



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.: 00905058 Amendment 2

DATE: October 26, 2005

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.:	34502
SDG No.:	MY21C2
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Dissolved Metals and Dissolved Molybdenum by ICP-MS and Dissolved Mercury
Samples:	19 Groundwater Samples (see Case Summary)
Collection Date:	August 22, 23, 24, 26, 29 and 30, 2005
Reviewer:	Stan Kott, 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: Edward Messer, CLP PO USEPA Region 4
Steve Remaley, CLP PO USEPA Region 9

CLP PO: FYI Action

SAMPLING ISSUES: Yes No

Data Validation Report

Case No.: 34502
SDG No.: MY21C2
Site: Alhambra
Laboratory: CompuChem (LIBRTY)
Reviewer: Stan Kott, ESAT/LDC
Date: October 26, 2005

I. CASE SUMMARY

Sample Information

Samples: MY21B9, MY21C2 through MY21C9, MY21D0, MY21D1, MY21D3 through MY21D9, and MY21E0
Concentration and Matrix: Low Concentration Groundwater
Analysis: CLP Dissolved Metals plus Dissolved Molybdenum by ICP-MS and Dissolved Mercury
SOW: ILM05.3 and Modification Reference Number 1256.0
Collection Date: August 22, 23, 24, 26, 29 and 30, 2005
Sample Receipt Date: August 25, 27, 30 and 31, 2005
Preparation Date: September 1, 2005
Analysis Date: September 2 and 22, 2005

Field QC

Field Blanks (FB): MY21C6
Equipment Blanks (EB): MY21D0, MY21D4, MY21D6, and MY21D9
Background Samples (BG): Not Provided
Field Duplicates (D1): MY21C2 and MY21C3
(D2): MY21C4 and MY21C5

Laboratory QC

Method Blanks & Associated Samples: PBW and samples listed above
Matrix Spike: MY21B9S
Duplicates: MY21B9D
ICP Serial Dilution: MY21B9L

Analysis: CLP Dissolved Metals plus Dissolved Molybdenum by ICP-MS and Dissolved Mercury

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
CLP ICP-MS Metals and Molybdenum	September 1, 2005	September 22, 2005
Mercury	September 1, 2005	September 2, 2005
Percent Solids	Not Applicable	Not Applicable

CLP PO Action

None.

Sampling Issues

1. The results for zinc in samples MY21D1, MY21D3, MY21D5, MY21D7, and MY21E0 are qualified and estimated high (J+) because of equipment blank contamination. The reported results of 6.7 µg/L, 2.4 µg/L, and 11.2 µg/L for zinc in equipment blank samples MY21D4, MY21D6, and MY21D9, respectively, exceed the contract required quantitation limit (CRQL) of 2.0 µg/L.
2. The cooler containing samples MY21D5 and MY21D6 arrived at the laboratory with a temperature of 7.9°C. This temperature exceeds the temperature of 4°± 2°C specified in the Statement of Work (SOW). Since the water samples were preserved to a pH less than 2, no adverse effect on the quality of the data is expected.

Additional Comments

Temperature indicator bottles were not found in the sample shipping coolers received at the laboratory. The laboratory used an infrared thermometer to determine the sample temperatures. No adverse effects on data quality are expected.

The laboratory indicated that sample MY21C1 listed on the Traffic Report/Chain of Custody (TR/COC) record form was not found in the shipping cooler and contacted the Sample Management Office (SMO). Region 9 indicated that the sampler provided three sample containers for MY21B9 and that sample MY21C1 was not collected for this SDG. (See attachment.)

The TR/COC record form for samples collected on August 24, 2005 did not have a sampler signature or relinquished by signature and date in the respective form fields. No adverse effect on data quality is expected.

The collection data for samples MY21B9 and MY21C2 through MY21C6 were not provided on a standard EPA CLP TR/COC record form. Station locations for the samples listed above are not provided. The effect on data quality is not known.

The samples of this SDG were analyzed for CLP Dissolved Metals plus dissolved molybdenum by ICP-MS under Modified Analysis Request (MAR), Modification Reference Number 1256.0. Dissolved mercury was analyzed by the CLP cold vapor atomic absorption method.

Analytical results are listed in Table 1A with qualifications. Definitions of data qualifiers used in Table 1A are listed in Table 1B.

This report was prepared in accordance with the following documents:

- X Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- X *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1256.0, Title: MS061505, August 23, 2005;
- X *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration* ILM05.3, March 2004; and
- X *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic*

II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1. Data Completeness	Yes	
2. Sample Preservation and Holding Times	Yes	
3. Calibration	Yes	
a. Initial		
b. Initial and Continuing Calibration Verification		
c. CRQL Check Standard (CRI)		
d. ICP-MS Tuning Analysis		
4. Blanks	No	B,C
5. ICP Interference Check Sample (ICS)	Yes	
6. Laboratory Control Sample (LCS)	Yes	
7. Duplicate Sample Analysis	Yes	
8. Matrix Spike Sample Analysis	No	D
9. ICP Serial Dilution Analysis	Yes	
10. ICP-MS Internal Standards	N/A	
11. Field Duplicate Sample Analysis	No	E
12. Sample Quantitation	Yes	A
13. Overall Assessment	Yes	

N/A = Not Applicable

III. VALIDITY AND COMMENTS

- A. Results above the method detection limit (MDL) but below the CRQL (denoted with an "L" qualifier) are estimated and flagged "J" in Table 1A.

Results above the MDL but below the CRQL are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of quantitation.

- B. The following results are qualified as estimated high and flagged "J+" in Table 1A due to equipment blank contamination.

X Zinc in samples MY21D1, MY21D3, MY21D5, MY21D7, and MY21E0

The results for samples associated with the equipment blanks that are greater than the MDL are qualified as estimated high (J+) unless the concentration of the analyte in the sample exceeds 5 times the amount in any associated blank.

The reported results of 6.7 µg/L, 2.4 µg/L, and 11.2 µg/L for zinc in equipment blank samples MY21D4, MY21D6, and MY21D9, respectively, exceed the 2.0 µg/L CRQL.

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

C. The following results are reported as non-detected in Table 1A due to low level calibration, equipment, and field blank contamination.

- X Antimony in samples MY21B9, MY21C6, MY21C7, MY21D1, MY21D3, MY21D5, and MY21E0
- X Chromium in samples MY21D1, MY21D4, MY21D9, and MY21E0
- X Copper in samples MY21C9, MY21D3, and MY21D5
- X Zinc in samples MY21D0 and MY21D8

The values for antimony in the initial calibration blank (0.66 µg/L) and in continuing calibration blanks CCB1 (0.42 µg/L), CCB2 (0.39 µg/L), CCB3 (0.36 µg/L), and CCB4 (0.37 µg/L) are greater than the MDL but less than the CRQL. Sample results greater than or equal to the MDL but less than the CRQL are reported as non-detected (U) at the CRQL.

The values for chromium (0.20 µg/L) and zinc (0.82 µg/L) in the field blank sample MY21C6 are greater than the MDL but less than the CRQL. Sample results greater than or equal to the MDL but less than the CRQL are reported as non-detected (U) at the CRQL.

The copper values of 0.29 µg/L, 0.33 µg/L, and 0.35 µg/L in equipment blank samples MY21D0, MY21D4, and MY21D6, respectively, are greater than the MDL but less than the CRQL. The results for samples associated with these equipment blanks that are greater than or equal to the MDL but less than the CRQL are reported as non-detected (U) at the CRQL.

An initial calibration blank (ICB) consists of deionized, distilled water and reagents. It is analyzed at the beginning of each analytical run, immediately after the initial calibration verification (ICV) standard to monitor analyte carry-over.

A continuing calibration blank (CCB) consists of deionized, distilled water and reagents. It is analyzed after the continuing calibration verification (CCV) standard, at a frequency of every 10 samples and at the end of the analytical run to monitor analyte carry-over.

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

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. Contaminants that are found in the field blank which are absent in the laboratory preparation 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 source of contamination.

D. The following results are estimated and flagged "J" or "UJ" in Table 1A because matrix spike recovery results are outside method QC limits.

- X Silver in samples MY21B9, MY21C2 through MY21C5, MY21C7, MY21C8, MY21C9, MY21D1, MY21D3, MY21D5, MY1D7, MY21D8, and MY21E0

Matrix spike recoveries for silver in QC sample MY21B9S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for silver is presented below and is based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Silver	46	-54

Results above the MDL are considered quantitatively uncertain. Results reported for silver in the samples listed above may be biased low and, where non-detected, false negatives may exist.

According to the inorganic SOW, when the pre-digestion spike recovery results for ICP-MS analytes fall outside the control limits of 75-125%, a post-digestion spike must be performed for those elements that do not meet the specified criteria. The following post-digestion spike recovery result was obtained.

MY21B9A	
Analyte	Post-Digestion Spike, % Recovery
Silver	99

Since the post-digestion spike recovery was acceptable, the low pre-digestion spike recovery result (46%) obtained for silver may indicate poor laboratory technique or matrix effects which may interfere with accurate analysis, depressing the analytical result.

The matrix spike sample analysis provides information about the effect of the sample matrix on the digestion and measurement methodology.

- E. Absolute differences of 5.5, 2.0, and 6.9 were obtained for copper, nickel, and zinc in the analysis of field duplicate pair samples MY21C4 and MY21C5. These absolute differences exceed the respective CRQLs for copper (2.0 µg/L), nickel (1.0 µg/L), and zinc (2.0 µg/L). Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a ≤ 20 RPD or \leq CRQL criteria for precision. 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, high levels of solids in the sample, or poor sampling or laboratory technique.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared in accordance with the document *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, October 2004.

- U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+ The result is an estimated quantity, but the result may be biased high.
- J- The result is an estimated quantity, but the result may be biased low.
- R The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
- UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

Joan Purdie

From: Garey, David [David.Garey@dyncorp.com]
Sent: Tuesday, August 30, 2005 12:51 PM
To: Alice Evans; Joan Purdie
Cc: Mary O'Donnell
Subject: Region 09 | Case 34502 | Lab LIBRTY | Issue Multiple | FINAL

Joan, please find below the remainder of your resolutions.

Issue 2: The TR/COC list sample ID MY21C1 but the Lab did not receive this sample.
Resolution 2: Per Region 9, there will be no sample MY21C1 to be analyzed.

Issue 3: The TR/COC lists 2 containers for the Lab QC sample, MY21B9, however the Lab received 3. Could one of the MY21B9 containers be MY21C1?
Resolution 3: Per Region 9, sample MY21B9 is comprised of 3 bottles.

Please let me know if you have any additional questions.

Thanks,

DG

-----Original Message-----

From: Bauer.Richard@epamail.epa.gov [mailto:Bauer.Richard@epamail.epa.gov]
Sent: Tuesday, August 30, 2005 12:23 PM
To: david.garey@dyncorp.com
Cc: R9RSCC@epamail.epa.gov
Subject: Fw: (8-21) Region 09 | Case 34502 | Lab LIBRTY | Issue Multiple

Here is response from samplers. Let me know if there are still questions.

----- Forwarded by Richard Bauer/R9/USEPA/US on 08/30/2005 09:20 AM-----

Hi Rich,

Here's what I can tell you about about issues 2 and 3 below:

Because we've been instructed to do so this event, we are collecting 3 bottles for the metals Lab QC samples. This is what we did on 8/22/05, and the sample was labeled as MY21B9. I suspect that the denotation of 2 bottles on the COC was an oversight/remnant of our usual collection of only 2 bottles (i.e., twice the volume) for Lab QC samples. Sample MY21C1 was supposed to be a field blank. However, because we were holding on to samples that day (8/22/05) until a lab got assigned, we inadvertently didn't prepare the field blank. So, I think we should inform the lab that there is no sample to be analyzed for COC sample MY21C1 and that sample MW21B9 is comprised of 3 bottles, not two.

8/30/2005

12

