



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: June 29, 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.:	32989
SDG No.:	MY1C21
Laboratory:	Ceimic Corporation (CEIMIC)
Analysis:	CLP Dissolved Metals By ICP-AES
Samples:	16 Groundwater Samples (see Case Summary)
Collection Date:	June 22, 23, and 24, 2004
Reviewer:	Stan Kott, 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: Jennie Han-Liu, CLP PO USEPA Region 1
Steve Remaley, CLP PO USEPA Region 9

CLP PO: FYI Action

SAMPLING ISSUES: Yes No

Data Validation Report

Case No.: 32989
SDG No.: MY1C21
Site: Omega Chem OU2
Laboratory: Ceimic Corporation (CEIMIC)
Reviewer: Stan Kott, ESAT/LDC
Date: June 29, 2006

I. CASE SUMMARY

Sample Information

Samples: MY1C21 through MY1C28 and MY1C30 through MY1C37
Concentration and Matrix: Low Concentration Groundwater
Analysis: CLP Dissolved Metals By ICP-AES
SOW: ILM05.3 and Modification Reference Number AES060304.0
Collection Date: June 22, 23, and 24, 2004
Sample Receipt Date: June 24, 2004
Preparation Date: July 8, 2004
Analysis Date: July 8 and 9, 2004

Field QC

Field Blanks (FB): Not Provided
Equipment Blanks (EB): Not Provided
Background Samples (BG): Not Provided
Field Duplicates (D1): MY1C26 and MY1C27

Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Water (PBW) and samples listed above
Matrix Spike: MY1C34S
Duplicates: MY1C34D
ICP Serial Dilution: MY1C34L

Analysis: CLP Dissolved Metals By ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	July 8, 2004	July 8 and 9, 2004
Percent Solids	Not Applicable	Not Applicable

CLP PO Action

The non-detected results for silver in all samples are rejected (R) since less than 50% of the silver in the aqueous laboratory control sample (LCS) was recovered.

Sampling Issues

None.

Additional Comments

Note that Ceimic Corporation laboratory is no longer in operation.

The samples in this SDG were analyzed for select metals (aluminum, calcium, iron, magnesium, potassium, and sodium) plus boron and silicon by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number AES060304.0.

The laboratory was instructed by Region 9 to report elements scheduled for ICP-MS analysis in SDG MY1C22 in this SDG due to suspected matrix interferences and carryovers that occurred during ICP-MS analysis. The laboratory also notes that only the elements specified in Modification Reference Number AES060304.0 were included in the matrix spike sample. (See Comment E and attached SDG Narrative.)

All samples were analyzed at a 3-fold dilution due to silicon concentrations that exceeded the instrument's linear range. No adverse effect on data quality is expected.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW), except as noted, have been met.

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), Tracking Number: 1103.0, Modification Reference Number: AES060304.0, June 9, 2004;
- 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 Data Review*, October 2004.

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)		
4.	Blanks	Yes	C
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	No	A
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	No	E
9.	ICP Serial Dilution Analysis	No	D
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
12.	Sample Quantitation	Yes	B
13.	Overall Assessment	Yes	

N/A = Not Applicable

III. VALIDITY AND COMMENTS

- A. The following non-detected results are rejected and flagged "R" in Table 1A because an aqueous laboratory control sample (LCS) recovery result is outside method QC limits.

X Silver in all samples

The percent recovery for silver is presented below and is based on an ideal recovery of 100%.

Analyte	% Recovery
Silver	10

The results reported for silver in all samples are below the method detection limit (MDL) and are considered unacceptable because less than 50% of silver in the aqueous LCS was recovered. The low LCS recovery indicates an analytical deficiency and false negatives may exist.

The purpose of the LCS is to monitor the overall performance of all steps in the analysis under ideal conditions, including sample preparation.

- B. Results above the MDL but below the contract required quantitation limit (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.

- C. The following results are reported as non-detected (U) in Table 1A due to low level preparation blank (PBW) contamination.

X Aluminum in samples MY1C23, MY1C24, MY1C27, MY1C35, and MY1C37
X Manganese in samples MY1C24, MY1C25, and MY1C28

Aluminum (49.6 µg/L) and manganese (2.1 µg/L) were found in preparation blank PBW. These results are greater than their respective MDLs but less than the respective CRQLs. Sample results greater than or equal to the MDL but less than the CRQL are reported as non-detected (U) at the respective CRQL.

A preparation blank is an analytical control that contains distilled, deionized water, or baked sand for solid matrices, and reagents, which is carried through the entire analytical procedure. The preparation blank is used to determine the level of contamination introduced by the laboratory during preparation and analysis.

- D. The following results are estimated and flagged "J" in Table 1A because an ICP serial dilution result is outside method QC limits.

X Potassium in all samples

The percent difference for the ICP serial dilution analysis of sample MY1C34L did not meet the 10% criterion for potassium as shown below.

Analyte	% Difference
Potassium	-32

Results reported for potassium in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The result for the diluted sample was lower than the original. Therefore, the reported potassium sample results may be biased high.

A five-fold dilution of the laboratory QC sample is performed in association with the ICP procedure to indicate whether interference exists due to sample matrix effects. If the analyte concentration is sufficiently high (minimally a factor of 50 above the MDL in the original sample), the five fold serial dilution must agree within 10% of the original results after correction for dilution.

- E. Inadequate matrix-specific laboratory QC was performed for this SDG. The Inorganic SOW states that at least one matrix spike sample analysis shall be performed on each group of samples of a similar matrix type and concentration or for each SDG. The laboratory performed one matrix spike sample analysis. However, antimony, arsenic, barium, beryllium cadmium, chromium, cobalt, copper, lead, manganese, nickel, selenium, silver, thallium, vanadium, and zinc were not included in the matrix spike sample. The effect on data quality is not known.

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.

SDG Narrative

Laboratory Name: Ceimic Corporation

Case No.: 32989

SDG No.: MY1C21

Contract: 68W02063

Modification Tracking Number: 1103.0

Ceimic Project No.: 040598

The following ILM05.3 (ICP-AES/MS) sixteen groundwater samples were received at CEIMIC Corporation on June 24 and 25, 2004:

<u>EPA ID</u>	<u>Ceimic ID</u>
MY1C26	040598-01
MY1C27	040598-02
MY1C28	040598-03
MY1C30	040598-04
MY1C31	040598-05
MY1C32	040598-06
MY1C33	040598-07
MY1C21	040598-08
MY1C22	040598-09
MY1C23	040598-10
MY1C24	040598-11
MY1C25	040598-12
MY1C34	040598-13
MY1C34	040598-13
MY1C34	040598-13
MY1C35	040598-14
MY1C36	040598-15
MY1C37	040598-16

Comments on Data Package

The samples in this SDG for Case 32989 were received for Total Metals analysis by ICP-MS; Total Aluminum, Calcium, Iron, Magnesium, Sodium, Potassium, Boron, and Silicon analysis by ICP-AES, Total Mercury analysis by CVAA, and Total Cyanide analysis by automated spectrophotometry. This SDG reports only the results of the ICP-AES analysis.

SDG MY1C22 reports the ICP-MS analysis of these samples; however, due to suspected matrix interferences and carryovers, many non-compliances exist. After consultation with Steve Remaley, the CLP Project Officer at EPA Region IX, the ICP-AES analysis in this SDG (MY1C21) will report all ICP-MS target analytes in addition to the eight requested analytes listed above. The ICP-MS analysis is still reported as scheduled for SDG MY1C22.

The above samples were digested in accordance with the Inorganic Statement of Work (SOW) ILM05.3, with modifications as listed in the Request for Quote for Modified Analysis with Tracking Number 1103.0. A copy of the modifications is included immediately after this SDG Narrative.

When ICP-AES raw data has been reprocessed in an SDG, the words "Reprocessed on" followed by the date and time of reprocessing will sometimes be printed in the header of each standard and sample raw data report. The word "Reprocessed" is used when the original sequence data is regenerated after it was collected and processed with incorrect information (such as sample information, standard nomenclature) or settings (such as background correction, internal standard, dilution factor, QC concentration, wrong IEC table, etc.)

QA/QC Samples:

Matrix spike and duplicate analysis – as well as ICP serial dilution – were performed on sample MY1C34 as indicated on the Traffic Report / Chain of Custody. A post-digestion spike was not required for this SDG. **Only those elements originally requested for ICP-AES analysis – Al, Ca, Fe, Mg, K, Na, B, Si – are included in the matrix spike analysis.**

Observations:

A "U" flag in the C column on the sample result forms (Form IA-IN) indicates that the concentration of that analyte in the sample is undetected at the experimentally-determined method detection limit (MDL). If analytes are detected at concentrations between the Contract Required Quantitation Limit (CRQL) and the MDL, a "J" flag is shown in the C column on the Form IA-IN.

The raw data for the MDL study of Boron and Silicon is located in SDG MY1C04.

The "N" and "*" qualifiers do not apply to this SDG. The "E" qualifier applies to Potassium for high concentration difference between sample MY1C34 and its serial dilution. Silicon is detected in all samples in this SDG at concentrations exceeding the experimentally-determined linear range concentration (10,000 ug/L) of the ICP-AES instrument. The sample digestates were reanalyzed at a dilution; these reanalyses are denoted by "D" qualifier on the Form IA-IN.

All target analytes for this SDG are monitored and reported on Form IIB-IN for the CRQL Check (CRI) standards, although seven elements (Al, Ba, Ca, Fe, Mg, Na, and K) do not require monitoring.

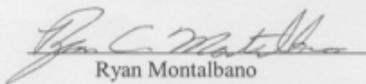
Due to limitations in the reporting software used for this SDG, a separate Form XIII-IN is included for the analytes Boron and Silicon.

Deviations from Contract:

None other than those described in the Modified Analysis and those prescribed by Mr. Remaley.

End of SDG Narrative.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or his designee, as verified by the following signature.



Ryan Montalbano
Supervisor, Inorganic Laboratories

07/15/2004

Date

