butylphthalate, butylbenzylphthalate, bis(2-ethylhexyl)phthalate, and di-n-octylphthalate in samples Y2916, Y2919, and Y2923

{Benzo(a)pyrene-d12}

X Benzo(b)fluoranthene, beno(k)fluorothene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, and benzo(g,h,i)perylene in samples Y2916 and Y2927

The DMC recoveries outside QC limits are shown below.

<u>Sample</u>	<u>DMC</u>	<u>% Recovery</u>	<u>QC Limits</u>
Y2916	4-Methylphenol-d8	36	38-95
Y2927	4-Methylphenol-d8	30	38-95
Y2918	4-Chloroaniline-d4	0	8-70
Y2927	4-Chloroaniline-d4	1	8-70
Y2916	Dimethylphthalate-d6	61	62-102
Y2919	Dimethylphthalate-d6	61	62-102
Y2923	Dimethylphthalate-d6	60	62-102
Y2916	Benzo(a)pyrene-d12	52	54-120
Y2927	Benzo(a)pyrene-d12	48	54-120

Since results are nondetected, false negatives may exist. It should be noted that results for analytes associated with very low 4-chloroaniline-d4 recoveries have been qualified as rejected (see Comment A). The samples were not reanalyzed.

- E. The MS/MSD recoveries for 4-nitrophenol (118%/125%) and MSD recovery for pentachlorophenol (105%) in the QC sample Y2926 (an equipment blank) did not meet the criteria for accuracy of 10-80% and 9-103%, respectively, as specified in the SOW. Since Y2926 is an equipment blank, not a field sample, MS/MSD results are not meaningful (see Sampling Issues section).

*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 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.

