

**Appendix J**  
**Data Validation Reports**

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**Data Validation Reports**

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**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105068 Amendment 3

DATE: September 19, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	None Provided
SDG No.:	06-363
Laboratory:	CHESTER LabNet
Analysis:	PM10 and X-Ray Fluorescence (XRF)
Samples:	6 Teflon Air Filter Samples (see Case Summary)
Collection Dates:	November 21, 27, and December 3, 2006
Reviewers:	Stan Kott, ESAT/Laboratory Data Consultants and Kevin Woodruff, ESAT/ICF International

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

SAMPLING ISSUES: [ ] Yes [X] No



## Data Validation Report

Case No.: None Provided  
SDG No.: 06-363  
Site: Asarco Hayden  
Laboratory: CHESTER LabNet  
Reviewers: Stan Kott, ESAT/LDC and  
Kevin Woodruff, ESAT/ICF International  
Date: September 19, 2007

### I. CASE SUMMARY

#### Sample Information

Hayden Samples: MY-112106, MY-112706, and MY-120306  
Winkelman Samples: HS-112106, HS-112706, and HS-120306  
Concentration and Matrix: Low Concentration 47mm Teflon Air Filter  
Analysis: PM10 and X-Ray Fluorescence (XRF)  
SOW: EPA Compendium Methods IO-3.1 and IO-3.3  
Collection Date: November 21, 27, and December 3, 2006  
Sample Receipt Date: December 11, 2006  
Preparation and Weighing Dates: October 16 and December 12, 2006  
XRF Analysis Date: December 14, 2006

#### Field QC

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

#### Laboratory QC

Method Blanks: Not Applicable  
Associated Samples: Samples listed above  
Laboratory Duplicate: RT4359 (MY-112106)

Analysis: PM10 and XRF

<u>Analyte</u>	<u>Sample Preparation Date</u>	<u>Analysis Date</u>
PM10 Preparation/Weighing	October 16, 2006	December 12, 2006
XRF Metals	Not Applicable	December 14, 2006

#### Sampling Issues

None.

#### Additional Comments

**As directed by the EPA TOM, a Tier 3 data review was performed.**

The Hayden air monitoring station analytes and contract required quantitation limits (CRQLs) provided in Table 1A are from the Statement of Work, dated August 10, 2006, Table 1. The Winkleman air monitoring station analytes and CRQLs provided in Table 1A are from the Statement of Work, dated August 10, 2006, Table 2.

ESAT could not check calculations from instrument raw data counts to final results due to the complexity of the calculations and due to insufficient information about the instrument software algorithms. This difficulty is noted in analytical method IO-3.3 and by the laboratory. The  $\mu\text{g}/\text{filter}$  and  $\mu\text{g}/\text{meter}^3$  ( $\mu\text{g}/\text{m}^3$ ) concentrations were recalculated as per laboratory instructions. (See Attachment 1.)

The laboratory indicated that the instrument calculated arsenic concentrations for samples MY-112106 and MY-120306 were lowered by the analyst after examining the spectra. No adverse effect on data quality is expected. (See Attachment 2.)

Results reported in  $\mu\text{g}/\text{m}^3$  are calculated using a standard  $24 \pm 2.4$  cubic meter sample size. No adverse effect on data quality is expected.

This report was prepared in accordance with the following documents:

- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1, Selection, Preparation and Extraction of Filter Material*, June 1999;
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.3, Determination of Metals in Ambient Particulate Matter Using X-Ray Fluorescence (XRF) Spectroscopy*, June 1999;
- *Statement of Work; 10 August 2006; Project: Perform Ambient Air Tests on Air Filters Gathered from the ASARCO Hayden Plant Site, AZ. Purchase Order: W91238-06-P-TBD; Issued by USACE Sacramento District*;
- *Standard Operating Procedure XR-002.02; Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex 770); CHESTER LabNet*, July 3, 2003; and
- *Region 9 Standard Operating Procedure 906, Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*.

## 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 Preparation and Weighing	Yes	
3.	Calibration	Yes	
4.	Blank	Yes	A
5.	Standard Reference Materials (SRM)	Yes	
6.	Duplicate Sample Analysis	Yes	
7.	Matrix Spike Sample Analysis	N/A	
8.	Field Duplicate Sample Analysis	N/A	
9.	Sample Quantitation	No	B
10.	Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

A. The following results are reported without blank corrections in Table 1A.

- Antimony, barium, cadmium, and silver in samples HS-112106, HS-112706, and HS-120306

The laboratory indicated that, in order to lower the detection limit and uncertainty for cadmium, the counting time was extended to 1440 for the sp4 analytes by using instrument protocol 9. Since blank correction values were determined using instrument protocol 6 with a counting time of 180, the resulting higher uncertainties would increase the detection limit for cadmium. In order to maintain the lower detection limit, all sp4 analytes are reported without blank correction. (See Attachment 2.) Since the concentrations for the analytes listed above are less than three times their respective uncertainties, no adverse effect on data quality is expected.

*The blank correction values are determined using ten blank Teflon air filters analyzed using protocol 6. Protocol 6 has a 180 counting time for the sp4 analytes. Blank correction values are determined at the initial calibration of the XRF instrument.*

B. The following results are estimated and flagged "J" in Table 1A because a data processing sub-routine was not utilized to make corrections to the affected analyte results.

- Arsenic and selenium in samples MY-120306 and MY-112106

The laboratory indicates that whenever bismuth is detected, a separate data processing sub-routine is utilized to correct the data for arsenic and selenium. (See Attachment 2.) The data indicate bismuth concentrations of  $0.0758 \pm 0.0049 \mu\text{g}/\text{cm}^2$  and  $0.2361 \pm 0.0125 \mu\text{g}/\text{cm}^2$  for samples MY-120306 and MY-112106, respectively. Since arsenic and selenium data were not reprocessed, results reported for arsenic and selenium in the samples listed above are considered quantitatively uncertain.



ANALYTICAL RESULTS  
Table 1A

SDG No.: 06-363

Case No.: None  
Site: Asarco Hayden  
Lab: Chester LabNet  
Reviewer: Stan Kott, ESAT/LDC  
Date: September 19, 2007

Analysis Type: PM10 Teflon Air Filters for Select  
Total Metals by XRF

QUALIFIED DATA  
Concentration in ug / m3

PARAMETER	Hayden MY-112706 6177003 11/27/2006		Hayden MY-120306 6177003 12/03/2006		Hayden MY-112106 6177003 11/21/2006		CRQL	
	Result	Com	Val	Com	Result	Com	Val	Com
ANTIMONY	* 0.0100 ± 0.0039				* 0.0000 ± 0.0035			
ARSENIC	0.0484 ± 0.0066	J			0.1040 ± 0.0127	B		
BARIIUM	0.0186 ± 0.0056				* 0.0007 ± 0.0029			
CADMIUM	* 0.0042 ± 0.0028				* 0.0032 ± 0.0025			
CHROMIUM	0.0025 ± 0.0007				* 0.0008 ± 0.0004			
COBALT	* 0.0000 ± 0.0105				* 0.0000 ± 0.0037			
COPPER	1.196 ± 0.1338				0.2930 ± 0.0329			
MANGANESE	0.0170 ± 0.0020				0.0079 ± 0.0010			
NICKEL	* 0.0000 ± 0.0009				* 0.0008 ± 0.0008			
SELENIUM	0.0108 ± 0.0013	J			* 0.0008 ± 0.0004			
SILVER	* 0.0013 ± 0.0026				* 0.0000 ± 0.0024			
VANADIUM	* 0.0000 ± 0.0016				* 0.0000 ± 0.0008			
PM10 Net Mass	47.42 ± 4.76				30.75 ± 3.10			

\* Concentration is less than three times the uncertainty

PARAMETER	Winkelman HS-120306 6177003 12/03/2006		Winkelman HS-112706 6177003 11/27/2006		Winkelman HS-112106 6177003 11/21/2006		CRQL	
	Result	Com	Val	Com	Result	Com	Val	Com
ANTIMONY	* 0.0004 ± 0.0011				* 0.0000 ± 0.0011			
ARSENIC	* 0.0000 ± 0.0004	A			* 0.0007 ± 0.0010			
BARIIUM	* 0.0033 ± 0.0017				* 0.0004 ± 0.0017			
CADMIUM	* 0.0000 ± 0.0008	A			* 0.0007 ± 0.0008			
CHROMIUM	* 0.0003 ± 0.0004				* 0.0000 ± 0.0004			
COBALT	* 0.0000 ± 0.0011				* 0.0000 ± 0.0012			
COPPER	0.0107 ± 0.0014				0.1022 ± 0.0115			
MANGANESE	* 0.0006 ± 0.0005				0.0016 ± 0.0005			
NICKEL	* 0.0000 ± 0.0007				* 0.0000 ± 0.0007			
SELENIUM	* 0.0000 ± 0.0004				0.0011 ± 0.0004			
SILVER	* 0.0006 ± 0.0008				* 0.0013 ± 0.0008			
VANADIUM	* 0.0000 ± 0.0003	A			* 0.0011 ± 0.0004			
PM10 Net Mass	6.50 ± 0.77				10.08 ± 1.09			

\* Concentration is less than three times the uncertainty

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit

N/A - Not Applicable

NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



## 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.



## Attachment 1

Gretchen-

Here's what I dug up to answer the questions. I had to zip the files since your e-mail server wouldn't allow the size of e-mail with uncompressed files.

Gravimetry-

I have attached two files that contain all the gravimetry for the project. They include the temperature and humidity data.

I don't quite understand the question about the batch quality checks for filter contamination. I read through IO-3.1 and didn't see anything in there about batch quality checks. Does she mean filter inspection? If she does, we don't have any data for that. If a filter is defective, it isn't used. We don't keep a record of defective filters.

XRF

The calibration and blank data are attached for the two instruments that were used to analyze the samples. XRF spectrometers are very stable and do not require calibration very often. The blank data is not specific to this project, but is from filters from the same lot of filters used for the Asarco project.

We do not guarantee that the reported MDLs will meet the CRQLs. This was discussed when we were bidding on the project. The samples are analyzed for the counting times required to achieve the required detection limits on interference free samples, but corrections are made for various reasons which can cause the detection limit to rise to above the CRQL.

I e-mailed the raw data for reports 06-363 and 07-021 on June 8. I am resending it in this e-mail.

As for the sample calculation, it isn't really possible to calculate the ug/cm2 concentrations by hand due to the complexity of XRF. The calculations to get to ug/filter and ug/m3 are quite easy; multiply the ug/cm2 number by the deposit area (11.3) for ug/filter and divide the ug/filter concentration by the volume (usually 24) to get ug/m3. I can send you our XRF SOP if you want to take a stab at calculating the ug/cm2 concentration.

Hopefully this is enough information. If Rose needs anything else, maybe it would be best if she could call me directly so I understand exactly what she wants.

Paul Duda  
CHESTER LabNet  
12242 SW Garden Place  
Tigard, OR 97223  
pduda@chesterlab.net  
<http://www.chesterlab.net>  
(503) 624-2183 ext. 100  
fax (503) 624-2653



Attachment 2

Rose Fong/R9/USEPA/US  
08/20/2007 11:30 AM

To Stan Kott/R9/USEPA/US@EPA  
cc  
bcc  
Subject Fw: Asarco PM10 \ XRF Additional Questions

----- Forwarded by Rose Fong/R9/USEPA/US on 08/20/2007 11:30 AM -----



"Paul Duda"  
<pduda@chesterlab.net>  
08/16/2007 03:58 PM

To <Gretchen.Engel@CH2M.com>  
cc <Melanie.West@CH2M.com>,  
<jennifer.holland@CH2M.com>,  
<kevin.murdock@CH2M.com>, Rose  
Fong/R9/USEPA/US@EPA, <bains.foehr@CH2M.com>  
Subject RE: Asarco PM10 \ XRF Additional Questions

Gretchen-

Sorry for the delay. I've been on vacation and am just now getting caught up.

Attached is the missing gravimetry.

The As discrepancies for samples 06-T4357 and 06-T4359 were due to corrections done after the analyst examined the spectra. The printout you received did not have the hand corrections included. I scanned the pages in question and have attached them.

Whenever we detect Bismuth, As and Se are affected. A separate processing routine is utilized to make the corrections and unfortunately the printouts for that routine didn't make it into the electronic version of the file I sent earlier. A scanned copy is attached for samples 06-T4684 and 06-T4686.

The uncorrected concentrations and uncertainties were used for all the samples with discrepancies for Ag, Cd and Sb in order to keep the uncertainties low. These samples all had one condition run at protocol 9 in order to lower the Cd detection limit. Blank correction values are determined using similar media; 10 filters at protocol 6. The resulting uncertainties when applied to protocol 9 sensitivities will significantly raise the detection limits and defeat the purpose of increasing the counting times used for protocol 9 to lower the detection limit. All the Winkelman samples that are analyzed with extended counting times have the sp4 analytes reported without blank corrections. I hope this all makes sense. If not, I recommend talking to our XRF analyst, Rick Sarver. He can be reached at the phone number below.

Paul Duda  
CHESTER LabNet  
12242 SW Garden Place  
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pduda@chesterlab.net  
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**ICF International / Laboratory Data Consultants**

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

**MEMORANDUM**

**TO:** John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

**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.: 00105068 Amendment 3

**DATE:** March 27, 2008

**SUBJECT:** Tier 1A Data Evaluation Memo for the Asarco Hayden project, SDGs: 06-373, 07-009, 07-040, 07-056, 07-093, and 07-130

**SUMMARY:** 44 Teflon air filter samples received by CHESTER LabNet were analyzed for PM10 and select metals by X-Ray Fluorescence (XRF).

A forms-only evaluation of the data packages was performed to identify any key analytical issues/deficiencies affecting data quality. This evaluation approach is employed when in-depth data review is not required as indicated by the data user. During this limited evaluation, areas of concern were noted (see Lettered and Additional Comments).

The evaluation included: a review of the data package for completeness, review of the chain of custody forms (against laboratory reported information, for signatures, for sample condition upon receipt by the laboratory and for sample preservation), review of holding times, review of QC summaries, review of blanks for contamination, random check of reported results against raw data, and a random check of raw data for interference problems or system control problems.

The following data quality issues should be noted:

- A. The PM10 and XRF metal results for sample MY-102806 in SDG 07-009 and sample MY-030907 in SDG 07-093 are rejected and should be flagged "R" due to compromised filter medium. Method IO-3.3, Section 8.3, specifies that filter samples are to be checked for invalidating conditions such as holes and tears which would prevent quantitative analysis.
- B. The XRF metal results for sample MY-121506 in SDG 06-373 are estimated and should be flagged "J" due to the filter being sampled on the wrong side. Method IO-3.3, Section 8.2 specifies that the sample should be collected on the side of the filter with the supporting ring. The PM10 gravimetric data sheet indicates the filter was

sampled on the wrong side.

- C. The following results are estimated and should be flagged "J" because of duplicate results outside the ESAT  $\forall$ 35 relative percent difference (RPD) QC limit. Duplicate results that do not meet the  $\forall$ 35 RPD criterion for precision are listed below.

SDG	Sample	Analyte	Duplicate RPD and uncertainty
07-040	MY-020707	Chromium	60.1 $\pm$ 46.2
07-093	HS-031507	Arsenic	40.6 $\pm$ 30.9
07-093	HS-031507	Selenium	192.6 $\pm$ 70.7
07-093	MY-032107	Barium	41.4 $\pm$ 41.7
07-093	MY-032107	Selenium	145.5 $\pm$ 28.7
07-130	MY-042007	Arsenic	-39.6 $\pm$ 36.7

Results for analytes listed above in the samples listed above are considered quantitatively uncertain.

- D. The antimony, barium, cadmium, and silver results for all Winkelman samples are reported without blank corrections. The laboratory indicated that, in order to lower the detection limit and uncertainty for cadmium, the counting time was extended to 1440 for the sp4 analytes by using instrument protocol 9. Since blank correction values were determined using instrument protocol 6 with a counting time of 180, the resulting higher uncertainties would increase the detection limit for cadmium. In order to maintain the lower detection limit, all sp4 analytes are reported without blank correction. Since the concentrations for the analytes listed above are less than three times their respective uncertainties, no adverse effect on data quality is expected.

Additional Comments:

1. The Hayden air monitoring station analytes and contract required quantitation limits (CRQLs) are from the Statement of Work, dated August 10, 2006, Table 1. The Winkelman air monitoring station analytes and CRQLs are from the Statement of Work, dated August 10, 2006, Table 2.
2. The Chain of Custody (COC) record form did not specify a sample to be used for laboratory quality control (QC). As a result, the laboratory selected the QC samples. The effect on data quality is not known.
3. For SDG 07-040, samples HS-012607 and MY-012607 have an incorrect sample date of 1/27/07. The COC indicates the correct sample date is 1/26/07. No adverse effect on data quality is expected.
4. Results reported in  $\mu\text{g}/\text{m}^3$  are calculated using a standard  $24 \pm 2.4$  cubic meter sample size. No adverse effect on data quality is expected.

A Table 1A was not requested.

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.

## 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.







**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) RF  
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.: 00105068 Amendment 3

DATE: September 13, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	None Provided
SDG No.:	07-021
Laboratory:	CHESTER LabNet
Analysis:	PM10 and X-Ray Fluorescence (XRF)
Samples:	4 Teflon Air Filter Samples (see Case Summary)
Collection Dates:	January 8 and 14, 2007
Reviewers:	Stan Kott, ESAT/Laboratory Data Consultants and Kevin Woodruff, ESAT/ICF International

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

SAMPLING ISSUES: [ ] Yes [X] No



## Data Validation Report

Case No.: None Provided  
SDG No.: 07-021  
Site: Asarco Hayden  
Laboratory: CHESTER LabNet  
Reviewers: Stan Kott, ESAT/LDC and  
Kevin Woodruff, ESAT/ICF International  
Date: September 13, 2007

### I. CASE SUMMARY

#### Sample Information

Hayden Samples: MY-010807 and MY-011407  
Winkelman Samples: HS-010807 and HS-011407  
Concentration and Matrix: Low Concentration 47mm Teflon Air Filter  
Analysis: PM10 and X-Ray Fluorescence (XRF)  
SOW: EPA Compendium Methods IO-3.1 and IO-3.3  
Collection Date: January 8 and 14, 2007  
Sample Receipt Date: January 23, 2007  
Preparation and Weighing Dates: November 28, 2006 and January 23, 2007  
Analysis Date: January 26, 2007

#### Field QC

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

#### Laboratory QC

Method Blanks: Not Applicable  
Associated Samples: Samples listed above  
Laboratory Duplicate: RT4684 (MY-010807)

Analysis: PM10 and XRF

<u>Analyte</u>	<u>Sample Preparation Date</u>	<u>Analysis Date</u>
PM10 Preparation/Weighing	November 28, 2006	January 23, 2007
XRF Metals	Not Applicable	January 26, 2007

#### Sampling Issues

None.

#### Additional Comments

**As directed by the EPA TOM, a Tier 3 data review was performed.**

The Hayden air monitoring station analytes and contract required quantitation limits (CRQLs) provided in Table 1A are from Statement of Work, dated August 10, 2006, Table 1. The Winkleman air monitoring station analytes and CRQLs provided in Table 1A are from Statement of Work, dated August 10, 2006, Table 2.

ESAT could not check calculations from instrument raw data counts to final results due to the complexity of the calculations and due to insufficient information about the instrument software algorithms. This difficulty is noted in analytical method IO-3.3 and by the laboratory. The  $\mu\text{g}/\text{filter}$  and  $\mu\text{g}/\text{meter}^3$  ( $\mu\text{g}/\text{m}^3$ ) concentrations were recalculated as per laboratory instructions. (See Attachment 1.)

The laboratory indicated that since bismuth was detected in samples MY-010807 and MY-011407, arsenic and selenium results were calculated using a separate data processing subroutine. No adverse effect on data quality is expected. (See Attachment 2.)

Results reported in  $\mu\text{g}/\text{m}^3$  are calculated using a standard  $24 \pm 2.4$  cubic meter sample size. No adverse effect on data quality is expected.

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:

- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1, Selection, Preparation and Extraction of Filter Material*, June 1999;
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.3, Determination of Metals in Ambient Particulate Matter Using X-Ray Fluorescence (XRF) Spectroscopy*, June 1999;
- *Statement of Work; 10 August 2006; Project: Perform Ambient Air Tests on Air Filters Gathered from the ASARCO Hayden Plant Site, AZ. Purchase Order: W91238-06-P-TBD; Issued by USACE Sacramento District;*
- *Standard Operating Procedure XR-002.02; Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex 770); CHESTER LabNet, July 3, 2003; and*
- *Region 9 Standard Operating Procedure 906, Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages.*

## 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 Preparation and Weighing	Yes	
3.	Calibration	Yes	
4.	Blank	Yes	A
5.	Standard Reference Materials (SRM)	Yes	
6.	Duplicate Sample Analysis	Yes	
7.	Matrix Spike Sample Analysis	N/A	
8.	Field Duplicate Sample Analysis	N/A	
9.	Sample Quantitation	Yes	
10.	Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

A. The following results are reported without blank corrections in Table 1A.

- Antimony, barium, cadmium, and silver in samples HS-010807 and HS-011407

The laboratory indicated that, in order to lower the detection limit and uncertainty for cadmium, the counting time was extended to 1440 for the sp4 analytes by using instrument protocol 9. Since blank correction values were determined using instrument protocol 6 with a counting time of 180, the resulting higher uncertainties would increase the detection limit for cadmium. In order to maintain the lower detection limit, all sp4 analytes are reported without blank correction. (See Attachment 2.) Since the concentrations for the analytes listed above are less than three times their respective uncertainties, no adverse effect on data quality is expected.

*The blank correction values are determined using ten blank Teflon air filters analyzed using protocol 6. Protocol 6 has a 180 counting time for the sp4 analytes. Blank correction values are determined at the initial calibration of the XRF instrument.*



ANALYTICAL RESULTS  
Table 1A

Case No. : ACE(68)35637  
 Site : Asarco Hayden  
 Lab : Chester LabNet  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : September 13, 2007

Analysis Type : PM10 Teflon Air Filters for  
 Select Total Metals by XRF

QUALIFIED DATA  
 Concentration in ug / m3

PARAMETER	Hayden		Hayden		CRQL					
	Result	Com	Val	Com	Result	Com	Val	Com	Result	Com
ANTIMONY	* 0.0114 ± 0.0042									
ARSENIC	0.1524 ± 0.0178				0.0008					
BARIUM	0.0427 ± 0.0057				0.52					
CADMIUM	0.0141 ± 0.0034				0.0037					
CHROMIUM	* 0.0012 ± 0.0005				0.0033					
COBALT	* 0.0000 ± 0.0093				0.0069					
COPPER	2.734 ± 0.3058				0.75					
MANGANESE	0.0091 ± 0.0012				0.051					
NICKEL	* 0.0000 ± 0.0010				0.0040					
SELENIUM	0.1217 ± 0.0136				1.60					
SILVER	* 0.0077 ± 0.0030				0.079					
VANADIUM	* 0.0000 ± 0.0005				0.40					
PM10 Net Mass	38.71 ± 3.893				50					

\* Concentration is less than three times the uncertainty

PARAMETER	Winkelman		Winkelman		CRQL					
	Result	Com	Val	Com	Result	Com	Val	Com	Result	Com
ANTIMONY	* 0.0000 ± 0.0011	A								
ARSENIC	* 0.0000 ± 0.0004				0.0008					
BARIUM	* 0.0000 ± 0.0014	A			0.52					
CADMIUM	* 0.0000 ± 0.0008	A			0.0083					
CHROMIUM	* 0.0000 ± 0.0003				0.0033					
COBALT	* 0.0000 ± 0.0009				0.0069					
COPPER	0.0159 ± 0.0020				0.75					
MANGANESE	* 0.0011 ± 0.0004				0.051					
NICKEL	* 0.0012 ± 0.0007				0.0040					
SELENIUM	* 0.0000 ± 0.0003				1.60					
SILVER	* 0.0000 ± 0.0008				0.079					
VANADIUM	* 0.0000 ± 0.0003	A			0.40					
PM10 Net Mass	4.250 ± 0.5952				50					

\* Concentration is less than three times the uncertainty

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 N/A - Not Applicable NA - Not Analyzed



## 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.



## Attachment 1

Gretchen-

Here's what I dug up to answer the questions. I had to zip the files since your e-mail server wouldn't allow the size of e-mail with uncompressed files.

Gravimetry-

I have attached two files that contain all the gravimetry for the project. They include the temperature and humidity data.

I don't quite understand the question about the batch quality checks for filter contamination. I read through IO-3.1 and didn't see anything in there about batch quality checks. Does she mean filter inspection? If she does, we don't have any data for that. If a filter is defective, it isn't used. We don't keep a record of defective filters.

XRF

The calibration and blank data are attached for the two instruments that were used to analyze the samples. XRF spectrometers are very stable and do not require calibration very often. The blank data is not specific to this project, but is from filters from the same lot of filters used for the Asarco project.

We do not guarantee that the reported MDLs will meet the CRQLs. This was discussed when we were bidding on the project. The samples are analyzed for the counting times required to achieve the required detection limits on interference free samples, but corrections are made for various reasons which can cause the detection limit to rise to above the CRQL.

I e-mailed the raw data for reports 06-363 and 07-021 on June 8. I am resending it in this e-mail.

As for the sample calculation, it isn't really possible to calculate the ug/cm<sup>2</sup> concentrations by hand due to the complexity of XRF. The calculations to get to ug/filter and ug/m<sup>3</sup> are quite easy; multiply the ug/cm<sup>2</sup> number by the deposit area (11.3) for ug/filter and divide the ug/filter concentration by the volume (usually 24) to get ug/m<sup>3</sup>. I can send you our XRF SOP if you want to take a stab at calculating the ug/cm<sup>2</sup> concentration.

Hopefully this is enough information. If Rose needs anything else, maybe it would be best if she could call me directly so I understand exactly what she wants.

Paul Duda  
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Attachment 2



Rose Fong/R9/USEPA/US  
08/20/2007 11:30 AM

To Stan Kolt/R9/USEPA/US@EPA  
cc  
bcc

Subject Fw: Asarco PM10 \ XRF Additional Questions

----- Forwarded by Rose Fong/R9/USEPA/US on 08/20/2007 11:30 AM -----



"Paul Duda"  
<pduda@chesterlab.net>  
08/16/2007 03:58 PM

To <Gretchen.Engel@CH2M.com>  
cc <Melanie.West@CH2M.com>,  
<jennifer.holland@CH2M.com>,  
<kevin.murdock@CH2M.com>, Rose  
Fong/R9/USEPA/US@EPA, <bains.foehr@CH2M.com>  
Subject RE: Asarco PM10 \ XRF Additional Questions

Gretchen-

Sorry for the delay. I've been on vacation and am just now getting caught up.

Attached is the missing gravimetry.

The As discrepancies for samples 06-T4357 and 06-T4359 were due to corrections done after the analyst examined the spectra. The printout you received did not have the hand corrections included. I scanned the pages in question and have attached them.

Whenever we detect Bismuth, As and Se are affected. A separate processing routine is utilized to make the corrections and unfortunately the printouts for that routine didn't make it into the electronic version of the file I sent earlier. A scanned copy is attached for samples 06-T4684 and 06-T4686.

The uncorrected concentrations and uncertainties were used for all the samples with discrepancies for Ag, Cd and Sb in order to keep the uncertainties low. These samples all had one condition run at protocol 9 in order to lower the Cd detection limit. Blank correction values are determined using similar media; 10 filters at protocol 6. The resulting uncertainties when applied to protocol 9 sensitivities will significantly raise the detection limits and defeat the purpose of increasing the counting times used for protocol 9 to lower the detection limit. All the Winkelman samples that are analyzed with extended counting times have the sp4 analytes reported without blank corrections. I hope this all makes sense. If not, I recommend talking to our XRF analyst, Rick Sarver. He can be reached at the phone number below.

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**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

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.: 00105122

DATE: April 11, 2008

SUBJECT: Tier 1A Data Evaluation Memo for the Asarco Hayden project, SDGs: 07-161,  
07-181, 07-194, 07-232, 07-265, and 07-377

SUMMARY: 41 Teflon air filter samples received by CHESTER LabNet were analyzed for  
PM10 and select metals by X-Ray Fluorescence (XRF).

A forms-only evaluation of the data packages was performed to identify any key analytical issues/deficiencies affecting data quality. This evaluation approach is employed when in-depth data review is not required as indicated by the data user. During this limited evaluation, areas of concern were noted (see Lettered and Additional Comments).

The evaluation included: a review of the data package for completeness, review of the chain of custody forms (against laboratory reported information, for signatures, for sample condition upon receipt by the laboratory and for sample preservation), review of holding times, review of QC summaries, review of blanks for contamination, random check of reported results against raw data, and a random check of raw data for interference problems or system control problems.

The following data quality issues should be noted:

- A. PM10 and XRF analyte results for sample HS-050207 in SDG 07-161, sample MY-050207 in SDG 07-181, and sample MY-070707 in SDG 07-265 are rejected and should be flagged "R" due to compromised filter medium. For PM10 analysis, the *Quality Assurance Handbook for Air Pollution Measurements Systems*, Volume II: Part 1, Section 12 and, for XRF analysis, the *Method IO-3.3*, Section 8.3, specify that filter samples are to be checked for invalidating conditions such as holes, tears, non-uniform deposit density (NUDD), or other flaws which may affect the collection efficiency of the filter and quantitative analysis. The PM10 and XRF sample data sheets indicate invalidating conditions for the samples listed above were present.

- B. The XRF results for the analytes in the samples listed below are rejected and should be flagged "R" due to a non-uniform analyte deposit noted on the XRF sample data sheets. Method IO-3.3, section 8.3, specifies that a non-uniform deposit density cannot be quantitatively measured by XRF.

SDG	Sample	Analyte
07-161	HS-051407	Copper
07-265	HS-070707	Zinc
07-377	MY-092907	Manganese
07-377	MY-100507	Manganese
07-377	MY-101107	Manganese

- C. The XRF analyte results for the following samples are estimated and should be flagged "J" due to a non-uniform metal deposit present in the sample. If one of the analytes in a sample cannot be quantitatively measured (as stated in Comment B) the quantitation of the other metals in the sample may not be correct.

- Antimony, arsenic, barium, cadmium, chromium, cobalt, manganese, nickel, selenium, silver, and vanadium in sample HS-051407
- Antimony, arsenic, barium, cadmium, chromium, cobalt, copper, manganese, nickel, selenium, silver, and vanadium in sample HS-070707
- Antimony, arsenic, barium, cadmium, chromium, cobalt, copper, nickel, selenium, silver, and vanadium in sample MY-092907, MY-100507, and MY-101107

Results for analytes listed above in the samples listed above are considered quantitatively uncertain.

- D. The following results are estimated and should be flagged "J" because of duplicate results outside the ESAT  $\pm 35$  relative percent difference (RPD) QC limit. Duplicate results that do not meet the  $\pm 35$  RPD criterion for precision are listed below.

SDG	Sample	Analyte	Duplicate RPD and uncertainty
07-161	MY-051407	Barium	$36.6 \pm 51.5$
07-232	MY-070107	Chromium	$-50.6 \pm 26.9$
07-377	MY-101107	Antimony	$-53.7 \pm 90.7$
07-377	MY-101107	Selenium	$-48.0 \pm 25.0$

Results for analytes listed above in the samples listed above are considered quantitatively uncertain.

- E. The antimony, barium, cadmium, and silver results for all Winkelman samples are reported without blank corrections. The laboratory indicated that, in order to lower the detection limit and uncertainty for cadmium, the counting time was extended to 1440 for the sp4 analytes by using instrument protocol 9. Since blank correction values were determined using instrument protocol 6 with a counting time of 180, the resulting higher uncertainties would increase the detection limit for cadmium. In order to maintain the lower detection limit, all sp4 analytes are reported without blank correction. The effect on data quality is not known.

Additional Comments:

1. The Hayden air monitoring station analytes and contract required quantitation limits (CRQLs) are from the Statement of Work, dated August 10, 2006, Table 1. The Winkelman air monitoring station analytes and CRQLs are from the Statement of Work, dated August 10, 2006, Table 2.
2. The Chain of Custody (COC) record form did not specify a sample to be used for laboratory quality control (QC). As a result, the laboratory selected the QC samples. The effect on data quality is not known.
3. For SDG 07-194, two Hayden samples have identical sample identification of MY-061307. The laboratory was able to identify the samples by the 6/13/2007 and 6/19/2007 sampling dates listed on the COC and their respective filter lot numbers. No adverse effect on data quality is expected.
4. For SDG 07-265, bismuth was found in Hayden samples MY-071907 and MY-073107 and Winkelman samples HS-070107, HS-071307 and HS-071907. The arsenic and selenium results for these samples were corrected for bismuth interference as required by the laboratory's protocol. No adverse effect on data quality is expected.
5. Results reported in  $\mu\text{g}/\text{m}^3$  are calculated using a standard  $24 \pm 2.4$  cubic meter sample size. No adverse effect on data quality is expected.

A Table 1A was not requested.

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.



## 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.
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- J+ The result is an estimated quantity, but the result may be biased high.
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- 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.





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MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

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.: 00105122

DATE: April 17, 2008

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	None Provided
SDG No.:	07-325
Laboratory:	CHESTER LabNet
Analysis:	PM10 and X-Ray Fluorescence (XRF)
Samples:	8 Teflon Air Filter Samples (see Case Summary)
Collection Dates:	August 24, 30, September 5, 11, and 17, 2007
Reviewers:	Stan Kott, ESAT/Laboratory Data Consultants and Kevin Woodruff, ESAT/ICF International

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

SAMPLING ISSUES:  Yes  No



## Data Validation Report

Case No.: None Provided  
SDG No.: 07-325  
Site: Asarco Hayden  
Laboratory: CHESTER LabNet  
Reviewers: Stan Kott, ESAT/LDC and  
Kevin Woodruff, ESAT/ICF International  
Date: April 17, 2008

### I. CASE SUMMARY

#### Sample Information

Hayden Samples: MY-083007, MY-090507, MY-091107, and  
MY-091707  
Winkelman Samples: HS-082407, HS-090507, HS-091107, and HS-091707  
Matrix: 47mm Teflon Air Filter  
Analyses: PM10 and X-Ray Fluorescence (XRF)  
SOW: EPA Compendium Methods IO-3.1 and IO-3.3  
Collection Date: August 24, 30, September 5, 11, and 17, 2007  
Sample Receipt Date: September 21, 2007  
Preparation / Weighing Dates: July 20 and August 9 / September 24, 2007  
XRF Analysis Date: September 28 through October 2, 2007

#### Field QC

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

#### Laboratory QC

Method Blanks: Not Applicable  
Associated Samples: Samples listed above  
Laboratory Replicate: RT3893 (MY-091107)

Analysis: PM10 and XRF

<u>Analyte</u>	<u>Sample Preparation Date</u>	<u>Analysis Date</u>
PM10 Preparation/Weighing	July 20 and August 9, 2007	September 24, 2007
XRF Metals	Not Applicable	September 28 through October 2, 2007

#### Sampling Issues

None.

#### Additional Comments

**As directed by the EPA TOM, a Tier 3 data review was performed.**

The laboratory gravimetric data were evaluated for quality control (QC) criteria and errors in calculations and checked against the raw data supplied by Chester LabNet. No errors were found.

The Hayden air monitoring station analytes and contract required quantitation limits (CRQLs) provided in Table 1A are from the Statement of Work, dated August 10, 2006, Table 1. The Winkleman air monitoring station analytes and CRQLs provided in Table 1A are from the Statement of Work, dated August 10, 2006, Table 2.

ESAT could not check calculations from instrument raw data counts to final results due to the complexity of the calculations and due to insufficient information about the instrument software algorithms. This difficulty is noted in analytical method IO-3.3 and by the laboratory. The  $\mu\text{g}/\text{filter}$  and  $\mu\text{g}/\text{meter}^3$  ( $\mu\text{g}/\text{m}^3$ ) concentrations were recalculated as per laboratory instructions. (See Attachment 1.)

Results reported in  $\mu\text{g}/\text{m}^3$  are calculated using a standard  $24\pm 2.4$  cubic meter sample size. No adverse effect on data quality is expected.

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:

- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1, Selection, Preparation and Extraction of Filter Material*, June 1999;
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.3, Determination of Metals in Ambient Particulate Matter Using X-Ray Fluorescence (XRF) Spectroscopy*, June 1999;
- *Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II: Part 1, Ambient Air Quality Monitoring Program Quality System Development*, EPA-454/R-98-004, August 1998;
- *Statement of Work; 10 August 2006; Project: Perform Ambient Air Tests on Air Filters Gathered from the ASARCO Hayden Plant Site, AZ. Purchase Order: W91238-06-P-TBD; Issued by USACE Sacramento District*;
- *Standard Operating Procedure XR-006.01; Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex 771); CHESTER LabNet*, August 6, 2003; and
- *Region 9 Standard Operating Procedure 906, Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*.

## 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 Preparation and Weighing	Yes	
3.	Calibration	Yes	
4.	Blank	Yes	
5.	Standard Reference Materials (SRM)	Yes	
6.	Replicate Sample Analysis	No	A
7.	Matrix Spike Sample Analysis	N/A	
8.	Field Duplicate Sample Analysis	N/A	
9.	Sample Quantitation	Yes	
10.	Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

- A. The following results are estimated and are flagged "J" in Table 1A because of replicate results outside the ESAT  $\pm 35$  relative percent difference (RPD) QC limit. Replicate results that do not meet the  $\pm 35$  RPD criterion for precision are listed below.

<u>Sample</u>	<u>Analyte</u>	<u>Replicate RPD and uncertainty</u>
MY-091107	Antimony	-42.1 $\pm$ 42.5
MY-091107	Manganese	-41.8 $\pm$ 14.8

Results for analytes listed above in sample MY-091107 are considered quantitatively uncertain.

*Replicate analyses demonstrate the analytical precision obtained for each sample matrix. The imprecision between replicate results may be due to sample non-homogeneity or poor laboratory technique.*



## Attachment 1

Gretchen-

Here's what I dug up to answer the questions. I had to zip the files since your e-mail server wouldn't allow the size of e-mail with uncompressed files.

Gravimetry-

I have attached two files that contain all the gravimetry for the project. They include the temperature and humidity data.

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XRF

The calibration and blank data are attached for the two instruments that were used to analyze the samples. XRF spectrometers are very stable and do not require calibration very often. The blank data is not specific to this project, but is from filters from the same lot of filters used for the Asarco project.

We do not guarantee that the reported MDLs will meet the CRQLs. This was discussed when we were bidding on the project. The samples are analyzed for the counting times required to achieve the required detection limits on interference free samples, but corrections are made for various reasons which can cause the detection limit to rise to above the CRQL.

I e-mailed the raw data for reports 06-363 and 07-021 on June 8. I am resending it in this e-mail.

As for the sample calculation, it isn't really possible to calculate the ug/cm2 concentrations by hand due to the complexity of XRF. The calculations to get to ug/filter and ug/m3 are quite easy; multiply the ug/cm2 number by the deposit area (11.3) for ug/filter and divide the ug/filter concentration by the volume (usually 24) to get ug/m3. I can send you our XRF SOP if you want to take a stab at calculating the ug/cm2 concentration.

Hopefully this is enough information. If Rose needs anything else, maybe it would be best if she could call me directly so I understand exactly what she wants.

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## TABLE 1B

### DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

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- UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.



ANALYTICAL RESULTS

Case No. : None  
 Site : Asarco Hayden  
 Lab : Chester LabNet  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : April 17, 2008

SDG No. : 07-325

Table 1A

QUALIFIED DATA  
 Concentration in ug / m3

Analysis Type : PM10 Teflon Air Filters for Select  
 Total Metals by XRF

Station Location :		Hayden			Hayden			Hayden			Hayden			CRQL				
Sample ID :		MY-083007			MY-090507			MY-091107			MY-091707							
Filter Lot # :		7129003			7129003			7129003			7129003							
Collection Date :		8/30/2007			9/5/2007			9/11/2007			9/17/2007							
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com									
ANTIMONY	0.0102 ± 0.0030			* 0.0000 ± 0.0024			0.0120 ± 0.0032	J	A	* 0.0014 ± 0.0025			4					
ARSENIC	0.1794 ± 0.0202			0.0071 ± 0.0011			0.1894 ± 0.0213			0.0251 ± 0.0030			0.0008					
BARIUM	0.0287 ± 0.0052			* 0.0029 ± 0.0043			0.0403 ± 0.0063			* 0.0076 ± 0.0041			0.52					
CADMIUM	0.0351 ± 0.0045			* 0.0002 ± 0.0018			0.0324 ± 0.0043			* 0.0032 ± 0.0019			0.0037					
CHROMIUM	0.0025 ± 0.0007			* 0.0014 ± 0.0007			* 0.0016 ± 0.0007			* 0.0006 ± 0.0007			0.00033					
COBALT	* 0.0000 ± 0.0014			* 0.0000 ± 0.0015			* 0.0000 ± 0.0017			* 0.0000 ± 0.0013			0.00069					
COPPER	3.677 ± 0.4112			0.6116 ± 0.0685			3.697 ± 0.4134			0.3699 ± 0.0415			0.75					
MANGANESE	0.0072 ± 0.0011			0.0344 ± 0.0046			0.0193 ± 0.0030	J	A	0.0328 ± 0.0044			0.051					
NICKEL	* 0.0000 ± 0.0009			* 0.0000 ± 0.0008			* 0.0000 ± 0.0010			* 0.0000 ± 0.0008			0.0040					
SELENIUM	0.0838 ± 0.0094			0.0104 ± 0.0012			0.0551 ± 0.0062			0.0062 ± 0.0008			1.60					
SILVER	0.0175 ± 0.0028			* 0.0022 ± 0.0017			* 0.0034 ± 0.0021			* 0.0000 ± 0.0017			0.079					
VANADIUM	* 0.0022 ± 0.0014			0.0149 ± 0.0020			* 0.0022 ± 0.0014			0.0074 ± 0.0013			0.40					
PM10 Net Mass	35.00 ± 3.525			43.54 ± 4.374			40.58 ± 4.080			41.29 ± 4.1501			50					

\* Concentration is less than three times the uncertainty

Station Location :		Winkelman			Winkelman			Winkelman			Winkelman			CRQL				
Sample ID :		HS-082407			HS-090507			HS-091107			HS-091707							
Filter Lot # :		7129003			7129003			7129003			7129003							
Collection Date :		8/24/2007			9/5/2007			9/11/2007			9/17/2007							
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com									
ANTIMONY	* 0.0000 ± 0.0008			0.0027 ± 0.0009			* 0.0007 ± 0.0008			* 0.0000 ± 0.0008			4					
ARSENIC	0.0079 ± 0.0012			* 0.0017 ± 0.0007			* 0.0000 ± 0.0006			* 0.0000 ± 0.0006			0.0008					
BARIUM	* 0.0051 ± 0.0023			* 0.0021 ± 0.0023			* 0.0000 ± 0.0017			0.0108 ± 0.0032			0.52					
CADMIUM	0.0024 ± 0.0007			* 0.0000 ± 0.0006			* 0.0003 ± 0.0006			* 0.0000 ± 0.0006			0.00083					
CHROMIUM	* 0.0001 ± 0.0004			* 0.0000 ± 0.0004			* 0.0000 ± 0.0004			* 0.0002 ± 0.0005			0.00033					
COBALT	* 0.0000 ± 0.0010			* 0.0000 ± 0.0010			* 0.0014 ± 0.0008			* 0.0000 ± 0.0012			0.00069					
COPPER	0.3176 ± 0.0356			0.2216 ± 0.0249			0.0191 ± 0.0023			0.0555 ± 0.0064			0.75					
MANGANESE	0.0096 ± 0.0012			0.0129 ± 0.0021			0.0022 ± 0.0005			0.0307 ± 0.0041			0.051					
NICKEL	* 0.0000 ± 0.0007			* 0.0000 ± 0.0007			* 0.0000 ± 0.0007			* 0.0001 ± 0.0007			0.0040					
SELENIUM	0.0024 ± 0.0004			0.0041 ± 0.0006			* 0.0000 ± 0.0003			* 0.0000 ± 0.0003			1.60					
SILVER	0.0026 ± 0.0007			0.0020 ± 0.0006			* 0.0011 ± 0.0006			* 0.0008 ± 0.0006			0.079					
VANADIUM	0.0017 ± 0.0006			0.0028 ± 0.0007			* 0.0000 ± 0.0005			* 0.0038 ± 0.0009			0.40					
PM10 Net Mass	19.62 ± 2.006			22.71 ± 2.309			6.042 ± 0.7339			32.29 ± 3.256			50					

\* Concentration is less than three times the uncertainty

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit

N/A - Not Applicable

NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00905068

DATE: January 27, ~~2006~~ ~~January 27, 2006~~

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	34768
SDG No.:	MY29L5
Laboratory:	CompuChem (LIBRTY)
Analysis:	Total Metals plus Boron, Molybdenum, and Cyanide
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	November 16 and 17, 2005
Reviewer:	Kendra DeSantolo, 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

| 00905068-6139/34768/ [MY29L5 RPT.doc](#) ~~MY29L5 RPT~~



## Data Validation Report

Case No.: 34768  
SDG No.: MY29L5  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Kendra DeSantolo, ESAT/LDC  
Date: January 23, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY29L5 through MY29L9, MY29M0 through MY29M5, MY29M7, MY29M8, MY29M9, MY29N1, MY29N2, MY29N3, MY29N5, MY29N6, and MY29N7  
Concentration and Matrix: Medium Concentration Soil  
Analysis: Total Metals plus Boron, Molybdenum, and Cyanide  
SOW: ILM05.3 and Modified Analysis Request (MAR) 1318.3  
Collection Date: November 16 and 17, 2005  
Sample Receipt Date: November 23, 2005  
Preparation Date: November 28 and 29, 2005  
Analysis Date: November 28, 29, and 30, 2005

#### Field QC

Field Blanks (FB): Not provided  
Equipment Blanks (EB): Not provided  
Background Samples (BG): Not provided  
Field Duplicates (D1): MY29L6 and MY29L7

#### Laboratory QC

Method Blanks & Associated Samples: PBS and samples listed above  
Matrix Spike: MY29M0S  
Duplicates: MY29M0D  
ICP Serial Dilution: MY29M0L

Analysis: Total Metals plus Boron, Molybdenum, and Cyanide

<u>Analyte</u>	<u>Sample Preparation and Digestion/Distillation Date</u>	<u>Analysis Date</u>
ICP-AES Metals	November 28, 2005	November 28, 2005
Mercury	November 28, 2005	November 29, 2005
Cyanide	November 29, 2005	November 30, 2005
Percent Solids	November 28, 2005	November 29, 2005

#### CLP PO Action

1. The CRQL standard (CRI) was not analyzed at the contract required quantitation limits (CRQLs) specified in MAR Modification Reference Number 1318.3 for the metal analytes.
2. The laboratory indicates in the SDG Narrative that the matrix spike sample was spiked at twice the concentration specified in the SOW.

## Sampling Issues

1. Cyanide was not listed on the Traffic Report/Chain of Custody (TR/COC) report forms. Region 9 instructed the laboratory to follow the scheduling and analyze for cyanide.
2. The sampler did not provide a signature in the Sampler Signature block on the TR/COC record forms. No adverse effect on data quality is expected.
3. The TR/COC analysis key indicates analysis for Br. The laboratory analyzed for boron (B) as specified in MAR 1318.3.

## Additional Comments

The samples of this SDG were analyzed for CLP total metals plus total boron and total molybdenum by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1318.3. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide was analyzed by the CLP spectrophotometric method.

MAR 1318.3 requires CRQLs that differ from the standard CRQLs provided in the SOW. See Table 1A for the required modified CRQLs.

The laboratory informed Region 9 that the current method detection limit (MDL) for selenium was 0.34 mg/kg. MAR 1318.3 required a 0.30 mg/kg CRQL for selenium. Region 9 allowed the laboratory to increase the sample size to 2 grams to achieve the ICP-AES CRQLs required by MAR 1318.3.

The laboratory indicated in the SDG Narrative that the matrix spike sample was inadvertently spiked at two times the required concentration (“double spiked”). The effect on data quality is not known.

The laboratory diluted and reanalyzed all samples, except MY29N2, at a 3 time or 5 time dilution to stay within the instrument’s linear range for calcium, copper, iron, manganese, and zinc. No adverse effect on data quality is expected.

The laboratory indicated in the SDG Narrative that the results from the diluted analysis for manganese in sample MY29M5 and calcium in sample MY29M9 were below the 25% cutoff for these analytes specified in the SOW. Since the diluted results were less than the linear range of the instrument and greater than the continuing calibration verifications (CCVs) concentrations for the respective analytes, 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), Modification Reference Number: 1318.3, Title: Lowered CRQLs with the addition of B and Mo, November 17, 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 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	No	B
	a. Initial		
	b. Initial and Continuing Calibration Verification		
	c. CRQL Check Standard (CRI)		
4.	Blanks	Yes	C
5.	ICP Interference Check Sample (ICS)	No	D
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	No	E
9.	ICP Serial Dilution Analysis	No	F
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	No	G
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 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.*

- B. The following results are estimated and flagged "J" in Table 1A because the CRQL standard (CRI) was not analyzed at the modified CRQLs specified in MAR 1318.3.

- X Antimony in samples MY29L5, MY29L6, MY29M3, MY29M4, MY29M8, MY29N1, and MY29N2
- X Mercury in samples MY29L6, MY29L7, MY29L9, MY29M3, MY29M5, MY29M9, MY29N3, and MY29N5
- X Selenium in samples MY29M7 and MY29N1
- X Thallium in samples MY29L6, MY29L7, and MY29M5

The SOW requires a CRI be analyzed to verify the instrument can achieve the specified CRQLs. Results above the MDL but less than two times the CRQL are considered quantitatively uncertain. The results reported for the analytes listed above in the samples listed above are estimated.

*The inorganic SOW specifies that the laboratory must analyze a CRI standard immediately following the initial calibration verification (ICV), at the beginning, end, and after every 20 analytical samples for each analytical run in order to verify linearity near the CRQL.*

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

X Sodium in samples MY29L6, MY29L7, MY29L8, MY29M0, MY29M1, MY29M3, MY29M4, MY29N5, MY29M7, MY29M8, MY29M9, MY29N1, MY29N2, MY29N3, and MY29N5

The value for sodium (66.3 mg/kg) in preparation blank sample PBS is 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.

*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 are flagged "J" in Table 1A due to possible ICP interelement interference problems.

X Arsenic in samples MY29M0 and MY29M5  
X Cadmium in samples MY29L5, MY29L7, MY29L8, MY29L9, MY29M0, MY29M1, MY29M3 through MY29M5, MY29M7 through MY29M9, MY29N1, MY29N2, MY29N3, and MY29N5 through MY29N7  
X Thallium in samples MY29N3 and MY29N5

Results for arsenic, cadmium, and thallium in the samples listed above were reported from an undiluted analysis that contained copper and iron concentrations greater than the true value specified for the ICP interference check sample (ICS). Therefore, the applied interelement correction (IEC) factor may not compensate sufficiently for the interference. The results for the above listed analytes may be biased low.

*The ICP ICS solutions A and AB are analyzed to determine the effects of high concentrations of interfering elements on each analyte determined by ICP. Solution A consists of the interferents (Al, Ca, Fe, and Mg), and Solution AB consists of the analytes mixed with the interferents.*

*When the estimated concentration produced by the interfering element is greater than twice the CRQL and also is greater than 10% of the reported concentration of the affected element, the results of the affected elements are estimated.*

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

X Antimony, arsenic, beryllium, cadmium, cobalt, molybdenum, nickel, and selenium in all samples

Matrix spike recoveries for these analytes in QC sample MY29M0S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for each analyte are presented below and are based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Antimony	3	-97
Arsenic	69	-31
Beryllium	72	-28
Cadmium	71	-29
Cobalt	74	-26
Molybdenum	67	-33
Nickel	72	-28
Selenium	69	-31

Results above the MDL are considered quantitatively uncertain. Results reported for antimony, arsenic, beryllium, cadmium, cobalt, molybdenum, nickel, and selenium in all samples 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 analytes (except silver) 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 results were obtained.

Analyte	Post-Digestion Spike, % Recovery
Antimony	46
Arsenic	108
Beryllium	18
Cadmium	122
Cobalt	78
Molybdenum	78
Nickel	77
Selenium	397

Since the post-digestion spike recovery was acceptable for arsenic, cadmium, cobalt, molybdenum, and nickel, the low pre-digestion spike recovery result obtained for these analytes may indicate sample non-homogeneity, poor laboratory technique or matrix effects which may interfere with accurate analysis, depressing the analytical result.

Since both the post- and pre-digestion spikes did not meet the QC criteria for antimony and beryllium, matrix effects may be present in the sample digestate which may depress the analyte signal during analysis. The selenium post- and pre-digestion spikes did not meet the QC criteria due to possible sample digestate matrix effects which may interfere with the analyte signal during analysis.

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

F. The following results are estimated and flagged "J" or "UJ" in Table 1A because ICP serial dilution results are outside method QC limits.

X Aluminum, arsenic, barium, boron, cadmium, chromium, cobalt, lead, magnesium, molybdenum, nickel, and vanadium in all samples

The percent difference for the ICP serial dilution analysis of sample MY29M0L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Aluminum	+12
Arsenic	+13
Barium	+11
Boron	+45
Cadmium	+12
Chromium	+17
Cobalt	+20
Lead	+21
Magnesium	+16
Molybdenum	+17
Nickel	+22
Vanadium	+16

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The results for the diluted sample were higher than the original. Therefore, the reported sample results may be biased low.

*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.*

G. A relative percent difference (RPD) of 48 was obtained for calcium in the analysis of field duplicate pair samples MY29L6 and MY29L7. Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\sqrt{35}$  RPD criterion 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, sample non-homogeneity, 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.

**ANALYTICAL RESULTS**

Case No. : 34768                      SDG No. : MY29L5  
 Site : ASARCO  
 Lab : CompuChem (LIBRTY)  
 RGviewer : Kendra DeSantolo, ESAT/LDC  
 Date : January 23, 2006

**Table 1A**

**QUALIFIED DATA**                      **Analysis Type :** Medium Concentration Soil Samples For  
**Concentration in mg/Kg (Dry Weight)**                      Total Metals plus Boron, Molybdenum, and Cyanide

Station Location :	SPW-SED-12AS-111605			SPW-SED-11AS-111605			SPW-SED-11AD-111605			SPW-SED-14AS-111605			SPW-SED-13AS-111605			SPW-SED-01AS-111605			SPW-SED-06AS-111605		
Sample ID :	MY29L5			MY29L6 D1			MY29L7 D1			MY29L8			MY29L9			MY29M0			MY29M1		
Collection Date :	11/16/2005			11/16/2005			11/16/2005			11/16/2005			11/16/2005			11/16/2005			11/16/2005		
PARAMETER	Result	Val	Com																		
ALUMINUM	16000	J	F	3580	J	F	3850	J	F	6560	J	F	11400	J	F	18500	J	F	13400	J	F
ANTIMONY	1.0	J-	BE	0.97	J-	BE	2.1	J-	E	1.2	J-	E	1.4	J-	E	1.2	J-	E	1.5	J-	E
ARSENIC	14.3	J	EF	17.7	J	EF	19.0	J	EF	9.5	J	EF	13.5	J	EF	23.1	J	DEF	28.9	J	EF
BARIIUM	41.1L	J	AF	27.8L	J	AF	31.2L	J	AF	36.9L	J	AF	53.9L	J	AF	57.5L	J	AF	59.6L	J	AF
BERYLLIUM	0.26U	J	E	0.27U	J	E	0.27U	J	E	0.26U	J	E									
CADMIUM	2.6	J	DEF	2.2	J	EF	2.3	J	DEF	1.6	J	DEF	1.9	J	DEF	2.9	J	DEF	4.8	J	DEF
CALCIUM	54500			13500		G	8280		G	33000			43600			65200			56100		
CHROMIUM	17.3	J	F	10.9	J	F	11.0	J	F	9.0	J	F	16.0	J	F	18.2	J	F	15.0	J	F
COBALT	26.1	J	EF	7.1	J	EF	8.2	J	EF	9.8	J	EF	17.4	J	EF	30.2	J	EF	20.1	J	EF
COPPER	6060			2860			3050			2230			5170			10500			7050		
IRON	26900			11400			13100			12900			26900			35200			24700		
LEAD	35.6	J	F	62.6	J	F	64.6	J	F	38.6	J	F	65.3	J	F	51.5	J	F	129	J	F
MAGNESIUM	17900	J	F	2680	J	F	2880	J	F	6080	J	F	11000	J	F	17600	J	F	13500	J	F
MANGANESE	481			98.3			109			196			355			676			406		
MERCURY	0.051U			0.061	J	B	0.068	J	B	0.021L	J	A	0.058	J	B	0.050L	J	A	0.14		
NICKEL	31.3	J	EF	8.2L	J	AEF	8.8L	J	AEF	11.5	J	EF	24.4	J	EF	32.5	J	EF	25.3	J	EF
POTASSIUM	1040			1180			1260			1250			1320			1270			1590		
SELENIUM	0.15U	J	E	0.76	J	E	0.80	J	E	0.65	J	E	0.15U	J	E	0.16U	J	E	0.15U	J	E
SILVER	1.2			1.3			1.5			1.1			1.3			3.1			3.9		
SODIUM	264			250U		C	250U		C	251U		C	339			259U		C	252U		C
THALLIUM	1.7			0.39	J	B	0.48	J	B	0.81			1.3			1.1			1.4		
VANADIUM	64.7	J	F	18.8	J	F	20.9	J	F	30.4	J	F	49.9	J	F	65.4	J	F	51.0	J	F
ZINC	211			93.9			104			95.1			200			270			256		
CYANIDE	2.5U			2.5U			2.5U			2.5U			2.6U			2.6U			2.5U		
MOLYBDENUM	35.1	J	EF	28.6	J	EF	32.7	J	EF	41.2	J	EF	39.0	J	EF	64.2	J	EF	32.7	J	EF
BORON	2.4	J	F	1.1	J	F	1.3	J	F	1.7	J	F	3.1	J	F	3.1	J	F	2.6	J	F
PERCENT SOLIDS	98.8%			99.8%			99.8%			99.6%			97.7%			96.5%			99.4%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

ANALYTICAL RESULTS

Case No. : 34768 SDG No. : MY29L5  
 Site : ASARCO  
 Lab : CompuChem (LIBRTY)  
 Reviewer : Kendra DeSantolo, ESAT/LDC  
 Date : January 23, 2006

Table 1A

QUALIFIED DATA  
 Concentration in mg/Kg (Dry Weight)

Analysis Type : Medium Concentration Soil Samples For  
 Total Metals plus Boron, Molybdenum, and Cyanide

Station Location :	SPW-SED-03AS-111605			SPW-SED-05AS-111605			SPW-SED-04AS-111605			SPW-SED-02AS-111605			SPW-SED-15AS-111705			SPW-SED-07AS-111705			SPW-SED-08AS-111705		
Sample ID :	MY29M2			MY29M3			MY29M4			MY29M5			MY29M7			MY29M8			MY29M9		
Collection Date :	11/16/2005			11/16/2005			11/16/2005			11/16/2005			11/17/2005			11/17/2005			11/17/2005		
PARAMETER	Result	Val	Com																		
ALUMINUM	16000	J	F	10200	J	F	18100	J	F	17800	J	F	11800	J	F	14000	J	F	11800	J	F
ANTIMONY	1.5	J-	E	0.99	J-	BE	1.0	J-	BE	2.0	J-	E	1.5	J-	E	1.0	J-	BE	1.3	J-	E
ARSENIC	23.2	J	EF	24.7	J	EF	15.1	J	EF	29.7	J	DEF	16.4	J	EF	5.5	J	EF	18.3	J	EF
BARIUM	68.1L	J	AF	70.0L	J	AF	39.1L	J	AF	66.6L	J	AF	70.4L	J	AF	66.8L	J	AF	94.0	J	F
BERYLLIUM	0.26U	J	E	0.26U	J	E	0.27U	J	E	0.27U	J	E	0.26U	J	E	0.26U	J	E	0.26U	J	E
CADMIUM	3.5	J	EF	3.3	J	DEF	2.2	J	DEF	3.4	J	DEF	2.1	J	DEF	0.94	J	DEF	3.8	J	DEF
CALCIUM	79500			46500			51100			61900			34500			35200			36500		
CHROMIUM	16.6	J	F	13.1	J	F	17.2	J	F	18.3	J	F	13.9	J	F	9.8	J	F	11.6	J	F
COBALT	22.9	J	EF	14.6	J	EF	26.7	J	EF	29.7	J	EF	16.4	J	EF	15.8	J	EF	16.6	J	EF
COPPER	7230			6260			5970			12500			4920			926			4950		
IRON	29300			17700			27900			36200			22200			17500			19900		
LEAD	118	J	F	89.7	J	F	30.9	J	F	67.0	J	F	60.8	J	F	24.1	J	F	90.4	J	F
MAGNESIUM	17100	J	F	9470	J	F	17800	J	F	17300	J	F	10700	J	F	11100	J	F	11700	J	F
MANGANESE	482			291			599			622			369			451			358		
MERCURY	0.12			0.076	J	B	0.020L	J	A	0.093	J	B	0.047L	J	A	0.051U			0.082	J	B
NICKEL	29.4	J	EF	19.8	J	EF	31.1	J	EF	32.2	J	EF	18.5	J	EF	14.4	J	EF	18.2	J	EF
POTASSIUM	1480			2030			1150			1470			1570			1080			2150		
SELENIUM	0.15U	J	E	0.32	J	E	0.15U	J	E	1.2	J	E	0.22	J	BE	0.15U	J	E	0.97	J	E
SILVER	3.8			3.4			1.4			4.9			2.3			0.29			3.3		
SODIUM	265			252U		C	256U		C	256U		C	254U		C	253U		C	253U		C
THALLIUM	2.0			0.85			1.6			0.62	J	B	1.4			2.1			1.3		
VANADIUM	65.9	J	F	38.1	J	F	69.6	J	F	63.3	J	F	50.3	J	F	53.5	J	F	43.7	J	F
ZINC	264			207			192			283			176			71.9			250		
CYANIDE	2.5U			2.5U			2.6U			2.6U			2.5U			2.5U			2.5U		
MOLYBDENUM	31.4	J	EF	38.5	J	EF	35.4	J	EF	86.0	J	EF	41.8	J	EF	3.8	J	EF	32.7	J	EF
BORON	4.0	J	F	3.2	J	F	2.7	J	F	3.5	J	F	3.5	J	F	2.4	J	F	3.2	J	F
PERCENT SOLIDS	98.8%			99.2%			97.8%			97.6%			98.6%			98.7%			98.8%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 34768                      SDG No. : MY29L5  
 Site : ASARCO  
 Lab : CompuChem (LIBRTY)  
 Reviewer : Kendra DeSantolo, ESAT/LDC  
 Date : January 23, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/Kg (Dry Weight)**

**Analysis Type :** Medium Concentration Soil Samples For  
 Total Metals plus Boron, Molybdenum, and Cyanide

PARAMETER	SPW-SED-09AS-111705			SPW-SED-10AS-111705			PHW-SED-01AS-111705			PHW-SED-02BS-111705			PHW-SED-03AS-111705			PHW-SED-04AS-111705			MDL		
	Result	Val	Com	Result	Val	Com															
ALUMINUM	11400	J	F	10200	J	F	15500	J	F	13800	J	F	15600	J	F	16200	J	F	2.0		
ANTIMONY	0.96	J-	BE	0.85	J-	BE	3.3	J-	E	3.2	J-	E	2.1	J-	E	2.2	J-	E	0.20		
ARSENIC	13.0	J	EF	9.0	J	EF	35.7	J	EF	37.9	J	EF	36.4	J	EF	25.4	J	EF	0.30		
BARIUM	95.8	J	F	74.2L	J	AF	70.5L	J	AF	72.8L	J	AF	89.3	J	F	84.8	J	F	0.072		
BERYLLIUM	0.26U	J	E	0.26U	J	E	0.27U	J	E	0.28U	J	E	0.26U	J	E	0.26U	J	E	0.012		
CADMIUM	2.0	J	DEF	1.8	J	DEF	3.7	J	DEF	4.1	J	DEF	3.9	J	DEF	3.4	J	DEF	0.032		
CALCIUM	22600			30100			57800			36400			50400			51400			21.8		
CHROMIUM	9.8	J	F	10.1	J	F	19.1	J	F	23.9	J	F	20.3	J	F	19.9	J	F	0.079		
COBALT	13.6	J	EF	13.7	J	EF	33.2	J	EF	40.6	J	EF	27.9	J	EF	29.8	J	EF	0.062		
COPPER	3150			2440			6030			11100			8270			6070			0.17		
IRON	17700			16300			43300			58900			33700			35000			3.1		
LEAD	57.8	J	F	45.4	J	F	130	J	F	140	J	F	110	J	F	104	J	F	0.19		
MAGNESIUM	8430	J	F	8920	J	F	14300	J	F	13300	J	F	14200	J	F	16000	J	F	2.5		
MANGANESE	333			332			502			505			563			576			0.73		
MERCURY	0.050L	J	A	0.050U			0.065	J	B	0.076	J	B	0.11			0.030L	J	A	0.020		
NICKEL	14.3	J	EF	15.3	J	EF	32.6	J	EF	39.7	J	EF	32.5	J	EF	33.9	J	EF	0.16		
POTASSIUM	2060			1460			1480			1320			1890			1630			3.7		
SELENIUM	0.18	J	BE	0.15U	J	E	0.56	J	E	2.0	J	E	0.67	J	E	0.15U	J	E	0.34		
SILVER	2.4			1.2			3.1			3.9			4.0			2.1			0.15		
SODIUM	253U		C	251U		C	255U		C	265U		C	292			283			20.6		
THALLIUM	0.93			1.3			2.0	J	D	1.7	J	D	1.4			2.1			0.33		
VANADIUM	40.8	J	F	42.6	J	F	68.9	J	F	63.2	J	F	67.0	J	F	72.7	J	F	0.069		
ZINC	170			121			510			497			478			525			0.38		
CYANIDE	2.5U			2.5U			2.6U			2.7U			2.5U			2.5U			0.26		
MOLYBDENUM	22.4	J	EF	13.3	J	EF	40.2	J	EF	48.8	J	EF	65.8	J	EF	45.2	J	EF	0.32		
BORON	3.3	J	F	2.6	J	F	2.9	J	F	3.4	J	F	3.1	J	F	2.9	J	F	0.10		
PERCENT SOLIDS	98.8%			99.5%			98.0%			94.2%			98.2%			98.7%			N/A		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 34768                      SDG No. : MY29L5  
 Site : ASARCO  
 Lab : CompuChem (LIBRTY)  
 Reviewer : Kendra DeSantolo, ESAT/LDC  
 Date : January 23, 2006

Table 1A

**QUALIFIED DATA**  
**Concentration in mg/Kg (Dry Weight)**

**Analysis Type :** Medium Concentration Soil Samples For  
 Total Metals plus Boron, Molybdenum, and Cyanide

Sample ID : CRQL																						
PARAMETER	Result	Val	Com																			
ALUMINUM	50.0																					
ANTIMONY	0.99																					
ARSENIC	0.39																					
BARIUM	161.3																					
BERYLLIUM	0.52																					
CADMIUM	0.40																					
CALCIUM	500																					
CHROMIUM	0.40																					
COBALT	9.7																					
COPPER	16.6																					
IRON	200																					
LEAD	7.7																					
MAGNESIUM	500																					
MANGANESE	100																					
MERCURY	0.050																					
NICKEL	18.2																					
POTASSIUM	500																					
SELENIUM	0.30																					
SILVER	0.50																					
SODIUM	500																					
THALLIUM	0.70																					
VANADIUM	2.0																					
ZINC	38.9																					
CYANIDE	2.5																					
MOLYBDENUM	2.0																					
BORON	0.50																					

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00905090

DATE: May 24, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None
Case No.:	35036
SDG No.:	MY2DM3
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Total Metals and Total Cyanide
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 20, 21, and 22, 2006
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: Cynthia Gurley, 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.: 35036  
SDG No.: MY2DM3  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: May 24, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2DM3 through MY2DM9, MY2DN0 through MY2DN9, MY2DP0, MY2DP1, and MY2DP2  
Concentration and Matrix: Low and Medium Concentration Soil  
Analysis: CLP Total Metals and Total Cyanide  
SOW: ILM05.3 and Modified Analysis Request 1337.0  
Collection Date: February 20, 21, and 22, 2006  
Sample Receipt Date: February 25, 2006  
Preparation Date: March 1 and 2, 2006  
Analysis Date: March 6, 9, 16, 17, 18, and 23, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY2DN9 and MY2DP0

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Soil (PBS) and samples listed above  
Matrix Spike: MY2DP2S  
Duplicates: MY2DP2D  
ICP Serial Dilution: MY2DP2L

Analysis: Total Metals and Total Cyanide

<u>Analyte</u>	<u>Sample Preparation and Digestion/Distillation Date</u>	<u>Analysis Date</u>
ICP-AES Metals	March 2, 2006	March 16, 17, 18, and 23, 2006
Mercury	March 2, 2006	March 6, 2006
Cyanide	March 1, 2006	March 9, 2006
Percent Solids	March 2, 2006	March 3, 2006

#### CLP PO Action

None.

#### Sampling Issues

1. The Traffic Report/Chain of Custody (TR/COC) record form did not specify a sample to be used for laboratory quality control (QC). The laboratory selected sample MY2DP2 for laboratory QC analysis.

2. One of the coolers containing samples for this SDG arrived at the laboratory with a temperature of 6.8°C. This temperature exceeds the temperature of 4°± 2°C specified in the Statement of Work (SOW); however, no adverse effect on data quality is expected.

### Additional Comments

The SDG Narrative requires minor editing to correct sample receipt date and laboratory QC sample identity. A corrected SDG Narrative was requested from the laboratory but has not been received to date. Data quality is not likely to be affected and this report is considered final.

The samples in this SDG were analyzed for CLP total metals plus boron and molybdenum by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1337.0. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide was analyzed by the CLP spectrophotometric method.

The SDG Narrative states that Region 9 approved an increase in the contract required quantitation limit (CRQL) for selenium from 0.30 mg/kg to 0.50 mg/kg.

To achieve the modified mercury CRQL specified in MAR 1337.0, the laboratory increased the mercury analysis sample size from 0.2 g to 0.5 g as permitted by MAR 1337.0.

Samples MY2DM6 through MY2DN4 and MY2DN8 through MY2DP0 were analyzed from diluted samples due to high analyte concentrations or interference problems. No adverse effect on data quality is expected.

CADRE R-flagged copper results for samples MY2DN1 through MY2DN4, MY2DP0, and MY2DP1, analyzed on March 19, 2006, because the last of five CRI analyses exceeded the 180 percent expanded recovery criterion specified in the National Functional Guidelines (NFG). Since the reported copper data for the samples listed above were analyzed between acceptable CRI recoveries, the R flags for the copper data in the Table 1A were removed.

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: 1337.0, January 18, 2006;
- 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	B
5.	ICP Interference Check Sample (ICS)	No	C
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	No	D
9.	ICP Serial Dilution Analysis	No	E
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	No	F
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 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.*

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

X Beryllium in samples MY2DN0, MY2DN1, MY2DN2, and MY2DN4 through MY2DP0

X Molybdenum in sample MY2DN6

X Sodium in samples MY2DM7, MY2DN1, MY2DN7, and MY2DP2

The beryllium (0.059 mg/kg), molybdenum (0.11 mg/kg), and sodium (48.6 mg/kg) results in preparation blank PBS are greater than the 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.*

C. The following results are estimated and are flagged "J-" or "J+" in Table 1A due to possible ICP interelement interference problems.

- X Cadmium, selenium, and thallium in samples MY2DM3, MY2DM5 through MY2DM9, MY2DN1, MY2DN5, MY2DN7 through MY2DN9, MY2DP0, and MY2DP1
- X Arsenic in samples MY2DM7, MY2DM8, MY2DN1, MY2DN5, MY2DN8, MY2DN9, and MY2DP0
- X Chromium in samples MY2DM6 through MY2DM9, and MY2DN8 through MY2DP0
- X Zinc in sample MY2DN1

Results for cadmium, selenium, and thallium in the samples listed above were reported from an undiluted analysis that contained iron concentrations greater than the true value specified for the ICP interference check sample (ICS). Therefore, the applied interelement correction (IEC) factor may not compensate sufficiently for the interference. The results for cadmium may be biased high and false positives may exist. The results for selenium and thallium may be biased low and false negatives may exist.

Results for arsenic, chromium, and zinc in the samples listed above were reported from an undiluted analysis that contained copper concentrations greater than the true value specified for the ICP ICS. Therefore, the applied interelement correction (IEC) factor may not compensate sufficiently for the interference. The results for arsenic, chromium, and zinc in the samples listed above may be biased high and false positives may exist.

*The ICP ICS solutions A and AB are analyzed to determine the effects of high concentrations of interfering elements on each analyte determined by ICP. Solution A consists of the interferents (Al, Ca, Fe, and Mg), and Solution AB consists of the analytes mixed with the interferents.*

*When the estimated concentration produced by the interfering element is greater than twice the CRQL and also is greater than 10% of the reported concentration of the affected element, the results of the affected elements are estimated.*

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

- X Antimony, arsenic, boron, molybdenum, selenium, and zinc in all samples

Matrix spike recoveries for the samples listed above in QC sample MY2DP2S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for each analyte are presented below and are based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Antimony	66	-34
Arsenic	62	-38
Boron	43	-57
Molybdenum	60	-40
Selenium	71	-29
Zinc	65	-35

Results above the MDL are considered quantitatively uncertain. Results reported for the analytes listed above in all samples may be biased low.

According to the inorganic SOW, when the pre-digestion spike recovery results for ICP analytes (except silver) 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 results for sample MY2DP2A were obtained.

Analyte	Post-Digestion Spike, % Recovery
Antimony	77
Arsenic	90
Boron	54
Molybdenum	79
Selenium	82
Zinc	78

Since the post-digestion spike recoveries were acceptable, the low pre-digestion spike recovery results obtained for antimony, arsenic, molybdenum, selenium, and zinc may indicate sample non-homogeneity, poor laboratory technique, or matrix effects which may interfere with accurate analysis, depressing analytical results. Since both the post- and pre-digestion spikes for boron did not meet the QC criteria, matrix effects may be present in the sample digestate which may depress the analyte signal during analysis.

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

- E. The following results are estimated and flagged "J" or "UJ" in Table 1A because ICP serial dilution results are outside method QC limits.
- X Aluminum, barium, chromium, cobalt, iron, lead, magnesium, manganese, molybdenum, nickel, vanadium, and zinc in all samples

The percent difference for the ICP serial dilution analysis of sample MY2EW9L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Aluminum	+15
Barium	+13
Chromium	+14
Cobalt	+15
Iron	+15
Lead	+17
Magnesium	+16
Manganese	+15
Molybdenum	+15
Nickel	+13
Vanadium	+13
Zinc	+16

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The results for the diluted sample were higher than the original. Therefore, the reported sample results may be biased low.

*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.*

- F. A relative percent difference (RPD) of 42 was obtained for cadmium in the analysis of field duplicate pair samples MY2DN9 and MY2DP0. Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\leq 35$  RPD criterion 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, sample non-homogeneity, 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.



In Reference to  
Case: 35036 SDG No.: MY2DM3

Contract Laboratory Program  
REGIONAL/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call: May 24, 2006

Laboratory Name: CompuChem (LIBRTY)

Lab Contact: Alice Evens or Bob Meierer

Region: 9

Regional Contact: Steve Remaley, CLP PO

ESAT Reviewer: Stan Kott, ESAT/LDC

Call Initiated By:      Laboratory   X   Region

In reference to data for the following sample(s):

SDG No.: MY2DM3 for all samples

Summary of Questions/issues Discussed:

The following item was noted during the review of this sample delivery group (SDG) data package. Please respond within 4 days as specified in ILM05.3 Statement of Work (SOW), Exhibit B, Section 2, 2.2. Send response and resubmissions to

ICF International/Laboratory Data Consultants, Inc.,  
Environmental Services Assistance Team, USEPA Region 9 Laboratory  
1337 S. 46th Street, Building 201, Richmond, CA 94804, FAX 510 412-2304.

1. The SDG Narrative indicates samples were received on February 11 and 16, 2006. The sample log-in sheet indicates samples were received on February 25, 2006. Please review the data and provide a corrected SDG Narrative.
2. The SDG Narrative indicates sample MY2DM0 was selected for laboratory QC analysis. The QC summaries in the data package indicate sample MY2DP2 was selected for QC analysis. Please review the data and provide a corrected SDG Narrative.
3. The SOW specifies that percent solids samples be dried at 103-105°C. Please provide the oven temperature for the percent solids analyzed on March 2 and 3, 2006.

Summary of Resolution: To be determined.

\_\_\_\_\_  
Regional Contact Signature

\_\_\_\_\_  
Date of Resolution

ANALYTICAL RESULTS

Case No. : 35036 SDG No. : MY2DM3  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 24, 2006

Table 1A

QUALIFIED DATA  
 Concentration in mg/kg (Dry Weight)

Analysis Type : Low Concentration Soil Samples  
 For Total Metals and Cyanide

Station Location : HGC-10-SED-0-022006			HGC-20-SED-0-022006			HGC-33-SED-0-022106			HGC-30-SED-0-022106			SR77-01-SED-0-022106			KS-01-SED-0-022106			KS-02-SED-0-022106			
Sample ID : MY2DM3			MY2DM4			MY2DM5			MY2DM6			MY2DM7			MY2DM8			MY2DM9			
Collection Date : 2/20/2006			2/20/2006			2/21/2006			2/21/2006			2/21/2006			2/21/2006			2/21/2006			
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com									
ALUMINUM	10900	J	E	5470	J	E	32700	J	E	15400	J	E	12600	J	E	15700	J	E	6810	J	E
ANTIMONY	0.80L	J	AD	0.50L	J	AD	2.7	J	D	3.5	J	D	2.6	J	D	5.2	J	D	20.9	J	D
ARSENIC	3.7	J	D	1.3	J	D	13.2	J	D	81.1	J	D	39.1	J+	CD	224	J+	CD	345	J	D
BARIUM	115L	J	AE	58.5L	J	AE	313L	J	AE	150L	J	AE	112L	J	AE	144L	J	AE	66.4L	J	AE
BERYLLIUM	0.44L	J	A	0.34L	J	A	1.1L	J	A	0.45L	J	A	0.41L	J	A	0.44L	J	A	0.55U		
CADMIUM	0.52	J+	C	0.15L	J	A	1.3	J+	C	9.5	J+	C	8.3	J+	C	25.8	J+	C	3.2	J+	C
CALCIUM	15700			4800			63400			17600			60000			120000			73700		
CHROMIUM	14.3	J	E	5.4	J	E	39.2	J	E	11.6	J	CE	25.5	J	CE	6.7	J	CE	34.4	J	CE
COBALT	10.7	J	E	5.2L	J	AE	32.0	J	E	27.3	J	E	15.8	J	E	75.2	J	E	42.8	J	E
COPPER	391			154			1600			9320			8130			56200			18400		
IRON	17300	J	E	9630	J	E	49600	J	E	25900	J	E	27200	J	E	49000	J	E	197000	J	E
LEAD	19.8	J	E	6.6L	J	AE	53.4	J	E	191	J	E	231	J	E	253	J	E	547	J	E
MAGNESIUM	6590	J	E	2700	J	E	22900	J	E	9920	J	E	10400	J	E	12900	J	E	2080	J	E
MANGANESE	382	J	E	236	J	E	941	J	E	418	J	E	382	J	E	383	J	E	65.2L	J	AE
MERCURY	0.022L	J	A	0.021U			0.098L	J	A	0.25			0.15			0.49			1.5		
NICKEL	17.8L	J	AE	6.3L	J	AE	54.7	J	E	31.3	J	E	34.3	J	E	72.9	J	E	32.6	J	E
POTASSIUM	3500			1450			9110			4300			1440			1810			2940		
SELENIUM	0.50U	J-	CD	0.52U	J	D	1.2L	J-	ACD	3.6	J-	CD	3.3	J-	CD	15.4	J-	CD	86.9	J-	CD
SILVER	0.19L	J	A	0.52U			0.73L	J	A	4.8			6.2			13.1			34.6		
SODIUM	529			301L	J	A	2610			849			501U		B	385L	J	A	1140		
THALLIUM	0.71U	J-	C	0.56L	J	A	3.4	J-	C	0.69L	J-	AC	0.70U	J-	C	1.2	J-	C	5.0	J-	C
VANADIUM	32.6	J	E	17.0	J	E	105	J	E	46.3	J	E	42.9	J	E	84.2	J	E	105	J	E
ZINC	97.9	J	DE	57.8	J	DE	260	J	DE	406	J	DE	400	J	DE	1460	J	DE	120	J	DE
CYANIDE	2.5U			2.6U			6.9U			2.5U			2.5U			0.52L	J	A	2.6U		
MOLYBDENUM	7.0	J	DE	3.5	J	DE	23.2	J	DE	65.4	J	DE	37.6	J	DE	240	J	DE	1510	J	DE
BORON	6.0	J-	D	2.5	J-	D	22.6	J-	D	7.5	J-	D	3.3	J-	D	7.8	J-	D	0.93	J-	D
PERCENT SOLIDS	99.1%			95.9%			36.3%			98.8%			99.8%			98.4%			94.7%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DM3  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 24, 2006

**Table 1A**

**QUALIFIED DATA**                      **Analysis Type : Low Concentration Soil Samples**  
**Concentration in mg/kg (Dry Weight)**                      **For Total Metals and Cyanide**

Station Location :	KS-03-SED-0-022106			KS-04-SED-0-022106			KS-06-SED-0-022106			KS-08-SED-0-022106			KS-09-SED-90-022106			UP-01-SED-0-022106			WSC-08-SED-0-022106		
Sample ID :	MY2DN0			MY2DN1			MY2DN2			MY2DN3			MY2DN4			MY2DN5			MY2DN6		
Collection Date :	2/21/2006			2/21/2006			2/21/2006			2/21/2006			2/21/2006			2/21/2006			2/21/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com									
ALUMINUM	5500	J	E	3090	J	E	4720	J	E	10500	J	E	9720	J	E	21700	J	E	4640	J	E
ANTIMONY	12.2	J	D	14.5	J	D	14.5	J	D	13.0	J	D	9.9	J	D	0.94L	J	AD	0.21L	J	AD
ARSENIC	140	J	D	60.1	J+	CD	2.0U	J	D	114	J	D	91.6	J	D	10.9	J+	CD	2.1	J	D
BARIUM	161L	J	AE	46.4L	J	AE	40.6L	J	AE	69.8L	J	AE	67.4L	J	AE	99.0L	J	AE	49.2L	J	AE
BERYLLIUM	0.52U		B	0.53U		B	0.52U		B	1.1U			0.53U		B	0.52U		B	0.52U		B
CADMIUM	15.3			4.1	J+	C	17.9			7.7			25.9			1.7	J+	C	0.090L	J	A
CALCIUM	198000			22600			14800			35200			31900			84300			9940		
CHROMIUM	0.40U	J	E	0.40U	J	E	2.0U	J	E	0.82U	J	E	2.0U	J	E	21.9	J	E	8.1	J	E
COBALT	47.8	J	E	99.1	J	E	120	J	E	75.8	J	E	135	J	E	22.2	J	E	5.1L	J	AE
COPPER	56700			68200			192000			92800			133000			1540			38.4		
IRON	54100	J	E	227000	J	E	182000	J	E	146000	J	E	142000	J	E	26100	J	E	9630	J	E
LEAD	391	J	E	325	J	E	333	J	E	539	J	E	552	J	E	41.1	J	E	7.0L	J	AE
MAGNESIUM	5150	J	E	1880	J	E	3980	J	E	7790	J	E	7680	J	E	22000	J	E	2880	J	E
MANGANESE	183	J	E	73.5L	J	AE	223	J	E	242	J	E	313	J	E	797	J	E	149	J	E
MERCURY	0.42			0.13			0.066			0.25			0.17			0.053			0.020U		
NICKEL	54.2	J	E	78.5	J	E	78.9	J	E	68.0	J	E	113	J	E	27.9	J	E	7.8L	J	AE
POTASSIUM	1410			1180			1710			2430			2290			1670			869		
SELENIUM	32.2	J	D	67.6	J-	CD	101	J	D	111	J	D	83.2	J	D	0.94	J-	CD	0.50U	J	D
SILVER	29.6			27.6			35.7			63.4			30.9			0.81			0.50U		
SODIUM	261L	J	A	506U		B	262L	J	A	641			394L	J	A	250L	J	A	247L	J	A
THALLIUM	2.5			2.4	J-	C	5.1			2.1			5.9			0.40L	J-	AC	0.70U		
VANADIUM	26.9	J	E	31.1	J	E	31.7	J	E	53.8	J	E	48.5	J	E	71.8	J	E	26.5	J	E
ZINC	662	J	DE	278	J	CDE	1770	J	DE	630	J	DE	2100	J	DE	113	J	DE	27.6L	J	ADE
CYANIDE	2.5U			2.5U			2.5U			2.6U			2.6U			2.5U			2.5U		
MOLYBDENUM	488	J	DE	1060	J	DE	2280	J	DE	1490	J	DE	2060	J	DE	4.4	J	DE	2.0U	J	BDE
BORON	2.5	J-	D	0.51U	J-	D	0.50U	J-	D	6.6	J-	D	2.6	J-	D	3.2	J-	D	2.3	J-	D
PERCENT SOLIDS	99.4%			98.8%			99.5%			97.6%			97.9%			99.6%			99.8%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DM3  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 24, 2006

**Table 1A**

**QUALIFIED DATA**                      **Analysis Type : Low Concentration Soil Samples**  
**Concentration in mg/kg (Dry Weight)**                      **For Total Metals and Cyanide**

Station Location : WSC-18-SED-0-022106			PCON-05-SED-0-022106			PCON-06-SED-0-022106			PCON-X-SED-0-022106			UPA-01-SED-0-022206			UPA-07-SED-0-022206			MDL			
Sample ID : MY2DN7			MY2DN8			MY2DN9 D1			MY2DP0 D1			MY2DP1			MY2DP2						
Collection Date : 2/21/2006			2/21/2006			2/21/2006			2/21/2006			2/22/2006			2/22/2006						
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	5400	J	E	13200	J	E	15700	J	E	15200	J	E	30000	J	E	9760	J	E	3.1		
ANTIMONY	0.50L	J	AD	3.7	J	D	3.9	J	D	3.9	J	D	1.3	J	D	1.2	J	D	0.18		
ARSENIC	2.3	J	D	17.6	J+	CD	76.2	J+	CD	74.9	J+	CD	6.7	J	D	9.1	J	D	32.0		
BARIIUM	54.2L	J	AE	100L	J	AE	227	J	E	218	J	E	60.6L	J	AE	74.2L	J	AE	0.047		
BERYLLIUM	0.54U		B	0.53U		B	0.52U		B	0.52U		B	0.52U		B	0.42L	J	A	0.022		
CADMIUM	0.030L	J+	AC	2.6	J+	C	8.9	J+	CF	13.7	J+	CF	1.6	J+	C	0.92			0.011		
CALCIUM	11300			33600			69100			74000			14800			3540			7.1		
CHROMIUM	10.9	J	E	4.4	J	CE	5.2	J	CE	4.9	J	CE	7.3	J	E	11.8	J	E	0.13		
COBALT	8.0L	J	AE	42.8	J	E	29.2	J	E	28.5	J	E	29.1	J	E	11.8	J	E	0.033		
COPPER	65.8			25500			23300			20700			854			882			0.083		
IRON	13500		E	59200	J	E	36800	J	E	34600	J	E	33700	J	E	21100	J	E	2.3		
LEAD	10.4	J	E	96.7	J	E	257	J	E	277	J	E	36.3	J	E	41.1	J	E	0.11		
MAGNESIUM	3740	J	E	9410	J	E	14300	J	E	14300	J	E	17600	J	E	4720	J	E	0.88		
MANGANESE	270	J	E	360	J	E	540	J	E	522	J	E	686	J	E	276	J	E	0.035		
MERCURY	0.021U			0.11			0.28			0.27			0.031L	J	A	0.052			0.042		
NICKEL	10.9L	J	AE	35.1	J	E	39.7	J	E	38.7	J	E	17.5L	J	AE	12.8L	J	AE	0.092		
POTASSIUM	1020			4730			2090			2250			1330			2670			2.1		
SELENIUM	0.52U	J-	CD	13.1	J-	CD	13.5	J-	CD	12.7	J-	CD	0.95	J-	CD	0.75	J	D	0.35		
SILVER	0.52U			5.3			19.0			16.7			0.37L	J	A	0.51			0.067		
SODIUM	520U		B	760			254L	J	A	261L	J	A	799			501U		B	13.3		
THALLIUM	0.49L	J-	AC	1.6	J-	C	0.63L	J-	AC	0.70U	J-	C	0.71U	J-	C	0.87			0.40		
VANADIUM	34.0	J	E	60.6	J	E	66.9	J	E	65.3	J	E	86.3	J	E	41.6	J	E	0.023		
ZINC	35.1L	J	ADE	410	J	DE	518	J	DE	544	J	DE	99.3	J	DE	106	J	DE	0.18		
CYANIDE	2.6U			2.6U			2.5U			2.5U			2.5U			2.5U			0.16		
MOLYBDENUM	1.0L	J	ADE	383	J	DE	128	J	DE	129	J	DE	3.5	J	DE	15.9	J	DE	0.068		
BORON	1.5	J-	D	2.5	J-	D	5.3	J-	D	4.9	J-	D	1.9	J-	D	1.5	J-	D	0.031		
PERCENT SOLIDS	96.2%			98.0%			99.7%			99.5%			99.2%			99.8%			N/A		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DM3  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 24, 2006

Table 1A

**QUALIFIED DATA**  
 Concentration in mg/kg (Dry Weight)

Analysis Type : Low Concentration Soil Samples  
 For Total Metals and Cyanide

Sample ID : CRQL																						
PARAMETER	Result	Val	Com																			
ALUMINUM	50.0																					
ANTIMONY	0.99																					
ARSENIC	0.39																					
BARIUM	161.3																					
BERYLLIUM	0.52																					
CADMIUM	0.40																					
CALCIUM	500																					
CHROMIUM	0.40																					
COBALT	9.7																					
COPPER	16.6																					
IRON	200																					
LEAD	7.7																					
MAGNESIUM	500																					
MANGANESE	100																					
MERCURY	0.050																					
NICKEL	18.2																					
POTASSIUM	500																					
SELENIUM	0.50																					
SILVER	0.50																					
SODIUM	500																					
THALLIUM	0.70																					
VANADIUM	2.0																					
ZINC	38.9																					
CYANIDE	2.5																					
MOLYBDENUM	2.0																					
BORON	0.50																					

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00905090

DATE: May 18, 2006

SUBJECT: Review of Analytical Data, Tier 2

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None
Case No.:	35036
SDG No.:	MY2DP4
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Total Metals and Total Cyanide
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 22 and 23, 2006
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: Cynthia Gurley, 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.: 35036  
SDG No.: MY2DP4  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: May 18, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2DP4 through MY2DR3  
Concentration and Matrix: Low and Medium Concentration Soil  
Analysis: CLP Total Metals and Total Cyanide  
SOW: ILM05.3 and Modification Reference Number 1337.0  
Collection Date: February 22 and 23, 2006  
Sample Receipt Date: February 25, 2006  
Preparation Date: March 2 and 9, 2006  
Analysis Date: March 9, 10, 13, and 15, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY2DQ2 and MY2DQ3  
(D2): MY2DQ6 and MY2DQ7

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Soil (PBS) and samples listed above  
Matrix Spike: MY2DQ8S  
Duplicates: MY2DQ8D  
ICP Serial Dilution: MY2DQ8L

Analysis: CLP Total Metals and Total Cyanide

<u>Analyte</u>	<u>Sample Preparation and Digestion/Distillation Date</u>	<u>Analysis Date</u>
ICP-AES Metals	March 2, 2006	March 13 and 15, 2006
Mercury	March 9, 2006	March 13, 2006
Cyanide	March 2, 2006	March 9, 2006
Percent Solids	March 9, 2006	March 10, 2006

#### CLP PO Action

None.

#### Sampling Issues

None.

## Additional Comments

**As directed by the TOPO, a Tier 2 review was performed (forms review of CADRE R-flagged results only). For this sample delivery group (SDG), only copper results for samples MY2DQ1 through MY2DQ7 and MY2DQ9 through MY2DR3 “R” flagged by CADRE were reviewed. The results for analytes not listed above were not reviewed.**

The copper result (16.9 mg/kg) reported on Form 1A for sample MY2DQ3 has a “J” flag indicating the result is greater than the MDL but less than the CRQL. This “J” flag was not indicated on the original CADRE Table 1A.

A revised CADRE Table 1A is attached.

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: 1337.0, January 18, 2006;
- 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
	a. Initial		
	b. Initial and Continuing Calibration Verification		
	c. CRQL Check Standard (CRI)		
4.	Blanks	Yes	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	Yes	
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
12.	Sample Quantitation	Yes	
13.	Overall Assessment	Yes	

N/A = Not Applicable

### III. VALIDITY AND COMMENTS

- A. CADRE R-flagged copper results for samples MY2DQ1 through MY2DQ7 and MY2DQ9 through MY2DR3, analyzed on March 15, 2006, because the 188 percent recovery (%R) for copper in the last of five CRI analyses exceeded the 180 %R expanded recovery criterion specified in the National Functional Guidelines (NFG). Since the reported copper data for the samples listed above were analyzed between acceptable CRI recoveries, the R flags for the copper data in the CADRE Table 1A are not warranted.

The reviewed copper results are presented in bold in the revised CADRE Table 1A attached.

*The inorganic SOW specifies that the laboratory must analyze a CRI standard immediately following the initial calibration verification (ICV), at the beginning, end, and after every 20 analytical samples for each analytical run in order to verify linearity near the CRQL.*



**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DP4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

**Tier 2 Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/Kg (Dry Weight)**

**Analysis Type : LOW Concentration Soil**  
**Samples For Total Metals and Cyanide**

Station Location :	PCON-11-SED-0-022206			PCON-14-SED-0-022206			PCON-20-SED-0-022206			PCON-21-SED-0-022206			PCON-22-SED-0-022206			PCON-23-SED-0-022206			PCON-24-SED-0-022206		
Sample ID :	MY2DP4			MY2DP5			MY2DP6			MY2DP7			MY2DP8			MY2DP9			MY2DQ0		
Collection Date :	2/22/2006			2/22/2006			2/22/2006			2/22/2006			2/22/2006			2/22/2006			2/22/2006		
Dilution Factor :	1.0			1.0			1.0			1.0			1.0			1.0			1.0		
PARAMETER	Result	Val	Com																		
ALUMINUM	20600			21200			6990			22800			14300			4480			12900		
ANTIMONY	3.2	J		1.7	J		38.2	J		7.3	J		4.1	J		27.6	J		2.8	J	
ARSENIC	40.0			0.41U	U		1720			140			91.3			635			13.2		
BARIIUM	173			68.7J			138J			244			124J			105J			85.3J		
BERYLLIUM	0.48J	UJ		1.0			0.12J	UJ		0.69			0.10J	UJ		0.10J	UJ		0.38J	UJ	
CADMIUM	11.1			2.8			11.3			15.0			8.6			9.0			2.6		
CALCIUM	19500			32500			13100			12700			31400			6100			9810		
CHROMIUM	9.5			0.42U	U		22.9			14.0			7.9			48.0			14.5		
COBALT	62.4			51.9			37.8			61.9			38.5			44.7			41.0		
COPPER	53800			46300			14100			35000			18500			12600			10200		
IRON	68000			36700			118000			45400			43600			150000			52900		
LEAD	231			68.7			260			329			200			222			65.4		
MAGNESIUM	14300			12000			5770			17300			11600			4180			10100		
MANGANESE	555			605			553			789			446			411			596		
MERCURY	0.41			0.095			0.17			0.48			0.17			0.085			0.052		
NICKEL	63.6			65.8			41.9			82.7			38.8			33.4			33.7		
POTASSIUM	7900			4050			3280			8940			2720			2180			6280		
SELENIUM	22.8	J		8.5	J		11.0	J		17.1	J		10.8	J		9.0	J		7.5	J	
SILVER	14.2			2.9			22.8			18.8			12.3			17.7			3.0		
SODIUM	728			2280			239J	J		468J	J		340J	J		271J	J		303J	J	
THALLIUM	1.7	J		0.74U	U		0.89	J		1.2	J		0.72	J		1.4	J		1.0	J	
VANADIUM	93.3			62.8			40.4			105			62.8			28.0			60.5		
ZINC	793			199			913			832			575			889			613		
CYANIDE	0.32J	J-		0.21J	J-		0.51J	J-		0.38J	J-		2.5U	U		2.8	J-		2.7U	U	
MOLYBDENUM	1240			122			203			278			188			189			231		
BORON	4.1			3.6			2.4			5.8			3.3			1.6			2.5		

Percent Solids	97.9%			94.3%			99.7%			98.3%			98.9%			99.7%			93.5%		
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Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DP4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

**Tier 2 Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/Kg (Dry Weight)**

**Analysis Type : LOW Concentration Soil**  
**Samples For Total Metals and Cyanide**

Station Location :	PCON-25-SED-0-022206			UPA-04-SED-1-022206			UPA-0X-SED-1-022206			KAW-02-SED-0-022206			WSC-31-SED-0-022306			PSMT-01-SES-0-022306			PSMT-X-SED-0-0222306		
Sample ID :	MY2DQ1			MY2DQ2			MY2DQ3			MY2DQ4			MY2DQ5			MY2DQ6			MY2DQ7		
Collection Date :	2/22/2006			2/22/2006			2/22/2006			2/22/2006			2/23/2006			2/23/2006			2/23/2006		
Dilution Factor :	1.0			1.0			1.0			1.0			1.0			1.0			1.0		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	1620			7000			7390			2590			10100			1070			498		
ANTIMONY	10.6	J		0.39J	J		0.28J	J		0.84J	J		1.0	J		30.7	J		36.8	J	
ARSENIC	18.1			1.8			2.2			1.6			4.0			107			121		
BARIUM	23.7J			88.5J			88.4J			23.2J			91.2J			265			239		
BERYLLIUM	0.52U	U		0.30J	UJ		0.31J	UJ		0.070J	UJ		0.21J	UJ		0.050J	UJ		0.050J	UJ	
CADMIUM	8.0			0.080J	UJ		0.11J	UJ		0.30J	UJ		0.69			22.8			17.6		
CALCIUM	2310			16800			19200			2210			60800			1350			1270		
CHROMIUM	0.40U	U		5.9			6.1			3.8			12.7			0.40U	U		0.40U	U	
COBALT	115			5.7J			5.9J			7.5J			10.2			151			162		
<b>COPPER</b>	<b>59700</b>		A	<b>16.6J</b>		A	<b>16.9J</b>		A	<b>2900</b>		A	<b>454</b>		A	<b>369000</b>		A	<b>380000</b>		A
IRON	163000			11500			12000			11200			14900			104000			108000		
LEAD	348			5.1J			5.5J			9.6			23.2			1200			1230		
MAGNESIUM	3130			3960			4200			1900			8560			1620			911		
MANGANESE	232			258			252			61.1J			359			82.2J			64.7J		
MERCURY	0.020U	U		0.020U	U		0.020U	U		0.025	J		0.24			0.065			0.074		
NICKEL	43.9			7.2J			7.6J			7.0J			16.1J			231			232		
POTASSIUM	616			1210			1260			1600			1950			182J	J		147J	J	
SELENIUM	24.4	J		0.51U	UJ		0.51U	UJ		2.3	J		0.67	J		162	J		176	J	
SILVER	24.3			0.51U	U		0.51U	U		0.78	J		0.39J	J		119			118		
SODIUM	240J	J		139J	J		152J	J		101J	J		554			125J	J		90.8J	J	
THALLIUM	3.7			0.72U	U		0.71U	U		0.70U	U		0.71U	U		3.2			3.3		
VANADIUM	26.6			22.4			23.2			14.1			37.3			6.9			4.5	UJ	
ZINC	4430			30.3J			31.2J			34.7J			123			2400			2260		
CYANIDE	2.5U	U		2.6U	U		2.5U	U		2.5U	U		0.39J	J		2.5U	U		2.5U	U	
MOLYBDENUM	1020			0.25J			0.12J			55.0			2.2			284			300		
BORON	4.9			2.5			2.6			0.85			5.1			4.6			4.5		
Percent Solids	99.8%			97.8%			98.1%			99.6%			98.2%			99.1%			99.1%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

**REVIEWED RESULTS ARE PRESENTED IN BOLD.**

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DP4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

**Tier 2 Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/Kg (Dry Weight)**

**Analysis Type : LOW Concentration Soil**  
**Samples For Total Metals and Cyanide**

Station Location :	SDD-01-SED-0-022306	SDD-02-SED-0-022306	SDD03-SED-0-022306	SD-01-SED-0-022306	HDS-I-101-09-106-022306	HDS-A-101-09-106-022306	Lab Blank														
Sample ID :	MY2DQ8	MY2DQ9	MY2DR0	MY2DR1	MY2DR2	MY2DR3	PBS														
Collection Date :	2/23/2006	2/23/2006	2/23/2006	2/23/2006	2/23/2006	2/23/2006															
Dilution Factor :	1.0	1.0	1.0	1.0	1.0	1.0	1.0														
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	23100			17500			18300			1220			10100			12100			3.2J		
ANTIMONY	7.5	J		3.7	J		2.6	J		10.4	J		2.9	J		5.8	J		0.99U		
ARSENIC	361			232			74.0			24.7			30.6			147			0.39U		
BARIUM	217			176			87.4J			27.8J			190			251			161U		
BERYLLIUM	0.38J	UJ		0.35J	UJ		0.30J	UJ		0.52U	U		0.22J	UJ		0.46J	UJ		0.020J		
CADMIUM	94.7			82.4			18.8			9.6			6.4			18.0			-0.04000J		
CALCIUM	41300			62700			61300			1850			32100			26400			500U		
CHROMIUM	72.0			16.4			3.8			0.40U	U		4.8			8.3			0.18J		
COBALT	46.8			32.8			29.2			84.9			20.5			24.5			-0.05000J		
<b>COPPER</b>	12100			<b>10200</b>		<b>A</b>	<b>27000</b>		<b>A</b>	<b>47200</b>		<b>A</b>	<b>12400</b>		<b>A</b>	<b>21200</b>		<b>A</b>	0.45J		
IRON	64200			34400			53000			187000			28700			29400			5.1J		
LEAD	308			220			112			468			1700			705			0.24J		
MAGNESIUM	15300			14600			16000			3250			7880			7920			2.2J		
MANGANESE	557			647			581			232			428			396			0.040J		
MERCURY	0.49			0.44			0.13			0.020	J		0.38			1.9			0.020U		
NICKEL	40.9			35.5			24.8			32.1			71.6			31.2			18.2U		
POTASSIUM	2410			1850			1020			416J	J		4750			3670			500U		
SELENIUM	17.4	J		8.2	J		7.9	J		18.8	J		10.6	J		18.1	J		0.50U		
SILVER	11.0			9.8			7.6			24.5			6.6			15.9			0.50U		
SODIUM	441J	J		388J	J		294J	J		236J	J		5730			1420			55.2J		
THALLIUM	0.73U	U		0.44J	UJ		0.71J	UJ		2.4	UJ		0.71U	U		0.71U	U		0.70U		
VANADIUM	70.6			65.0			69.0			24.4			44.5			42.0			2.0U		
ZINC	5300			3340			1380			5920			3080			1350			0.38J		
CYANIDE	2.6U	U		2.6U	U		2.6U	U		2.5U	U		0.57J	J		0.80J	J		2.5U		
MOLYBDENUM	235			103			119			1060			135			331			0.11J		
BORON	4.8			3.9			2.6			7.3			31.1			9.8			0.50U		

Percent Solids	96.5%	97.6%	97.6%	100.0%	98.7%	98.5%	100.0%
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Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

**REVIEWED RESULTS ARE PRESENTED IN BOLD.**

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2DP4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

**QUALIFIED DATA**

**Concentration in mg/Kg (Dry Weight)**

**Analysis Type : LOW Concentration Soil  
 Samples For Total Metals**

Station Location :																					
Sample ID :		CRQL																			
Collection Date :																					
Dilution Factor :																					
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	20.0																				
ANTIMONY	6.0																				
ARSENIC	1.0																				
BARIUM	20.0																				
BERYLLIUM	0.50																				
CADMIUM	0.50																				
CALCIUM	500																				
CHROMIUM	1.0																				
COBALT	5.0																				
COPPER	2.5																				
IRON	10.0																				
LEAD	1.0																				
MAGNESIUM	500																				
MANGANESE	1.5																				
MERCURY	0.10																				
NICKEL	4.0																				
POTASSIUM	500																				
SELENIUM	3.5																				
SILVER	1.0																				
SODIUM	500																				
THALLIUM	2.5																				
VANADIUM	5.0																				
ZINC	6.0																				
CYANIDE	2.5																				
MOLYBDENUM	2.0																				
BORON	0.50																				

Percent Solids

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00905090

DATE: May 18, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None
Case No.:	35036
SDG No.:	MY2ES4
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Total Metals and Total Cyanide
Samples:	15 Soil Samples (see Case Summary)
Collection Date:	March 7 and 8, 2006
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: Cynthia Gurley, 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.: 35036  
SDG No.: MY2ES4  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: May 18, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2ES4 through MY2ES7, MY2EW2 through MY2EW9, MY2EX0, MY2EX1, and MY2EX2  
Concentration and Matrix: Low Concentration Soil  
Analysis: CLP Total Metals and Total Cyanide  
SOW: ILM05.3 and Modified Analysis Request 1337.0  
Collection Date: March 7 and 8, 2006  
Sample Receipt Date: March 10, 2006  
Preparation Date: March 20 and 21, 2006  
Analysis Date: March 21, 22, April 3, and 4, 2006

#### Field QC

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

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Soil (PBS) and samples listed above  
Matrix Spike: MY2EW9S  
Duplicates: MY2EW9D  
ICP Serial Dilution: MY2EW9L

Analysis: Total Metals and Total Cyanide

<u>Analyte</u>	<u>Sample Preparation and Digestion/Distillation Date</u>	<u>Analysis Date</u>
ICP-AES Metals	March 21, 2006	April 3 and 4, 2006
Mercury	March 21, 2006	March 22, 2006
Cyanide	March 20, 2006	March 21, 2006
Percent Solids	March 21, 2006	March 22, 2006

#### CLP PO Action

Modified Analysis Request 1337.0 specifies molybdenum to be spiked at 20 mg/kg in the matrix spike sample. The laboratory inadvertently spiked molybdenum at 2000 mg/kg.

#### Sampling Issues

None.

## Additional Comments

The samples in this SDG were analyzed for CLP total metals plus boron and molybdenum by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1337.0. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide was analyzed by the CLP spectrophotometric method.

The SDG Narrative states that Region 9 approved an increase in contract required quantitation limit (CRQL) for selenium from 0.30 mg/kg to 0.50 mg/kg.

The laboratory increased the mercury analysis sample size from 0.2 g to 0.5 g as permitted by MAR 1337.0.

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: 1337.0, January 18, 2006;
- 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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	No	B
9.	ICP Serial Dilution Analysis	No	C
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
12.	Sample Quantitation	Yes	A
13.	Overall Assessment	Yes	

N/A = Not Applicable

### III. VALIDITY AND COMMENTS

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

*Results above the method detection limit (MDL) but below the contract required quantitation limit (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 estimated and flagged "J" in Table 1A because matrix spike recovery results are outside method QC limits.

X Antimony, copper, and lead in all samples

Matrix spike recoveries for the samples listed above in QC sample MY2EW9S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for each analyte are presented below and are based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Antimony	26	-74
Copper	72	-28
Lead	135	+35

Results above the MDL are considered quantitatively uncertain. Results reported for antimony and copper in all samples may be biased low. Results reported for lead in all samples may be biased high.

According to the inorganic SOW, when the pre-digestion spike recovery results for ICP analytes (except silver) 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 results for sample MY2EW9A were obtained.

Analyte	Post-Digestion Spike, % Recovery
Antimony	88
Copper	99
Lead	103

Since the post-digestion spike recoveries were acceptable, the low pre-digestion spike recovery results obtained for antimony and copper and the high pre-digestion spike recovery result obtained for lead may indicate sample non-homogeneity, poor laboratory technique, or matrix effects which may interfere with accurate analysis, depressing antimony and copper results and enhancing lead results.

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

C. The following results are estimated and flagged "J" in Table 1A because ICP serial dilution results are outside method QC limits.

X Boron, chromium, cobalt, iron, lead, manganese, nickel, and sodium in all samples

The percent difference for the ICP serial dilution analysis of sample MY2EW9L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Boron	+17
Chromium	+11
Cobalt	+13
Iron	+11
Lead	+13
Manganese	+11
Nickel	+14
Sodium	+17

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The results for the diluted sample were higher than the original. Therefore, the reported sample results may be biased low.

*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.*

## 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.



**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2ES4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

**Analysis Type : Low Concentration Soil Samples**  
**For Total Metals and Cyanide**

Station Location :	GR-SED-01-030706			GR-SED-X-030706			GR-SED-02-030706			GR-SED-03-030706			GR-SED-04-00806			SPR-SED-01-030806			SPR-SED-02-030806		
Sample ID :	MY2ES4			MY2ES5			MY2ES6			MY2ES7			MY2EW2			MY2EW3			MY2EW4		
Collection Date :	3/7/2006			3/7/2006			3/7/2006			3/7/2006			3/8/2006			3/8/2006			3/8/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	17500			18500			17800			19400			6750			4250			4540		
ANTIMONY	1.1	J	B	1.1	J	B	1.2L	J	AB	1.1L	J	AB	0.48L	J	AB	0.56L	J	AB	0.45L	J	AB
ARSENIC	2.1			1.4			4.4			5.0			1.1			2.6			2.0		
BARIUM	59.6L	J	A	59.4L	J	A	151L	J	A	177L	J	A	51.8L	J	A	69.6L	J	A	61.4L	J	A
BERYLLIUM	0.54U			0.55U			0.29L	J	A	0.46L	J	A	0.090L	J	A	0.22L	J	A	0.15L	J	A
CADMIUM	0.42U			0.42U			0.82U			0.060L	J	A	0.52U			0.12L	J	A	0.47U		
CALCIUM	32100			30300			39600			41900			8940			8080			7800		
CHROMIUM	14.4	J	C	14.0	J	C	14.0	J	C	15.5	J	C	6.0	J	C	2.9	J	C	4.3	J	C
COBALT	16.2	J	C	16.6	J	C	14.5L	J	AC	14.8L	J	AC	5.8L	J	AC	3.2L	J	AC	4.9L	J	AC
COPPER	45.0	J	B	62.2	J	B	191	J	B	392	J	B	29.3	J	B	143	J	B	16.8L	J	AB
IRON	28500	J	C	28800	J	C	22500	J	C	22500	J	C	10700	J	C	5480	J	C	8710	J	C
LEAD	9.8	J	BC	9.5	J	BC	11.7L	J	ABC	15.5L	J	ABC	3.4L	J	ABC	10.5	J	BC	6.7L	J	ABC
MAGNESIUM	9960			10900			9260			9910			3120			2110			2890		
MANGANESE	515	J	C	585	J	C	2270	J	C	2410	J	C	201	J	C	183	J	C	173	J	C
MERCURY	0.021U			0.021U			0.041U			0.049U			0.026U			0.023U			0.024U		
NICKEL	14.0L	J	AC	13.1L	J	AC	18.3L	J	AC	21.8L	J	AC	7.8L	J	AC	5.2L	J	AC	7.3L	J	AC
POTASSIUM	781			747			2520			2570			585L	J	A	876			715		
SELENIUM	0.52U			0.53U			1.3			1.3			0.65U			0.58U			0.59U		
SILVER	0.15L	J	A	0.53U			1.0U			1.2U			0.65U			0.58U			0.59U		
SODIUM	966	J	C	1100	J	C	2030	J	C	1230	J	C	1050	J	C	364L	J	AC	387L	J	AC
THALLIUM	0.73U			0.54L	J	A	1.4U			1.7U			0.91U			0.81U			0.83U		
VANADIUM	91.2			91.2			54.6			49.8			29.2			8.7			14.2		
ZINC	48.9			53.0			55.5L	J	A	63.9L	J	A	22.0L	J	A	20.0L	J	A	18.1L	J	A
CYANIDE	2.6U			2.7U			5.1U			6.1U			3.2U			2.9U			3.0U		
MOLYBDENUM	2.1U			2.1U			1.4L	J	A	2.2L	J	A	0.20L	J	A	1.6L	J	A	1.9L	J	A
BORON	2.4	J	C	2.5	J	C	10.2	J	C	10.9	J	C	1.7	J	C	1.4	J	C	1.9	J	C
PERCENT SOLIDS	96.1%			94.2%			48.9%			40.8%			77.2%			86.4%			84.6%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2ES4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

**Analysis Type : Low Concentration Soil Samples**  
**For Total Metals and Cyanide**

Station Location :	GR-SED-X-030806			GR-SED-06-030806			GR-SED-05-0308-6			GR-SED-07-030806			GR-SED-08-030806			GR-SED-09-030806			GR-SED-10-030806		
Sample ID :	MY2EW5			MY2EW6			MY2EW7			MY2EW8			MY2EW9			MY2EX0			MY2EX1		
Collection Date :	3/8/2006			3/8/2006			3/8/2006			3/8/2006			3/8/2006			3/8/2006			3/8/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	14500			13200			12800			20300			18900			13000			16300		
ANTIMONY	0.98L	J	AB	0.93L	J	AB	0.77L	J	AB	0.97L	J	AB	1.2L	J	AB	0.64L	J	AB	1.0L	J	AB
ARSENIC	2.9			2.7			2.7			4.6			4.2			2.8			4.5		
BARIUM	107L	J	A	97.9L	J	A	99.1L	J	A	154L	J	A	145L	J	A	113L	J	A	140L	J	A
BERYLLIUM	0.35L	J	A	0.39L	J	A	0.34L	J	A	0.54L	J	A	0.61L	J	A	0.40L	J	A	0.49L	J	A
CADMIUM	0.040L	J	A	0.69U			0.52U			0.040L	J	A	0.66U			1.1U			0.030L	J	A
CALCIUM	21700			21800			19900			36500			33900			27000			32300		
CHROMIUM	12.1	J	C	12.2	J	C	11.2	J	C	17.2	J	C	21.0	J	C	11.1	J	C	14.2	J	C
COBALT	10.0L	J	AC	9.3L	J	AC	8.8L	J	AC	14.6L	J	AC	13.6L	J	AC	9.2L	J	AC	12.1L	J	AC
COPPER	129	J	B	88.2	J	B	96.0	J	B	158	J	B	177	J	B	109	J	B	147	J	B
IRON	18200	J	C	17300	J	C	16000	J	C	24700	J	C	26300	J	C	15100	J	C	19600	J	C
LEAD	12.5	J	BC	11.7L	J	ABC	17.6	J	BC	16.4	J	BC	18.5	J	BC	11.6L	J	ABC	15.0	J	BC
MAGNESIUM	6520			5960			5720			9850			9070			6190			8050		
MANGANESE	473	J	C	437	J	C	458	J	C	1080	J	C	1010	J	C	1410	J	C	1470	J	C
MERCURY	0.027U			0.034U			0.026U			0.039L	J	A	0.033U			0.056U			0.034L	J	A
NICKEL	13.7L	J	AC	12.6L	J	AC	12.5L	J	AC	20.6L	J	AC	19.3L	J	AC	14.3L	J	AC	17.8L	J	AC
POTASSIUM	1850			2230			1740			3220			3320			2210			2800		
SELENIUM	0.68U			0.86U			0.66U			1.0U			0.82U			1.4U			0.93L	J	A
SILVER	0.68U			0.86U			0.66U			1.0U			0.82U			1.4U			0.97U		
SODIUM	729	J	C	664L	J	AC	681	J	C	2790	J	C	1550	J	C	867L	J	AC	1160	J	C
THALLIUM	0.95U			1.2U			0.57L	J	A	1.4U			1.1U			1.9U			1.4U		
VANADIUM	43.4			39.1			38.3			53.4			60.7			30.9			41.2		
ZINC	46.7L	J	A	47.8L	J	A	41.0L	J	A	70.7L	J	A	74.9			44.6L	J	A	63.5L	J	A
CYANIDE	3.4U			4.3U			3.3U			5.1U			4.1U			6.9U			4.8U		
MOLYBDENUM	0.60L	J	A	1.8L	J	A	0.46L	J	A	2.2L	J	A	2.4L	J	A	1.5L	J	A	1.5L	J	A
BORON	4.9	J	C	5.6	J	C	4.6	J	C	9.9	J	C	8.8	J	C	7.1	J	C	8.6	J	C
PERCENT SOLIDS	73.8%			58.2%			76.3%			49.4%			60.9%			36.0%			51.7%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35036                      SDG No. : MY2ES4  
 Site : ASARCO  
 Lab : LIBERTY ANALYTICAL CORPORATION  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : May 18, 2006

Table 1A

**QUALIFIED DATA**  
 Concentration in mg/kg (Dry Weight)

Analysis Type : Low Concentration Soil Samples  
 For Total Metals and Cyanide

Station Location : GR-SED-11-030806			MDL			CRQL															
Sample ID : MY2EX2																					
Collection Date : 3/8/2006																					
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	20000			3.1			50.0														
ANTIMONY	0.99L	J	AB	0.18			0.99														
ARSENIC	5.5			0.32			0.39														
BARIUM	176L	J	A	0.047			161.3														
BERYLLIUM	0.75L	J	A	0.022			0.52														
CADMIUM	0.11L	J	A	0.011			0.40														
CALCIUM	36900			7.1			500														
CHROMIUM	17.9	J	C	0.13			0.40														
COBALT	13.6L	J	AC	0.033			9.7														
COPPER	144	J	B	0.083			16.6														
IRON	23800	J	C	2.3			200														
LEAD	21.1	J	BC	0.11			7.7														
MAGNESIUM	9300			0.88			500														
MANGANESE	1170	J	C	0.035			100														
MERCURY	0.030L	J	A	0.042			0.050														
NICKEL	20.1L	J	A	0.092			18.2														
POTASSIUM	3760			2.1			500														
SELENIUM	0.82U			0.35			0.50														
SILVER	0.82U			0.067			0.50														
SODIUM	1390	J	C	13.3			500														
THALLIUM	1.2			0.40			0.70														
VANADIUM	50.0			0.023			2.0														
ZINC	74.1			0.18			38.9														
CYANIDE	4.1U			0.32			10.0														
MOLYBDENUM	1.4L	J	A	0.068			2.0														
BORON	10.8	J	C	0.031			0.50														
PERCENT SOLIDS	61.3%			N/A			N/A														

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

THROUGH: Rose Fong, ESAT Task Order Project Officer (TOPO)  
Quality Assurance (QA) Program, MTS-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.: 00905092

DATE: June 1, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None
Case No.:	35130
SDG No.:	MY2FG2
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Total Metals by ICP-MS and Total Mercury
Samples:	15 Water Samples (see Case Summary)
Collection Date:	March 7 and 8, 2006
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: Cynthia Gurley, 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.: 35130  
SDG No.: MY2FG2  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: June 1, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2FG2 through MY2FG9 and MY2FH0 through MY2FH6  
Concentration and Matrix: Low Concentration Water  
Analysis: CLP Total Metals by ICP-MS and Total Mercury  
SOW: ILM05.3 and Modified Analysis Request 1340.0  
Collection Date: March 7 and 8, 2006  
Sample Receipt Date: March 16, 2006  
Preparation Date: April 3 and 5, 2006  
Analysis Date: April 3, 4, and 5, 2006

#### Field QC

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

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Water (PBW) and samples listed above

Matrix Spike: MY2FG9S  
Duplicates: MY2FG9D  
ICP Serial Dilution: MY2FG9L

Analysis: CLP Total Metals by ICP-MS and Total Mercury

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	April 3 and 5, 2006	April 4 and 5, 2006
Mercury	April 3, 2006	April 3, 2006
Percent Solids	Not Applicable	Not Applicable

#### CLP PO Action

None.

#### Sampling Issues

1. The sampler provided the station location instead of the CLP inorganic sample number on both Traffic Report/Chain of Custody (TR/COC) record forms for this SDG. The laboratory contacted the Sample Management Office (SMO) and was provided with CLP sample numbers for this SDG. No adverse effect on data quality is expected.

2. There is no sampler's signature provided in the Sampler Signature field or sample relinquish information on the TR/COC record form for samples MY2FH2 through MY2FH6. No adverse effect on data quality is expected.
3. The cooler containing samples for this SDG arrived at the laboratory at a temperature of 6.7°C. This temperature exceeds the 4°± 2°C specified in the Statement of Work (SOW); however, no adverse effect on data quality is expected.

### Additional Comments

A Form 9 requires minor editing to correct several Contract Required Quantitation Limit (CRQL) values. A corrected Form 9 was requested from the laboratory but has not been received to date. Data quality is not likely to be affected and this report is considered final.

The samples in this SDG were analyzed for CLP total metals by ICP-MS plus aluminum, boron, iron, and molybdenum by ICP-MS under Modified Analysis Request (MAR), Modification Reference Number 1340.0. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide analysis is specified in MAR 1340.0; however, the laboratory indicates cyanide samples were not provided for this SDG.

The results for aluminum and iron for samples MY2FG2 through MY2FH6 in this SDG were determined by inductively coupled plasma-mass spectroscopy (ICP-MS). Note that aluminum and iron results are also reported for samples MY2FG2 through MY2FH6 in SDG MY2FG3, determined by inductively coupled plasma- atomic emission spectroscopy (ICP-AES).

The laboratory indicates the original sample preparation batch, prepared April 3, 2006, was contaminated with copper. The samples were prepared again on April 5, 2006 and analyzed for copper on April 5 and 6, 2006. No adverse effect on data quality is expected.

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: 1340.0, February 23, 2006;
- 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	B
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	No	C
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
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 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.*

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

- X Antimony in all samples
- X Iron in sample MY2FH6

Antimony (0.17 µg/L) and iron (10.2 µg/L) results in preparation blank PBW are greater than their respective MDLs but less than their respective CRQLs. In addition, antimony was found in the initial calibration blank (ICB) at 1.4 µg/L and in the continuing calibration blanks (CCBs) ranging between 0.64 µg/L and 0.75 µg/L which 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 their 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.*

*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.*

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

X Vanadium in all samples

The percent difference for the ICP serial dilution analysis of sample MY2FG9L did not meet the 10% criterion for vanadium shown below.

Analyte	% Difference
Vanadium	+15

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The results for the diluted sample were higher than the original. Therefore, the reported sample results for vanadium may be biased low.

*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.*

## 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.



In Reference to  
Case: 35130 SDG No.: MY2FG2

Contract Laboratory Program  
REGIONAL/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call: May 31, 2006

Laboratory Name: CompuChem (LIBRTY)

Lab Contact: Alice Evens or Bob Meierer

Region: 9

Regional Contact: Steve Remaley, CLP PO

ESAT Reviewer: Stan Kott, ESAT/LDC

Call Initiated By:      Laboratory   X   Region

In reference to data for the following samples: SDG No.: MY2FG2 all samples

Summary of Questions/issues Discussed:

The following items were noted during the review of this sample delivery group (SDG) data package. Please respond within 4 days as specified in ILM05.3 Statement of Work (SOW), Exhibit B, Section 2, 2.2. Send response and resubmissions to

ICF International/Laboratory Data Consultants, Inc.,  
Environmental Services Assistance Team, USEPA Region 9 Laboratory  
1337 S. 46th Street, Building 201, Richmond, CA 94804, FAX 510 412-2304.

1. The SDG Narrative indicates this SDG was analyzed according to Modification Reference Number (MRN): 1340.0; however, a copy of this document was not provided with the SDG Narrative. Please provide a copy of MRN: 1340.0.
2. Form 9, Method Detection Limits (page 49), does not reflect the CRQL concentration specified for beryllium, boron, cadmium, and silver in MRN: 1340.0. Please review the information and provide a corrected Form 9.

Summary of Resolution: To be determined.

\_\_\_\_\_  
Regional Contact Signature

\_\_\_\_\_  
Date of Resolution



**ANALYTICAL RESULTS**

Case No. : 35130 SDG No. : MY2FG2  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : June 1, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type :** Low Concentration Water Samples For CLP  
 Total Metals By ICP-MS and Total Mercury

Station Location :	GR-SW-01-030706			GR-SW-02-030706			GR-SW-03-030706			GR-SW-04-030806			GR-SW-05-030806			GR-SW-06-030806			GR-SW-07-030806		
Sample ID :	MY2FG2			MY2FG3			MY2FG4			MY2FG5			MY2FG6			MY2FG7			MY2FG8		
Collection Date :	3/7/2006			3/7/2006			3/7/2006			3/8/2006			3/8/2006			3/8/2006			3/8/2006		
PARAMETER	Result	Val	Com																		
ALUMINUM	738			904			816			944			1030			821			754		
ANTIMONY	2.0U		B																		
ARSENIC	3.0			3.0			2.9			2.8			2.8			2.9			2.7		
BARIUM	63.6			63.0			61.5			64.5			62.5			62.0			64.5		
BERYLLIUM	0.66U																				
CADMIUM	0.25U																				
CHROMIUM	0.52L	J	A	0.61L	J	A	0.60L	J	A	0.66L	J	A	0.67L	J	A	0.59L	J	A	0.55L	J	A
COBALT	0.50L	J	A	0.46L	J	A	0.48L	J	A	0.44L	J	A	0.45L	J	A	0.43L	J	A	0.45L	J	A
COPPER	4.9			5.4			6.0			4.5			5.6			6.5			6.8		
IRON	505			581			527			580			628			545			507		
LEAD	0.70L	J	A	0.68L	J	A	0.72L	J	A	0.66L	J	A	0.68L	J	A	0.72L	J	A	1.0		
MANGANESE	105			97.9			97.4			89.8			81.3			80.5			94.1		
MERCURY	0.20U																				
NICKEL	2.1			2.1			2.0			2.1			2.0			1.9			1.9		
SELENIUM	0.54L	J	A	0.45L	J	A	0.38L	J	A	0.54L	J	A	0.49L	J	A	0.38L	J	A	0.40L	J	A
SILVER	0.36U			0.36U			0.081L	J	A	0.36U			0.36U			0.057L	J	A	0.36U		
THALLIUM	1.0U																				
VANADIUM	6.2	J	C	6.2	J	C	6.1	J	C	5.9	J	C	5.6	J	C	5.7	J	C	5.9	J	C
ZINC	2.2			2.4			2.5			4.5			3.2			2.8			3.0		
MOLYBDENUM	4.3L	J	A	4.4L	J	A	4.4L	J	A	4.6L	J	A	4.3L	J	A	4.4L	J	A	4.6L	J	A
BORON	129			128			125			131			124			124			130		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35130 SDG No. : MY2FG2  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : June 1, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type :** Low Concentration Water Samples For CLP  
 Total Metals By ICP-MS and Total Mercury

Station Location :	GR-SW-08-030806			GR-SW-09-030806			GR-SW-10-030806			GR-SW-11-030806			GR-SW-X-030706			GR-SW-X-030806			SPR-SW-01-030806		
Sample ID :	MY2FG9			MY2FH0			MY2FH1			MY2FH2			MY2FH3			MY2FH4			MY2FH5		
Collection Date :	3/8/2006			3/8/2006			3/8/2006			3/8/2006			3/7/2006			3/8/2006			3/8/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com									
ALUMINUM	1090			1040			981			1090			869			1020			765		
ANTIMONY	2.0U		B	2.0U		B	2.0U		B	2.0U		B									
ARSENIC	3.0			3.2			2.8			3.1			2.8			2.9			5.2		
BARIUM	65.7			68.7			63.0			68.1			63.0			65.9			81.2		
BERYLLIUM	0.085L	J	A	0.66U			0.66U			0.066L	J	A	0.66U			0.66U			0.66U		
CADMIUM	0.25U			0.25U			0.25U			0.25U			0.25U			0.25U			0.12L	J	A
CHROMIUM	0.80L	J	A	0.75L	J	A	0.68L	J	A	0.81L	J	A	0.57L	J	A	0.71L	J	A	0.72L	J	A
COBALT	0.53L	J	A	0.56L	J	A	0.49L	J	A	0.63L	J	A	0.49L	J	A	0.48L	J	A	0.58L	J	A
COPPER	5.4			6.2			7.1			7.6			4.1			5.3			70.2		
IRON	718			687			640			738			597			664			552		
LEAD	0.87L	J	A	0.87L	J	A	0.86L	J	A	1.0			0.70L	J	A	0.72L	J	A	4.3		
MANGANESE	139			145			134			146			104			86.4			31.6		
MERCURY	0.20U			0.20U			0.20U			0.20U			0.20U			0.20U			0.20U		
NICKEL	2.3			2.1			2.0			2.3			2.0			2.0			1.3		
SELENIUM	0.90L	J	A	0.97L	J	A	0.49L	J	A	0.43L	J	A	0.49L	J	A	0.42L	J	A	0.37L	J	A
SILVER	0.36U			0.36U			0.36U			0.041L	J	A	0.36U			0.36U			0.050L	J	A
THALLIUM	1.0U			1.0U			1.0U			1.0U			1.0U			1.0U			1.0U		
VANADIUM	6.0	J	C	6.4	J	C	5.9	J	C	6.4	J	C	6.2	J	C	6.1	J	C	4.9	J	C
ZINC	3.6			4.2			3.3			3.7			2.5			3.1			7.1		
MOLYBDENUM	5.3L	J	A	5.3L	J	A	4.9L	J	A	5.4L	J	A	4.3L	J	A	4.6L	J	A	17.8L	J	A
BORON	130			139			125			136			126			133			188		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35130                      SDG No. : MY2FG2  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : June 1, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type :** Low Concentration Water Samples For CLP  
 Total Metals By ICP-MS and Total Mercury

Station Location : SPR-SW-02-030806				MDL			CRQL														
Sample ID : MY2FH6																					
Collection Date : 3/8/2006																					
PARAMETER	Result	Val	Com																		
ALUMINUM	64.5L	J	A	8.7			87.0														
ANTIMONY	2.0U		B	0.046			2.0														
ARSENIC	5.5			0.049			1.0														
BARIIUM	68.1			0.92			4.0														
BERYLLIUM	0.66U			0.065			0.66														
CADMIUM	0.25U			0.059			0.25														
CHROMIUM	0.24L	J	A	0.17			2.0														
COBALT	0.20L	J	A	0.14			1.0														
COPPER	8.8			0.28			2.0														
IRON	300U		B	7.3			300														
LEAD	0.14L	J	A	0.055			1.0														
MANGANESE	92.0			0.032			1.0														
MERCURY	0.20U			0.032			0.20														
NICKEL	0.55L	J	A	0.29			1.0														
SELENIUM	0.29L	J	A	0.15			5.0														
SILVER	0.36U			0.022			0.36														
THALLIUM	1.0U			0.073			1.0														
VANADIUM	4.4	J	C	0.040			1.0														
ZINC	2.4			0.30			2.0														
MOLYBDENUM	20.1L	J	A	0.085			182														
BORON	225			0.19			1.6														

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

THROUGH: Rose Fong, ESAT Task Order Project Officer (TOPO)  
Quality Assurance (QA) Program, MTS-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.: 00905090

DATE: June 1, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None
Case No.:	35130
SDG No.:	MY2FG3
Laboratory:	CompuChem (LIBRTY)
Analysis:	Select CLP Total Metals By ICP-AES
Samples:	15 Water Samples (see Case Summary)
Collection Date:	March 7 and 8, 2006
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: Cynthia Gurley, 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.: 35130  
SDG No.: MY2FG3  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: May 31, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2FG2 through MY2FG9 and MY2FH0 through MY2FH6  
Concentration and Matrix: Low Concentration Water  
Analysis: Select CLP Total Metals By ICP-AES  
SOW: ILM05.3  
Collection Date: March 7 and 8, 2006  
Sample Receipt Date: March 16, 2006  
Preparation Date: March 22, 2006  
Analysis Date: April 3, and 4, 2006

#### Field QC

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

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Water (PBW) and samples listed above  
Matrix Spike: MY2FG9S  
Duplicates: MY2FG9D  
ICP Serial Dilution: MY2FG9L

Analysis: Select CLP Total Metals By ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	March 22, 2006	April 3 and 4, 2006
Percent Solids	Not Applicable	Not Applicable

#### CLP PO Action

None.

#### Sampling Issues

1. The sampler provided the station location instead of the CLP inorganic sample number on both Traffic Report/Chain of Custody (TR/COC) record forms for this SDG. The laboratory contacted the Sample Management Office (SMO) and was provided with CLP sample numbers for this SDG. No adverse effect on data quality is expected.

2. There is no sampler's signature provided in the Sampler Signature field or sample relinquish information on the TR/COC record form for samples MY2FH2 through MY2FH6. No adverse effect on data quality is expected.
3. The cooler containing samples for this SDG arrived at the laboratory at a temperature of 6.7°C. This temperature exceeds the 4°± 2°C specified in the Statement of Work (SOW); however, no adverse effect on data quality is expected.

### Additional Comments

The results for aluminum and iron for samples MY2FG2 through MY2FH6 in this SDG were determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). Note that the aluminum and iron results are also reported for samples MY2FG2 through MY2FH6 in SDG MY2FG2, determined by inductively coupled plasma-mass spectroscopy (ICP-MS).

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 *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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	No	B
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
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 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.*

- B. 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 MY2EW9L did not meet the 10% criterion for potassium shown below.

Analyte	% Difference
Potassium	-14

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The results for the diluted sample were lower than the original. Therefore, the reported sample results for potassium 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.*



## 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.



**ANALYTICAL RESULTS**

Case No. : 35130 SDG No. : MY2FG3  
 Site : ASARCO  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : June 1, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type : LOW Concentration Water Samples**  
**For Select Total Metals By ICP-AES**

Station Location :	GR-SW-01-030706	GR-SW-02-030706	GR-SW-03-030706	GR-SW-04-030806	GR-SW-05-030806	GR-SW-06-030806	GR-SW-07-030806														
Sample ID :	MY2FG2	MY2FG3	MY2FG4	MY2FG5	MY2FG6	MY2FG7	MY2FG8														
Collection Date :	3/7/2006	3/7/2006	3/7/2006	3/8/2006	3/8/2006	3/8/2006	3/8/2006														
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com			
ALUMINUM	369			985			481			597			456			451			1080		
CALCIUM	58800			58400			58200			58400			58800			56500			59200		
IRON	306			702			377			434			340			345			771		
MAGNESIUM	17100			17100			16900			17000			17100			16400			17200		
POTASSIUM	4840L	J	AB	4960L	J	AB	4790L	J	AB	4890L	J	AB	4850L	J	AB	4670L	J	AB	4980L	J	AB
SODIUM	127000			126000			126000			127000			127000			122000			127000		

Station Location :	GR-SW-08-030806	GR-SW-09-030806	GR-SW-10-030806	GR-SW-11-030806	GR-SW-X-030706	GR-SW-X-030806	SPR-SW-01-030806														
Sample ID :	MY2FG9	MY2FH0	MY2FH1	MY2FH2	MY2FH3	MY2FH4	MY2FH5														
Collection Date :	3/8/2006	3/8/2006	3/8/2006	3/8/2006	3/7/2006	3/8/2006	3/8/2006														
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com			
ALUMINUM	966			258			1190			1180			1140			668			307		
CALCIUM	63400			66400			68500			68700			60500			59400			135000		
IRON	682			232			861			840			817			484			273		
MAGNESIUM	18300			18900			19700			19700			17700			17300			30200		
POTASSIUM	4980L	J	AB	4960L	J	AB	5280	J	B	5270	J	B	5160	J	B	4910L	J	AB	5140	J	B
SODIUM	127000			132000			135000			134000			130000			127000			152000		

Station Location :	SPR-SW-02-030806	MDL	CRQL															
Sample ID :	MY2FH6																	
Collection Date :	3/8/2006																	
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	200U			30.7			200											
CALCIUM	123000			195			5000											
IRON	43.5L	J	A	20.5			100											
MAGNESIUM	27400			41.7			5000											
POTASSIUM	6570	J	B	11.3			5000											
SODIUM	175000			121			5000											

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit, N/A - Not Applicable, NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank, TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00105020

DATE: October 11, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not Provided
Case No.:	35241
SDG No.:	MY2GE5
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Total Metals plus Boron and Molybdenum
Samples:	20 Dust Samples (see Case Summary)
Collection Date:	May 17 and 18, 2006
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: Cynthia Gurley, 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.: 35241  
SDG No.: MY2GE5  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: October 11, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2GE5 through MY2GE9, MY2GF0 through MY2GF9, and MY2GG0 through MY2GG4  
Concentration and Matrix: Low and Medium Concentration Dust  
Analysis: CLP Total Metals plus Boron and Molybdenum  
SOW: ILM05.3 and Modification Reference Number 1337.1  
Collection Date: May 17 and 18, 2006  
Sample Receipt Date: May 20, 2006  
Preparation Date: June 3, 2006  
Analysis Date: June 4, 5, 6, 7, 8, and 9, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY2GE8 and MY2GE9  
(D2): MY2GF7 and MY2GF8

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Solid (PBS) and samples listed above  
Matrix Spike: MY2GF4S  
Duplicates: MY2GF4D  
ICP Serial Dilution: MY2GF4L

Analysis: CLP Total Metals plus Boron and Molybdenum

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	June 3, 2006	June 4, 5, 7, 8, and 9, 2006
Mercury	June 3, 2006	June 5 and 6, 2006
Percent Solids	Not Analyzed	Not Analyzed

#### CLP PO Action

None.

#### Sampling Issues

No collection time was provided for sample MY2GF8 on the Traffic Report/Chain of Custody (TR/COC).

## Additional Comments

Form 1 edits are required from the laboratory. These edits were requested from the laboratory but have not been received to date. Data quality is not likely to be affected and this report is considered final. Refer to the attached communication record log (CRL) for details.

The samples in this SDG were analyzed for CLP total metals plus boron and molybdenum by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1337.1. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide analysis is specified in MAR 1337.1; however, the laboratory indicates Region 9 did not require cyanide analysis for this SDG.

The laboratory states in the SDG Narrative that there was insufficient sample to perform the percent solids analysis. Region 9 instructed the laboratory to assume 100% solids on Form 1s. Since the percent solids could not be verified, the results provided in Table 1A are on an as received basis. The effect on data quality is not known.

The laboratory indicates the volume of sample MY2GF1 was insufficient to perform both ICP metals and mercury analyses. The laboratory used 0.15 grams of sample for ICP analysis instead of 1.0 grams specified in the SOW. The ICP metals CRQLs for sample MY2GF1 were adjusted accordingly. Mercury analysis was not performed on sample MY2GF1.

All samples of this SDG, except MY2GF1, required 2, 5, 10, 20, or 25-fold dilution to bring analyte concentrations within the instrument's linear range. No adverse effect on data quality is expected.

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: 1337.1, May 3, 2006;
- 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	B
5.	ICP Interference Check Sample (ICS)	No	C
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	No	D
9.	ICP Serial Dilution Analysis	No	E
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	No	F
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 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.*

- B. The following results are reported as non-detected (U) in Table 1A due to low level continuing calibration blank (CCB) contamination.

X Beryllium in samples MY2GG0, MY2GG1, and MY2GG2

The beryllium (0.044 mg/kg) concentration in CCB9 is greater than the MDL but less than the CRQL. Sample results associated with CCB9 that are greater than or equal to the MDL but less than the CRQL are reported as non-detected (U) at the CRQL.

*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.*

C. The following results are estimated and are flagged "J" in Table 1A due to possible ICP interelement interference problems.

- X Cadmium in samples MY2GE5 through MY2GF0, MY2GF2 through MY2GF8, MY2GG0, MY2GG1, MY2GG3, and MY2GG4
- X Selenium in all samples except MY2GF2
- X Thallium in all samples except MY2GF1 and MY2GF9

Results for cadmium, selenium, and thallium in the samples listed above were reported from an undiluted analysis that contained iron concentrations greater than the true value specified for the ICP interference check sample (ICS). Therefore, the applied interelement correction (IEC) factor may not compensate sufficiently for the interference. The cadmium results for the samples listed above may be biased high. The selenium and thallium results for the samples listed above biased low and, where non-detected, false negatives may exist.

*The ICP ICS solutions A and AB are analyzed to determine the effects of high concentrations of interfering elements on each analyte determined by ICP. Solution A consists of the interferents (Al, Ca, Fe, and Mg), and Solution AB consists of the analytes mixed with the interferents.*

*When the estimated concentration produced by the interfering element is greater than twice the CRQL and also is greater than 10% of the reported concentration of the affected element, the results of the affected elements are estimated.*

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

- X Antimony, manganese, and selenium in all samples

Matrix spike recoveries for antimony, manganese, and selenium in QC sample MY2GF4S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for each analyte are presented below and are based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Antimony	25	-75
Manganese	65	-35
Selenium	69	-31

Results above the MDL are considered quantitatively uncertain. Results reported for antimony, manganese, and selenium in all samples may be biased low.

According to the inorganic SOW, when the pre-digestion spike recovery results for ICP analytes (except silver) 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 results for QC sample MY2GF4A were obtained.

Analyte	Post-Digestion Spike, % Recovery
Antimony	83
Manganese	81
Selenium	87

Since the post-digestion spike recoveries were acceptable, the low pre-digestion spike recovery results obtained for antimony, manganese, and selenium may indicate sample non-homogeneity, 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. The following results are estimated and flagged "J" in Table 1A because ICP serial dilution results are outside method QC limits.

X Boron, calcium, lead, magnesium, and zinc in all samples

The percent difference for the ICP serial dilution analysis of sample MY2GF4L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Boron	+28
Calcium	+11
Lead	+12
Magnesium	+11
Zinc	+26

Results reported for the analytes listed above 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 higher than the original. Therefore, the reported sample results for the analytes listed above may be biased low.

*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.*

- F. The following relative percent differences (RPDs) were obtained for field duplicate pair D1: MY2GE8 and MY2GE9 and are listed below.

Analyte	RPD
Boron	42
Copper	36
Iron	63
Selenium	39
Silver	39
Zinc	62

Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\sqrt{35}$  RPD criterion for precision. The effect on the quality of the data 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, sample non-homogeneity, 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.



In Reference to  
Case: 35241 SDG No.: MY2GE5

Contract Laboratory Program  
REGIONAL/LABORATORY COMMUNICATION SYSTEM

Communication Record Log

Date of Call: October 11, 2006

Laboratory Name: CompuChem (LIBRTY)

Lab Contact: Alice Evens or Bob Meierer

Region 9 Contact: Steve Remaley, CLP PO

ESAT Reviewer: Stan Kott, ESAT/LDC

Call Initiated By:      Laboratory   X   Region

In reference to data for the following sample(s): All

Summary of Questions/issues Discussed:

The following items were noted during the review of this sample delivery group (SDG) data package. Please respond within 4 days as specified in ILM05.3 Statement of Work (SOW), Exhibit B, Section 2, 2.2. Send response and resubmissions to

ICF International/Laboratory Data Consultants, Inc.,  
Environmental Services Assistance Team, USEPA Region 9 Laboratory  
1337 S. 46th Street, Building 201, Richmond, CA 94804, FAX 510 412-2304.

1. All Form 1s flag thallium results with "\*" indicating duplicate analysis results were not within control limits. Form 6 (Duplicates) does not flag thallium results. Please review data and provide corrected forms.
2. Amended Form 1s for samples MY2GE5 through MY2GF8, provided in the data package, flag selenium with an "E" indicating a serial dilution result outside control limits. Form 8 (Serial Dilutions) does not flag selenium results. Please review data and provided corrected forms.
3. Modification Reference Number (MRN) 1337.1 specifies that non-detected results are to be reported at the CRQLs specified in MRN 1337.1. Please review the data and provide corrected Form 1s for samples MY2GE5 through MY2GF8 that reflect the thallium CRQL specified in MRN 1337.1.

4. The results for the following analytes are greater than the MDL but less than the CRQL and require a "J" flag:
  - Beryllium in samples MY2GE5 through MY2GE9 and MY2GF0 through MY2GF8,
  - Barium in samples MY2GF0, MY2GF1, and MY2GF2,
  - Cobalt in samples MY2GE6, MY2GF0, and MY2GF1.Please review the data and provide corrected Form 1s.
  
5. The SDG Narrative states that the sampler did not designate a sample for laboratory QC. The laboratory used sample MY2GF4 for laboratory QC as specified on the COC. Please review the data and provide a corrected narrative.

**ANALYTICAL RESULTS**

Case No. : 35241 SDG No. : MY2GE5  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 11, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (As Received)**

Analysis Type : Low and Medium Concentration  
 Dust Samples For Total Metals

Station Location :	HDI-101-07-047			WDI-101-12-093			HDI-101-07-099			HDI-101-09-111B			HDI-1X			HDI-101-07-149		
Sample ID :	MY2GE5			MY2GE6			MY2GE7			MY2GE8 D1			MY2GE9 D1			MY2GF0		
Collection Date :	5/17/2006			5/17/2006			5/17/2006			5/17/2006			5/17/2006			5/17/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	5970			30800			13800			11000			13900			9030		
ANTIMONY	3.5	J	D	110	J	D	8.2	J	D	13.4	J	D	11.6	J	D	1.3	J	D
ARSENIC	43.7			10.2			119			130			170			8.3		
BARIIUM	298			459			210			171			217			137L	J	A
BERYLLIUM	0.16L	J	A	0.17L	J	A	0.19L	J	A	0.20L	J	A	0.27L	J	A	0.21L	J	A
CADMIUM	8.2	J	C	5.0	J	C	28.6	J	C	31.8	J	C	40.0	J	C	2.2	J	C
CALCIUM	76300	J	E	22300	J	E	51100	J	E	23900	J	E	24900	J	E	95100	J	E
CHROMIUM	41.4			20.1			24.9			24.7			23.1			17.5		
COBALT	31.1			5.5L			31.8			40.9			35.0			8.6L	J	A
COPPER	6670			915			29100			25800		F	37000		F	1420		
IRON	17500			16300			78300			171000		F	88900		F	19000		
LEAD	816	J	E	227	J	E	329	J	E	400	J	E	505	J	E	107	J	E
MAGNESIUM	6040	J	E	5400	J	E	7220	J	E	6710	J	E	8510	J	E	8830	J	E
MANGANESE	211	J	D	235	J	D	448	J	D	702	J	D	511	J	D	305	J	D
MERCURY	1.1			0.069			0.45			0.91			0.98			1.4		
NICKEL	20.2			32.6			41.9			82.4			57.3			24.3		
POTASSIUM	3190			3040			3000			3760			4720			2530		
SELENIUM	11.4	J	CD	2.5	J	CD	23.2	J	CD	24.3	J	CDF	36.2	J	CDF	1.3	J	CD
SILVER	6.7			0.96			10.3			20.3			30.2			0.97		
SODIUM	8930			6700			5310			12200			9650			7640		
THALLIUM	0.70U	J	C	0.70U	J	C	0.70U	J	C	0.70U	J	C	0.70U	J	C	0.70U	J	C
VANADIUM	20.7			21.8			31.9			39.6			52.7			29.2		
ZINC	2980	J	E	7220	J	E	9530	J	E	28400	J	EF	15000	J	EF	497	J	E
MOLYBDENUM	53.5			13.2			233			225			316			13.5		
BORON	32.4	J	E	71.2	J	E	33.1	J	E	26.9	J	EF	17.6	J	EF	27.2	J	E
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Detection Limit

**ANALYTICAL RESULTS**

Case No. : 35241                      SDG No. : MY2GE5  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 11, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (As Received)**

Analysis Type : Low and Medium Concentration  
 Dust Samples For Total Metals

Station Location :	HDI-101-07-111			HDA-101-07-111			HDI-101-09-077			HDA-101-09-077			HDI-101-07-09T			HDI-101-09-104		
Sample ID :	MY2GF1			MY2GF2			MY2GF3			MY2GF4			MY2GF5			MY2GF6		
Collection Date :	5/17/2006			5/17/2006			5/17/2006			5/17/2006			5/17/2006			5/17/2006		
PARAMETER	Result	Val	Com															
ALUMINUM	173000			7640			12300			9280			10000			8580		
ANTIMONY	36.7	J	D	12.7	J	D	4.3	J	D	3.1	J	D	7.3	J	D	4.2	J	D
ARSENIC	209			126			75.5			71.1			49.7			43.0		
BARIIUM	654L	J	A	138L	J	A	928			227			909			253		
BERYLLIUM	0.76L	J	A	0.26L	J	A	0.39L	J	A	0.31L	J	A	0.17L	J	A	0.25L	J	A
CADMIUM	465			23.8	J	C	14.8	J	C	8.8	J	C	8.7	J	C	9.8	J	C
CALCIUM	154000	J	E	39700	J	E	21400	J	E	28600	J	E	27800	J	E	18900	J	E
CHROMIUM	117			23.2			24.0			21.2			18.4			21.2		
COBALT	42.9L	J	A	32.3			22.2			14.6			18.2			14.0		
COPPER	28800			32200			15000			10100			15300			7970		
IRON	53200			39100			29100			19800			39000			18500		
LEAD	827	J	E	499	J	E	6050	J	E	620	J	E	186	J	E	216	J	E
MAGNESIUM	19800	J	E	5890	J	E	9070	J	E	6700	J	E	6200	J	E	6970	J	E
MANGANESE	927	J	D	254	J	D	378	J	D	275	J	D	286	J	D	274	J	D
MERCURY	NA			2.9			3.2			1.8			0.20			0.23		
NICKEL	136			43.8			34.0			24.3			42.0			25.6		
POTASSIUM	20500			2630			5880			3170			3040			5250		
SELENIUM	98.6	J	CD	31.8	J	D	16.7	J	CD	8.5	J	CD	13.6	J	CD	17.3	J	CD
SILVER	22.1			10.9			10.9			5.2			8.3			6.2		
SODIUM	39700			3930			2840			1390			3670			3870		
THALLIUM	4.7U			0.70U	J	C												
VANADIUM	84.2			34.9			51.7			38.4			28.1			35.0		
ZINC	8670	J	E	7450	J	E	1580	J	E	1220	J	E	7760	J	E	1520	J	E
MOLYBDENUM	314			248			169			119			133			127		
BORON	880	J	E	43.2	J	E	16.2	J	E	8.1	J	E	29.2	J	E	23.5	J	E
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Detection Limit

ANALYTICAL RESULTS

Case No. : 35241 SDG No. : MY2GE5  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRITY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 11, 2006

Table 1A

QUALIFIED DATA  
 Concentration in mg/kg (As Received)

Analysis Type : Low and Medium Concentration  
 Dust Samples For Total Metals

Station Location :	HDA-101-09-104			HDA-2X			HDI-101-09-070			HDA-101-09-070			HDI-101-07-061			HDA-101-07-061		
Sample ID :	MY2GF7 D2			MY2GF8 D2			MY2GF9			MY2GG0			MY2GG1			MY2GG2		
Collection Date :	5/17/2006			5/17/2006			5/17/2006			5/17/2006			5/18/2006			5/18/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	10100			9710			8320			7870			11200			10700		
ANTIMONY	2.9	J	D	2.7	J	D	3.0	J	D	2.3	J	D	6.7	J	D	10.2	J	D
ARSENIC	78.1			75.1			19.1			45.0			94.0			259		
BARIUM	192			190			3340			223			347			964		
BERYLLIUM	0.30L	J	A	0.30L	J	A	0.22L	J	A	0.52U		B	0.52U		B	0.52U		B
CADMIUM	8.8	J	C	8.4	J	C	23.0			5.6	J	C	19.4	J	C	51.4		
CALCIUM	18500	J	E	18900	J	E	45400	J	E	23800	J	E	35500	J	E	36900	J	E
CHROMIUM	15.7			15.1			29.9			25.0			33.6			23.9		
COBALT	16.2			16.1			15.9L			11.6			18.6			36.8		
COPPER	10600			10100			4300			7980			14000			34600		
IRON	20300			19600			15600			16800			22800			41700		
LEAD	546	J	E	480	J	E	54400	J	E	521	J	E	475	J	E	1100	J	E
MAGNESIUM	6800	J	E	6690	J	E	5070	J	E	5290	J	E	8410	J	E	8320	J	E
MANGANESE	300	J	D	337	J	D	209	J	D	204	J	D	294	J	D	334	J	D
MERCURY	0.80			0.81			1.4			1.4			1.5			1.0		
NICKEL	24.5			24.0			22.1			26.1			35.0			45.5		
POTASSIUM	3520			3310			3550			2530			3880			3630		
SELENIUM	9.2	J	CD	8.5	J	CD	6.6	J	CD	6.0	J	CD	18.6	J	CD	30.1	J	CD
SILVER	6.3			5.7			1.0U			4.2			9.0			13.1		
SODIUM	1690			1840			7360			1680			5040			2700		
THALLIUM	0.70U	J	C	0.70U	J	C	1.4U			0.70U	J	C	0.70U	J	C	0.70U	J	C
VANADIUM	40.4			38.5			23.9			30.2			35.2			41.3		
ZINC	675	J	E	661	J	E	8930	J	E	634	J	E	3330	J	E	3780	J	E
MOLYBDENUM	150			134			58.4			86.4			116			287		
BORON	7.4	J	E	7.6	J	E	90.5	J	E	19.5	J	E	59.7	J	E	10.1	J	E
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Detection Limit

ANALYTICAL RESULTS

Case No. : 35241 SDG No. : MY2GE5  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 11, 2006

Table 1A

QUALIFIED DATA  
 Concentration in mg/kg (As Received)

Analysis Type : Low and Medium Concentration  
 Dust Samples For Total Metals

Station Location : HDI-101-07-117			HDI-101-07-052			MDL			MRN 1337.1									
Sample ID : MY2GG3			MY2GG4						CRQL									
Collection Date : 5/18/2006			5/18/2006															
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	19100			14000			3.1			50								
ANTIMONY	3.8	J	D	3.6	J	D	0.18			0.99								
ARSENIC	39.6			31.3			0.32			0.39								
BARIIUM	173			127L	J	A	0.047			161.3								
BERYLLIUM	0.20L	J	A	0.13L	J	A	0.022			0.52								
CADMIUM	12.0	J	C	7.3	J	C	0.011			0.4								
CALCIUM	31000	J	E	39100	J	E	7.1			500								
CHROMIUM	22.0			24.2			0.13			0.4								
COBALT	14.9			13.1			0.033			9.7								
COPPER	8840			10400			0.83			16.6								
IRON	60300			19200			2.3			200								
LEAD	164	J	E	193	J	E	0.11			7.7								
MAGNESIUM	6890	J	E	6010	J	E	0.88			500								
MANGANESE	364	J	D	238	J	D	0.035			100								
MERCURY	0.18			0.38			0.042			0.05								
NICKEL	44.9			32.7			0.092			18.2								
POTASSIUM	3740			3120			2.1			500								
SELENIUM	10.0	J	CD	7.4	J	CD	0.35			0.5								
SILVER	5.7			7.0			0.067			0.5								
SODIUM	7180			3910			13.3			50								
THALLIUM	0.70U	J	C	0.70U	J	C	0.4			0.7								
VANADIUM	36.0			28.9			0.023			2								
ZINC	9710	J	E	1000	J	E	0.18			38.9								
MOLYBDENUM	83.6			66.2			0.068			2								
BORON	30.0	J	E	41.1	J	E	0.031			10								
Percent Solids	100.0%			100.0%			N/A			N/A								

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Detection Limit  
 MRN - Modification Reference Number



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00105021

DATE: October 27, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not Provided
Case No.:	35595
SDG No.:	MY2S00
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Total Metals by ICP-AES plus Boron and Molybdenum, Total Mercury, and Total Cyanide
Samples:	15 Sediment Samples (see Case Summary)
Collection Date:	August 22 and 23, 2006
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: Cynthia Gurley, 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.: 35595  
SDG No.: MY2S00  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: October 27, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2S00, MY2S02, MY2S04, MY2S06, MY2S08, MY2S10, MY2S12, MY2S14, MY2S16, MY2S18, MY2S20, MY2S22, MY2S24, MY2S26, and MY2S28  
Concentration and Matrix: Low Concentration Sediment  
Analysis: CLP Total Metals by ICP-AES plus Boron and Molybdenum, Total Mercury, and Total Cyanide  
SOW: ILM05.3 and Modification Request Number 1337.1  
Collection Date: August 22 and 23, 2006  
Sample Receipt Date: August 24, 2006  
Preparation Date: August 29 and September 1, 2006  
Analysis Date: September 5, 6, 8, and 9, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY2S02 and MY2S04  
(D2): MY2S18 and MY2S20

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Soil (PBS) and samples listed above  
Matrix Spike: MY2S12S  
Duplicates: MY2S12D  
ICP Serial Dilution: MY2S12L

Analysis: CLP Total Metals by ICP-AES plus Boron and Molybdenum, Total Mercury, and Total Cyanide

<u>Analyte</u>	<u>Sample Preparation and Digestion/Distillation Date</u>	<u>Analysis Date</u>
ICP-AES Metals	September 1, 2006	September 8 and 9, 2006
Mercury	September 1, 2006	September 5, 2006
Cyanide	August 29, 2006	September 6, 2006
Percent Solids	September 1, 2006	September 2, 2006

#### CLP PO Action

None.

## Sampling Issues

The sample coolers arrived at the laboratory with temperatures of 8.2°, 9.3°, 9.8°, and 10.0°C. These temperatures exceed the 4°±2°C temperature specified in the Statement of Work (SOW). No adverse effect on the quality of the data is expected.

## Additional Comments

The samples in this SDG were analyzed for CLP total metals by ICP-AES plus boron and molybdenum under Modified Analysis Request (MAR), Modification Reference Number 1337.1. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide was analyzed by the CLP spectrophotometric method.

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), Modification Reference Number: 1337.1, May 3, 2006;
- 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	B
5.	ICP Interference Check Sample (ICS)	No	C
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	No	D
9.	ICP Serial Dilution Analysis	No	E
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
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 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.*

- B. The following results are reported as non-detected (U) in Table 1A due to low level PBS contamination.

X Molybdenum in samples MY2S08, MY2S12, MY2S16, MY2S18, MY2S20, MY2S22, MY2S24, MY2S26, and MY2S28

X Sodium in samples MY2S08, MY2S10, MY2S12, MY2S14, MY2S16, MY2S18, MY2S20, MY2S22, MY2S24, and MY2S26

The molybdenum concentration (0.099 mg/kg) and sodium concentration (88.1 mg/kg) in PBS is greater than the MDL but less than the CRQL. Sample results that are 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.*

- C. The following results are estimated and are flagged "J" in Table 1A due to possible ICP interelement interference problems.

X Thallium in samples MY2S00, MY2S02, MY2S04, MY2S06, MY2S16, MY2S18, MY2S20, MY2S22, MY2S24, and MY2S28

Results for thallium in the samples listed above were reported from an undiluted analysis that contained iron concentrations greater than the true value specified for the ICP interference check sample (ICS). Therefore, the applied interelement correction (IEC) factor may not compensate sufficiently for the interference. The thallium results for the samples listed above may be biased low and, where non-detected, false negatives may exist.

*The ICP ICS solutions A and AB are analyzed to determine the effects of high concentrations of interfering elements on each analyte determined by ICP. Solution A consists of the interferents (Al, Ca, Fe, and Mg), and Solution AB consists of the analytes mixed with the interferents.*

*When the estimated concentration produced by the interfering element is greater than twice the CRQL and also is greater than 10% of the reported concentration of the affected element, the results of the affected elements are estimated.*

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

X Antimony in all samples

Matrix spike recovery for antimony in QC sample MY2D12S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for antimony are presented below and are based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Antimony	27	-73

Results above the MDL are considered quantitatively uncertain. Results reported for antimony in all samples 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 analytes (except silver) 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 for sample MY2S12A was obtained.

Analyte	Post-Digestion Spike, % Recovery
Antimony	84

Since the post-digestion spike recovery was acceptable, the low pre-digestion spike recovery result (27%) obtained for antimony may indicate sample non-homogeneity, 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. The following results are estimated and flagged "J" in Table 1A because ICP serial dilution results are outside method QC limits.

X Boron, lead, and nickel in all samples

The percent difference for the ICP serial dilution analysis of sample MY2W18L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Boron	+21
Lead	+12
Nickel	+11

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The boron, lead, and nickel results for the diluted sample were higher than the original. The reported boron, lead, and nickel sample results may be biased low.

*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.*



## 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.



**ANALYTICAL RESULTS**

Case No. : 35595                      SDG No. : MY2S00  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 27, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

Analysis Type : Low Concentration Sediment Samples  
 for CLP Total Metals and Cyanide

Station Location :	GR-SED-01-082206			GR-SED-02-082206			GR-SED-1X-082206			GR-SED-03-082206			GR-SED-04-082206			SPR-SED-01-082206		
Sample ID :	MY2S00			MY2S02 D1			MY2S04 D1			MY2S06			MY2S08			MY2S10		
Collection Date :	8/22/2006			8/22/2006			8/22/2006			8/22/2006			8/22/2006			8/22/2006		
PARAMETER	Result	Val	Com	Result	Val	Com												
ALUMINIUM	16200			18400			19600			20400			8850			4430		
ANTIMONY	0.57L	J	AD	0.71L	J	AD	0.64L	J	AD	0.52L	J	AD	0.47L	J	AD	0.21L	J	AD
ARSENIC	2.0			2.4			2.4			2.7			2.5			1.4		
BARIIUM	82.8L	J	A	126L	J	A	133L	J	A	158L	J	A	67.1L	J	A	101L	J	A
BERYLLIUM	0.52L	J	A	0.60			0.63L	J	A	0.67			0.35L	J	A	0.34L	J	A
CADMIUM	0.47U			0.46U			0.49U			0.50U			0.46U			0.43U		
CALCIUM	22200			22000			23700			31800			13300			8020		
CHROMIUM	11.8			8.8			10.1			10.2			6.4			4.1		
COBALT	11.9			10.5L	J	A	11.0L	J	A	10.5L	J	A	6.4L	J	A	3.4L	J	A
COPPER	57.5			74.8			80.4			71.9			49.9			9.1L	J	A
IRON	24200			19200			21000			20600			11600			6620		
LEAD	8.3L	J	AE	8.4L	J	AE	9.3L	J	AE	9.5L	J	AE	5.9L	J	AE	7.1L	J	AE
MAGNESIUM	9200			7910			8270			8400			4260			2420		
MANGANESE	508			440			497			461			286			155		
MERCURY	0.059U			0.058U			0.051L	J	A	0.099			0.059U			0.058U		
NICKEL	15.1L	J	AE	13.7L	J	AE	14.8L	J	AE	14.0L	J	AE	10.0L	J	AE	7.0L	J	AE
POTASSIUM	1210			1730			1860			1950			889			1020		
SELENIUM	1.0			0.78			0.80			0.86			0.59			0.53U		
SILVER	0.59U			0.57U			0.61U			0.63U			0.57U			0.53U		
SODIUM	748			831			892			826			621U		B	549U		B
THALLIUM	1.2	J	C	1.2	J	C	1.0	J	C	1.1	J	C	0.80U			0.75U		
VANADIUM	75.8			53.0			57.6			52.9			28.8			11.1		
ZINC	46.6			42.6L	J	A	46.9L	J	A	45.9L	J	A	24.2L	J	A	17.0L	J	A
CYANIDE	3.1U			2.9U			3.1U			3.3U			3.1U			2.7U		
MOLYBDENUM	2.4U			2.3U			2.4U			2.5U			2.5U		B	2.1U		
BORON	1.6L	J	AE	2.2L	J	AE	2.6L	J	AE	3.0L	J	AE	1.8L	J	AE	0.90L	J	AE
Percent Solids	81.3%			86.9%			81.8%			74.8%			80.5%			91.1%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit

N/A - Not Applicable

NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank,

TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35595                      SDG No. : MY2S00  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 27, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

Analysis Type : Low Concentration Sediment Samples  
 for CLP Total Metals and Cyanide

Station Location :	SPR-SED-02-082206			GR-SED-05-082206			GR-SED-06-082306			GR-SED-07-082306			GR-SED-2X-082306			GR-SED-08-082306		
Sample ID :	MY2S12			MY2S14			MY2S16			MY2S18 D2			MY2S20 D2			MY2S22		
Collection Date :	8/22/2006			8/22/2006			8/23/2006			8/23/2006			8/23/2006			8/23/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINIUM	11000			4240			11400			17200			14800			18400		
ANTIMONY	0.49L	J	AD	1.2U	J	D	0.68L	J	AD	0.58L	J	AD	0.60L	J	AD	0.70L	J	AD
ARSENIC	4.2			1.1			4.3			5.1			4.2			5.3		
BARIIUM	177L	J	A	76.4L	J	A	141L	J	A	164L	J	A	152L	J	A	170L	J	A
BERYLLIUM	0.76			0.25L	J	A	0.75			1.0			0.85			0.92		
CADMIUM	0.53U			0.50U			0.48U			0.59U			0.56U			0.68U		
CALCIUM	40600			8140			31500			31600			28100			34800		
CHROMIUM	11.8			4.3			18.4			14.7			13.6			14.7		
COBALT	6.6L	J	A	3.2L	J	A	7.9L	J	A	10.1L	J	A	9.6L	J	A	11.5L	J	A
COPPER	35.4			11.6L	J	A	68.1			134			117			142		
IRON	13700			6890			18700			19400			18400			20400		
LEAD	15.9	J	E	4.9L	J	AE	18.1	J	E	17.8	J	E	15.2	J	E	19.8	J	E
MAGNESIUM	6060			2260			5930			8550			7430			8670		
MANGANESE	399			113L	J	A	467			574			567			665		
MERCURY	0.066U			0.063U			0.077			0.088			0.091			0.10		
NICKEL	12.5L	J	AE	6.1L	J	AE	14.0L	J	AE	18.0L	J	AE	16.1L	J	AE	19.0L	J	AE
POTASSIUM	2840			969			2580			3730			3140			3490		
SELENIUM	0.66U			0.63U			0.73			0.70L	J	A	0.70U			0.82L	J	A
SILVER	0.66U			0.63U			0.61U			0.74U			0.70U			0.84U		
SODIUM	656U		B	627U		B	661U		B	750U		B	726U		B	845U		B
THALLIUM	0.60L	J	A	0.88U			0.85U	J	C	1.0U	J	C	0.81L	J	AC	1.2U	J	C
VANADIUM	26.5			14.6			47.5			40.0			39.8			42.1		
ZINC	48.0L	J	A	14.5L	J	A	51.6			63.1			55.9			66.9		
CYANIDE	3.3U			3.1U			3.3U			3.8U			3.6U			4.2U		
MOLYBDENUM	2.6U		B	2.5U			2.6U		B	3.0U		B	2.9U		B	3.4U		B
BORON	4.1L	J	AE	0.81L	J	AE	5.4L	J	AE	8.3L	J	AE	6.0L	J	AE	6.9L	J	AE
Percent Solids	76.2%			79.7%			75.7%			66.6%			68.9%			59.2%		

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit

N/A - Not Applicable

NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank,

TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35595                      SDG No. : MY2S00  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 27, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

Analysis Type : Low Concentration Sediment Samples  
 for CLP Total Metals and Cyanide

Station Location :	GR-SED-09-082306			GR-SED-10-082306			GR-SED-11-082306			MDL			CRQL					
Sample ID :	MY2S24			MY2S26			MY2S28											
Collection Date :	8/23/2006			8/23/2006			8/22/2006											
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINIUM	14900			13100			13700			3.1			50.0					
ANTIMONY	0.43L	J	AD	0.44L	J	AD	0.40L	J	AD	0.18			0.99					
ARSENIC	4.9			4.6			3.6			0.32			0.39					
BARIUM	180L	J	A	168L	J	A	140L	J	A	0.047			161.3					
BERYLLIUM	1.0			0.83			0.68L	J	A	0.022			0.52					
CADMIUM	0.59U			0.56U			0.58U			0.011			0.40					
CALCIUM	32700			28600			28100			7.1			500					
CHROMIUM	12.7			12.4			13.3			0.13			0.40					
COBALT	8.8L	J	A	8.5L	J	A	9.8L	J	A	0.033			9.7					
COPPER	93.0			91.7			107			0.083			16.6					
IRON	16900			15700			17500			2.3			200					
LEAD	21.2	J	E	18.5	J	E	13.9	J	E	0.11			7.7					
MAGNESIUM	7520			7050			7440			0.88			500					
MANGANESE	573			473			576			0.035			100					
MERCURY	0.087			0.087			0.076			0.042			0.050					
NICKEL	15.6L	J	AE	16.5L	J	AE	16.7L	J	AE	0.092			18.2					
POTASSIUM	3590			3080			2630			2.1			500					
SELENIUM	0.60L	J	A	0.51L	J	A	0.90			0.35			0.50					
SILVER	0.73U			0.69U			0.73U			0.067			0.50					
SODIUM	775U		B	751U		B	936			13.3			500					
THALLIUM	1.0U	J	C	0.97U			0.76L	J	AC	0.40			0.70					
VANADIUM	30.9			30.3			38.4			0.023			2.0					
ZINC	66.0			58.2			50.2L	J	A	0.18			38.9					
CYANIDE	3.9U			3.8U			3.7U			0.16			2.5					
MOLYBDENUM	3.1U		B	3.0U		B	2.9U		B	0.068			2.0					
BORON	6.2L	J	AE	5.5L	J	AE	5.2L	J	AE	0.031			10.0					
Percent Solids	64.5%			66.6%			67.8%			N/A			N/A					

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit

N/A - Not Applicable

NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank,

TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00105021

DATE: October 25, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not Provided
Case No.:	35595
SDG No.:	MY2W12
Laboratory:	CompuChem (LIBRTY)
Analysis:	CLP Dissolved Metals by ICP-MS plus Aluminum, Boron, Iron, and Molybdenum and Dissolved Mercury
Samples:	15 Water Samples (see Case Summary)
Collection Date:	August 22 and 23, 2006
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: Cynthia Gurley, 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.: 35595  
SDG No.: MY2W12  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: October 25, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2W12 through MY2W26  
Concentration and Matrix: Low Concentration Water  
Analysis: CLP Dissolved Metals by ICP-MS plus Aluminum, Boron, Iron, and Molybdenum and Dissolved Mercury  
SOW: ILM05.3 and Modification Request Number 1340.0  
Collection Date: August 22 and 23, 2006  
Sample Receipt Date: August 24, 2006  
Preparation Date: September 12, 2006  
Analysis Date: September 12 and 13, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY2W13 and MY2W14  
(D2): MY2W21 and MY2W22

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Water (PBW) and samples listed above  
Matrix Spike: MY2W18S  
Duplicates: MY2W18D  
ICP Serial Dilution: MY2W18L

Analysis: CLP Dissolved Metals by ICP-MS plus Aluminum, Boron, Iron, and Molybdenum and Dissolved Mercury

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-MS Metals	September 12, 2006	September 14, 2006
Mercury	September 12, 2006	September 13, 2006
Percent Solids	Not Applicable	Not Applicable

#### CLP PO Action

None.

## Sampling Issues

1. Water sample numbers on the Traffic Report/Chain of Custody (TR/COC) were incorrect. The laboratory was provided new water sample numbers from the Sample Management Office (SMO) as per Region 9 resolution. (See Attachment for corrected sample numbers.)
2. The sample coolers arrived at the laboratory with temperatures of 8.2°, 9.3°, 9.8°, and 10.0°C. These temperatures exceed the 4°±2°C temperature 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

The samples in this SDG were analyzed for CLP dissolved metals by ICP-MS plus aluminum, boron, iron, and molybdenum under Modified Analysis Request (MAR), Modification Reference Number 1340.0. Mercury was analyzed by the CLP cold vapor atomic absorption method. Cyanide analysis is specified in MAR 1340.0 and requested on the TR/COC; however, no cyanide analysis data were provided with this SDG.

Note that samples were analyzed for aluminum and iron by ICP-MS in this sample delivery group (SDG) and by ICP-AES in Case 35595, SDG MY2W13.

Sample MY2W20 was analyzed at a 2-fold dilution as required by the SOW because the 138 percent recovery for the scandium internal standard exceeds the 125 percent recovery control limit. The results for aluminum, arsenic, beryllium, boron, cadmium, chromium, cobalt, copper, iron, manganese, molybdenum, nickel, selenium, silver, vanadium, and zinc are reported from this 2-fold dilution. Sample MY2W21 was analyzed at a 2-fold dilution due to an aluminum concentration exceeding the instrument's linear range. No adverse effect on the quality of the data 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), Modification Reference Number: 1340.0, February 23, 2006;
- 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)		
	d. ICP-MS Tuning Analysis		
4.	Blanks	Yes	B
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	No	C
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	No	D
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 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.*

- B. The following results are reported as non-detected (U) in Table 1A due to low level initial calibration blank (ICB) and continuing calibration blank (CCB) contamination.

X Antimony in all samples

The antimony concentration (0.17 µg/L) in the ICB is greater than the MDL but less than the CRQL. The antimony concentrations in the CCBs range from 0.077 µg/L to 0.086 µg/L and are greater than the MDL but less than the CRQL. Sample results 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.*

- C. The following results are estimated and flagged "J" in Table 1A because of laboratory duplicate results outside method QC limits.

X Zinc in all samples

Laboratory duplicate results for sample MY2W18D do not meet  $\forall$ 20 relative percent difference (RPD) and  $\forall$ CRQL absolute difference criteria for precision as listed below.

Analyte	Laboratory Duplicate Result	CRQL
Zinc	6.6 $\mu$ g/L difference	2.0

Results for zinc in all samples are considered quantitatively uncertain.

*Duplicate analyses demonstrate the analytical precision obtained for each sample matrix. The imprecision between duplicate results may be due to high levels of solids in the sample or poor laboratory technique.*

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

X Iron, molybdenum, and nickel in all samples

The percent difference for the ICP serial dilution analysis of sample MY2W18L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Iron	+20
Molybdenum	-20
Nickel	+16

Results reported for the analytes listed above in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The iron and nickel results for the diluted sample were higher than the original. The reported iron and nickel sample results may be biased low. The molybdenum result for the diluted sample was lower than the original. 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. The following relative percent differences (RPDs) or absolute differences were obtained for the field duplicate pairs listed below.

Analyte	MY2W13 D1	MY2W21 D2
	MY2W14 D1	MY2W22 D2
	Result	Result
Aluminum	--	80 RPD
Barium	21 RPD	37 RPD
Chromium	--	2.7 µg/L difference
Cobalt	--	57 RPD
Copper	--	27 RPD
Iron	--	48 RPD
Lead	--	55 RPD
Manganese	46 RPD	49 RPD
Nickel	--	31 RPD
Zinc	21.3 µg/L difference	52 RPD

Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\sqrt{20}$  RPD or  $\sqrt{CRQL}$  absolute difference criteria for precision. The effect on the quality of the data 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, sample non-homogeneity, 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.







**ANALYTICAL RESULTS**

Case No. : 35595                      SDG No. : MY2W12  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 25, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type** : Low Concentration Water Samples  
 for Dissolved Metals by ICP-MS and  
 Dissolved Mercury

Station Location :	GR-SW-01-082206			GR-SW-02-082206			GR-SW-1X-082206			GR-SW-03-082206			GR-SW-04-082206			SPR-SW-01-082206		
Sample ID :	MY2W12			MY2W13 D1			MY2W14 D1			MY2W15			MY2W16			MY2W17		
Collection Date :	8/22/2006			8/22/2006			8/22/2006			8/22/2006			8/22/2006			8/22/2006		
PARAMETER	Result	Val	Com	Result	Val	Com												
ALUMINUM	4.3L	J	A	28.3L	J	A	84.3L	J	A	11.1L	J	A	4.0L	J	A	90.5		
ANTIMONY	2.0U		B	2.0U		B												
ARSENIC	7.0			5.6			6.5			6.3			6.0			5.6		
BARIUM	69.0			56.8		E	70.2		E	64.8			63.1			203		
BERYLLIUM	0.022L	J	A	0.66U			0.021L	J	A	0.031L	J	A	0.023L	J	A	0.66U		
CADMIUM	0.25U			0.25U														
CHROMIUM	2.0U			0.090L	J	A												
COBALT	0.32L	J	A	0.30L	J	A	0.38L	J	A	0.31L	J	A	0.30L	J	A	0.54L	J	A
COPPER	0.61L	J	A	2.6			1.1L	J	A	1.1L	J	A	0.82L	J	A	0.58L	J	A
IRON	254L	J	AD	243L	J	AD	370	J	D	260L	J	AD	233L	J	AD	829	J	D
LEAD	0.16L	J	A	0.074L	J	A	0.22L	J	A	0.21L	J	A	0.064L	J	A	0.30L	J	A
MANGANESE	6.2			12.9		E	20.5		E	5.7			4.4			89.5		
MERCURY	0.20U			0.20U														
NICKEL	2.8	J	D	2.3	J	D	2.8	J	D	2.8	J	D	2.6	J	D	3.0	J	D
SELENIUM	3.9L	J	A	0.76L	J	A	0.76L	J	A	0.85L	J	A	0.82L	J	A	2.8L	J	A
SILVER	0.36U			0.36U														
THALLIUM	1.0U			1.0U														
VANADIUM	9.7			8.9			10.2			9.8			9.6			7.7		
ZINC	2.1	J	C	4.3	J	CE	25.6	J	CE	19.1	J	C	16.3	J	C	2.8	J	C
MOLYBDENUM	4.4L	J	AD	3.9L	J	AD	4.2L	J	AD	4.4L	J	AD	4.1L	J	AD	16.7L	J	AD
BORON	115			111			113			119			112			150		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35595 SDG No. : MY2W12  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 25, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type :** Low Concentration Water Samples  
 for Dissolved Metals by ICP-MS and  
 Dissolved Mercury

Station Location :	SPR-SW-02-082206			GR-SW-05-082206			GR-SW-06-082306			GR-SW-07-082306			GR-SW-2X-082306			GR-SW-08-082306		
Sample ID :	MY2W18			MY2W19			MY2W20			MY2W21 D2			MY2W22 D2			MY2W23		
Collection Date :	8/22/2006			8/22/2006			8/23/2006			8/23/2006			8/23/2006			8/23/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ALUMINUM	9.8L	J	A	3.5L	J	A	16400			13500		E	7030		E	6.9L	J	A
ANTIMONY	2.0U		B	2.0U		B	2.0U		B	2.0U		B	2.0U		B	2.0U		B
ARSENIC	5.6			6.1			9.8			8.3			7.9			6.3		
BARIIUM	212			66.8			298			245		E	169		E	88.2		
BERYLLIUM	0.021L	J	A	0.027L	J	A	1.3L	J	A	0.94			0.55L	J	A	0.66U		
CADMIUM	0.25U			0.25U			0.42L	J	A	0.32			0.17L	J	A	0.25U		
CHROMIUM	2.0U			2.0U			9.8			6.7		E	4.0		E	2.0U		
COBALT	0.55L	J	A	0.30L	J	A	8.0			5.4		E	3.0		E	0.34L	J	A
COPPER	0.93L	J	A	2.4			43.5			29.9		E	22.7		E	2.5		
IRON	878	J	D	222L	J	AD	11700	J	D	8160	J	DE	5020	J	DE	368	J	D
LEAD	0.089L	J	A	0.059L	J	A	27.7			20.6		E	11.7		E	0.20L	J	A
MANGANESE	110			15.3			613			394		E	238		E	18.5		
MERCURY	0.20U			0.20U			0.076L	J	A	0.068L	J	A	0.035L	J	A	0.20U		
NICKEL	2.4	J	D	2.5	J	D	13.0	J	D	9.2	J	DE	6.7	J	DE	2.6	J	D
SELENIUM	1.3L	J	A	0.72L	J	A	1.3L	J	A	2.2L	J	A	1.6L	J	A	1.1L	J	A
SILVER	0.36U			0.36U			0.064L	J	A	0.053L	J	A	0.36U			0.36U		
THALLIUM	1.0U			1.0U			0.94			0.15L	J	A	0.077L	J	A	1.0U		
VANADIUM	8.0			10.7			27.6			20.1			16.8			9.9		
ZINC	2.2	J	C	4.4	J	C	68.5	J	C	48.9	J	CE	28.7	J	CE	2.8	J	C
MOLYBDENUM	21.9L	J	AD	4.3L	J	AD	2.5L	J	AD	3.9L	J	AD	5.2L	J	AD	7.0L	J	AD
BORON	175			113			127			121			126			128		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit

**ANALYTICAL RESULTS**

Case No. : 35595 SDG No. : MY2W12  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 25, 2006

**Table 1A**

**QUALIFIED DATA**  
**Concentration in ug/L**

**Analysis Type :** Low Concentration Water Samples  
 for Dissolved Metals by ICP-MS and  
 Dissolved Mercury

Station Location :			GR-SW-09-082306			GR-SW-10-082306			GR-SW-11-082306			MDL			CRQL					
Sample ID :			MY2W24			MY2W25			MY2W26											
Collection Date :			8/23/2006			8/23/2006			8/22/2006											
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com		
ALUMINUM	108			634			7.8L	J	A	2.4			87							
ANTIMONY	2.0U		B	2.0U		B	2.0U		B	0.059			2.0							
ARSENIC	6.2			6.9			6.6			0.082			1.0							
BARIUM	85.2			93.6			94.3			0.053			4.0							
BERYLLIUM	0.66U			0.027L	J	A	0.66U			0.018			0.66							
CADMIUM	0.25U			0.018L	J	A	0.25U			0.016			0.25							
CHROMIUM	0.099L	J	A	0.43L	J	A	2.0U			0.045			2.0							
COBALT	0.41L	J	A	0.74L	J	A	0.42L	J	A	0.021			1.0							
COPPER	3.9			4.2			7.9			0.021			2.0							
IRON	435	J	D	881	J	D	378	J	D	2.5			300							
LEAD	0.24L	J	A	1.1			0.084L	J	A	0.015			1.0							
MANGANESE	33.6			73.6			83.9			0.037			1.0							
MERCURY	0.20U			0.20U			0.20U			0.032			0.20							
NICKEL	2.7	J	D	3.5	J	D	2.8	J	D	0.046			1.0							
SELENIUM	0.80L	J	A	1.3L	J	A	1.2L	J	A	0.24			5.0							
SILVER	0.36U			0.36U			0.36U			0.012			0.36							
THALLIUM	1.0U			1.0U			1.0U			0.011			1.0							
VANADIUM	10.3			11.4			10.7			0.029			1.0							
ZINC	3.1	J	C	9.0	J	C	2.6	J	C	0.25			2.0							
MOLYBDENUM	5.9L	J	AD	6.4L	J	AD	6.7L	J	AD	0.044			182							
BORON	121			128			120			0.56			1.6							

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit

N/A - Not Applicable

NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank,

TB - Trip Blank, BG - Background Sample

CRQL - Contract Required Quantitation Limit



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00105021

DATE: October 24, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not Provided
Case No.:	35595
SDG No.:	MY2W13
Laboratory:	CompuChem (LIBRTY)
Analysis:	Select CLP Dissolved Metals by ICP-AES
Samples:	15 Water Samples (see Case Summary)
Collection Date:	August 22 and 23, 2006
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: Cynthia Gurley, 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.: 35595  
SDG No.: MY2W13  
Site: Asarco Hayden  
Laboratory: CompuChem (LIBRTY)  
Reviewer: Stan Kott, ESAT/LDC  
Date: October 24, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: MY2W12 through MY2W26  
Concentration and Matrix: Low Concentration Water  
Analysis: Select CLP Dissolved Metals by ICP-AES  
SOW: ILM05.3  
Collection Date: August 22 and 23, 2006  
Sample Receipt Date: August 24, 2006  
Preparation Date: September 1, 2006  
Analysis Date: September 7, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY2W13 and MY2W14  
(D2): MY2W21 and MY2W22

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank-Water (PBW) and samples listed above  
Matrix Spike: MY2W18S  
Duplicates: MY2W18D  
ICP Serial Dilution: MY2W18L

Analysis: Select CLP Dissolved Metals by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	September 1, 2006	September 7, 2006
Percent Solids	Not Applicable	Not Applicable

#### CLP PO Action

None.

#### Sampling Issues

1. Water sample identifications on the Traffic Report/Chain of Custody (TR/COC) were incorrect. The laboratory was provided new water sample identifications from the Sample Management Office (SMO) as per Region 9 resolution. (See Attachment for corrected sample numbers.)

2. The sample coolers arrived at the laboratory with temperatures of 8.2°, 9.3°, 9.8°, and 10.0°C. These temperatures exceed the 4°± 2°C temperature 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

Note that samples were analyzed for aluminum and iron by ICP-AES in this sample delivery group (SDG) and by ICP-MS in Case 35595, SDG MY2W12.

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 *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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	No	B
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	No	C
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 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.*

- B. 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 MY2W18L did not meet the 10% criterion for the analytes shown below.

Analyte	% Difference
Potassium	-19

Results reported for potassium in all samples are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. The potassium 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.*

- C. The following relative percent differences (RPDs) were obtained for field duplicate pair MY2W21 and MY2W22 and are listed below.

Analyte	RPD
Aluminum	29
Iron	28

Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\sqrt{20}$  RPD criterion for precision. The effect on the quality of the data 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, sample non-homogeneity, 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.



Attachment

**Joan Purdie**

---

**From:** Garey, David [dgarey2@fedcsc.com]  
**Sent:** Monday, August 28, 2006 11:50 AM  
**To:** 'jpurdie@compuchemlabs.com'  
**Subject:** RE: Region 09 | Case 35595 | Lab LIBRTY | Issue Multiple | FINAL

Yes, that is my error.

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

**From:** jpurdie@compuchemlabs.com [mailto:jpurdie@compuchemlabs.com]  
**Sent:** Monday, August 28, 2006 11:53 AM  
**To:** dgarey2@fedcsc.com  
**Subject:** RE: Region 09 | Case 35595 | Lab LIBRTY | Issue Multiple | FINAL

May I insert a M at the beginning of each id?

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

**From:** Garey, David [mailto:dgarey2@fedcsc.com]  
**Sent:** Monday, August 28, 2006 11:46 AM  
**To:** Alice Evans; Joan Purdie  
**Cc:** Mary O'Donnell  
**Subject:** Region 09 | Case 35595 | Lab LIBRTY | Issue Multiple | FINAL

Update to Issue 2 below.

Please find below the associated DM sample IDs for the water samples:

MY2R29 - Y2W12  
MY2S01 - Y2W13  
MY2S03 - Y2W14  
MY2S05 - Y2W15  
MY2S07 - Y2W16  
MY2S09 - Y2W17  
MY2S11 - Y2W18  
MY2S13 - Y2W19  
MY2S15 - Y2W20  
MY2S17 - Y2W21  
MY2S19 - Y2W22  
MY2S21 - Y2W23  
MY2S23 - Y2W24  
MY2S25 - Y2W25  
MY2S27 - Y2W26

Thanks,

David

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

**From:** jpurdie@compuchemlabs.com [mailto:jpurdie@compuchemlabs.com]  
**Sent:** Friday, August 25, 2006 12:15 PM  
**To:** dgarey2@fedcsc.com  
**Subject:** RE: Region 09 | Case 35595 | Lab LIBRTY | Issue Multiple |



**ANALYTICAL RESULTS**

Case No. : 35595 SDG No. : MY2W13  
 Site : ASARCO HAYDEN  
 Lab : COMPUCHEM (LIBRTY)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : October 24, 2006

**Table 1A**

**QUALIFIED DATA**  
 Concentration in ug/L

**Analysis Type :** Low Concentration Water Samples for  
 Select Dissolved Metals by ICP-AES

Station Location :		GR-SW-01-082206			GR-SW-02-082206			GR-SW-1X-082206			GR-SW-03-082206			GR-SW-04-082206			SPR-SW-01-082206		
Sample ID :		MY2W12			MY2W13 D1			MY2W14 D1			MY2W15			MY2W16			MY2W17		
Collection Date :		8/22/2006			8/22/2006			8/22/2006			8/22/2006			8/22/2006			8/22/2006		
PARAMETER	Result	Val	Com	Result	Val	Com													
ALUMINUM	200U			41.8L	J	A	90.8L	J	A	200U			200U			113L	J	A	
CALCIUM	36700			37400			38100			36500			37200			128000			
IRON	100U			28.9L	J	A	83.2L	J	A	100U			100U			52.4L	J	A	
MAGNESIUM	11900			12100			12200			11900			12000			25200			
POTASSIUM	5500	J	B	5320	J	B	5490	J	B	5500	J	B	5250	J	B	8010	J	B	
SODIUM	87400			88400			89300			87900			87400			119000			

Station Location :		SPR-SW-02-082206			GR-SW-05-082206			GR-SW-06-082306			GR-SW-07-082306			GR-SW-2X-082306			GR-SW-08-082306		
Sample ID :		MY2W18			MY2W19			MY2W20			MY2W21 D2			MY2W22 D2			MY2W23		
Collection Date :		8/22/2006			8/22/2006			8/23/2006			8/23/2006			8/23/2006			8/23/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	
ALUMINUM	1860			200U			22600			14100		C	10500		C	200U			
CALCIUM	135000			36900			91200			80700			69200			58100			
IRON	1210			100U			16500			9670		C	7300		C	100U			
MAGNESIUM	26400			11900			21100			18900			17800			16100			
POTASSIUM	8720	J	B	5460	J	B	10900	J	B	9240	J	B	8820	J	B	6100	J	B	
SODIUM	127000			86800			92100			97500			101000			102000			

Station Location :		GR-SW-09-082306			GR-SW-10-082306			GR-SW-11-082306			MDL			CRQL					
Sample ID :		MY2W24			MY2W25			MY2W26											
Collection Date :		8/23/2006			8/23/2006			8/22/2006											
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	
ALUMINUM	126L	J	A	698			200U			30.7			200						
CALCIUM	52000			55400			56400			195			5000						
IRON	87.1L	J	A	580			100U			20.5			100						
MAGNESIUM	15200			15900			16100			41.7			5000						
POTASSIUM	5990	J	B	6260	J	B	6160	J	B	11.3			5000						
SODIUM	100000			102000			103000			121			5000						

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable  
 NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

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.: 00105046

DATE: March 8, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	35920
SDG No.:	MY30P0
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 2 and 8, 2006
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: Cynthia Gurley, 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.: 35920  
SDG No.: MY30P0  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: March 8, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY30P0 through MY30P9, MY30Q0, MY30Q2  
through MY30Q9, and MY30R0  
Concentration and Matrix: Medium Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.3 and Modification Reference Number 1414.0  
Collection Date: February 8, 2006  
Sample Receipt Date: November 21, 2006  
Preparation Date: November 28 and 30, 2006  
Analysis Date: November 29, December 5 and 6, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY30P9 and MY30Q0

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples  
listed above  
Matrix Spike: MY30P0S  
Duplicates: MY30P0D  
ICP Serial Dilution: MY30P0L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	November 30, 2006	December 5 and 6, 2006
Percent Solids	November 28, 2006	November 29, 2006

#### CLP PO Action

None.

## Sampling Issues

The Traffic Report/Chain of Custody (TR/COC) record forms specified two samples, MY30P0 and MY30Q9, to be used for laboratory quality control (QC). After contacting the Sample Management Office (SMO), the laboratory selected sample MY30P0 for QC analysis. No adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1414.0.

The following samples were analyzed at the following dilutions due to copper concentrations that exceeded the instrument's linear range: two-fold – MY30P0, MY30P7, MY30P8, MY30Q6, and MY30Q9; three-fold – MY30P6 and MY30Q4; four-fold – MY30Q2; and ten-fold – MY30Q3. No adverse effect on data quality is expected.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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), Modification Reference Number: 1414.0, November 8, 2006;
- 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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	Yes	
10.	Field Duplicate Sample Analysis	Yes	
11.	Sample Quantitation	Yes	
12.	Overall Assessment	Yes	

N/A = Not Applicable

## III. OVERALL ASSESSMENT OF DATA

All of the method requirements specified in the USEPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) and Modification Reference Number 1414.0 have been met. Reported results for arsenic, copper, and lead in all of the samples were appropriate and correctly calculated.



## 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.



**ANALYTICAL RESULTS**

Case No. : 35920                      SDG No. : MY30P0  
 Site : ASARCO  
 Lab : BONNER ANALYTICAL TESTING CO. (BONNER)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : March 8, 2007

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

Analysis Type : Low Concentration Soil Samples  
 for Total Metals by ICP-AES

Station Location :		HSS-E-0-101-09-107-SU			HSS-F-0-101-09-107-SU			HSS-G-0-101-09-107-SU			HSS-H-0-101-09-107-SU			HSS-I-0-101-09-107-SU			HSS-J-1-101-09-107-SU		
Sample ID :		MY30P0			MY30P1			MY30P2			MY30P3			MY30P4			MY30P5		
Collection Date :		2/8/2006			2/8/2006			2/8/2006			2/8/2006			2/8/2006			2/8/2006		
PARAMETER	Result	Val	Com																
ARSENIC	37.9			41.3			26.1			23.0			25.2			9.9			
COPPER	8630			5480			6590			6190			5860			5070			
LEAD	566			841			144			167			652			162			
Percent Solids	98.9%			97.9%			98.9%			98.7%			98.2%			97.3%			

Station Location :		HSS-A-0-101-09-108-SU			HSS-B-0-101-09-108-RE			HSS-C-0-101-09-108-SU			HSS-D-0-101-09-108-SU			HSS-X-0-101-09-108-SU			HSS-F-0-101-09-108-SU		
Sample ID :		MY30P6			MY30P7			MY30P8			MY30P9 D1			MY30Q0 D1			MY30Q2		
Collection Date :		2/8/2006			2/8/2006			2/8/2006			2/8/2006			2/8/2006			2/8/2006		
PARAMETER	Result	Val	Com																
ARSENIC	33.1			40.2			45.9			27.5			31.8			58.7			
COPPER	16200			10300			10200			3320			3590			19700			
LEAD	133			487			2100			167			218			314			
Percent Solids	99.2%			98.6%			98.7%			98.1%			85.0%			99.1%			

Station Location :		HSS-G-0-101-09-108-SU			HSS-H-0-101-09-108-SU			HSS-I-0-101-09-108-SU			HSS-J-1-101-09-108-SU			HSS-A-0-101-09-064-SU			HSS-B-0-101-09-064-SU		
Sample ID :		MY30Q3			MY30Q4			MY30Q5			MY30Q6			MY30Q7			MY30Q8		
Collection Date :		2/8/2006			2/8/2006			2/8/2006			2/2/2006			2/8/2006			2/8/2006		
PARAMETER	Result	Val	Com																
ARSENIC	51.5			62.6			41.1			58.1			28.9			15.2			
COPPER	20600			14500			7050			11300			6120			4980			
LEAD	421			496			2470			936			140			127			
Percent Solids	99.1%			98.7%			98.1%			98.7%			98.7%			99.5%			

Station Location :		HSS-D-0-101-09-064-SU			HSS-E-0-101-09-064-SU			MDL			CRQL								
Sample ID :		MY30Q9			MY30R0														
Collection Date :		2/8/2006			2/8/2006														
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	
ARSENIC	42.1			22.7			0.44			1.0									
COPPER	10700			6660			0.12			2.5									
LEAD	294			126			0.45			1.0									
Percent Solids	99.0%			98.3%			N/A			N/A									

Val - Validity. Refer to Data Qualifiers in Table 1B.

Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.

MDL - Method Detection Limit                      N/A - Not Applicable                      NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs

FB - Field Blank, EB - Equipment Blank,

TB - Trip Blank, BG - Background Sample





**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

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.: 00105046

DATE: March 6, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	35920
SDG No.:	MY3194
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 9 and 10, 2006
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: Cynthia Gurley, 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.: 35920  
SDG No.: MY3194  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: March 6, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3194, MY3195, MY3197, MY3198, MY3199,  
MY31A0 through MY31A9, and MY31B0 through  
MY31B4  
Concentration and Matrix: Medium Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.3 and Modification Reference Number 1414.0  
Collection Date: February 9 and 10, 2006  
Sample Receipt Date: November 21, 2006  
Preparation Date: November 27 and 28, 2006  
Analysis Date: November 28 and 29, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY31A0 and MY3199  
(D2): MY31A4 and MY31A5  
(D3): MY31B2 and MY31B3

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples  
listed above

Matrix Spike: MY31A6S  
Duplicates: MY31A6D  
ICP Serial Dilution: MY31A6L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	November 28, 2006	November 29, 2006
Percent Solids	November 27, 2006	November 28, 2006

#### CLP PO Action

None.

## Sampling Issues

None.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1414.0.

Samples MY31A1, MY31B1, and MY31B4 were analyzed at two, three, and five-fold dilutions, respectively, due to copper 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), Modification Reference Number: 1414.0, November 8, 2006;
- 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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	No	A
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	Yes	
10.	Field Duplicate Sample Analysis	Yes	
11.	Sample Quantitation	Yes	
12.	Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

- A. The following results are estimated and flagged "J" in Table 1A because a laboratory duplicate result is outside method QC limits.

X Lead in all samples

The lead result for laboratory duplicate sample MY31A6D does not meet the  $\leq 35$  relative percent difference (RPD) criterion for precision as listed below.

Analyte	Laboratory Duplicate, RPD
Lead	102

Results for lead in all samples are considered quantitatively uncertain.

*Duplicate analyses demonstrate the analytical precision obtained for each sample matrix. The imprecision between duplicate results may be due to sample non-homogeneity or poor 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.



**ANALYTICAL RESULTS**

Case No. : 35920                      SDG No. : MY3194  
 Site : ASARCO  
 Lab : BONNER ANALYTICAL TESTING CO. (BONNER)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : March 6, 2007

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

Analysis Type : Low Concentration Soil Samples  
 for Select Total Metals by ICP-AES

Station Location :		HSS-X-1-101-09-088-SU			HSS-A-0-101-09-084-SU			HSS-C-0-101-09-084-SU			HSS-D-0-101-09-084-SU			HSS-E-0-101-09-084-SU			HSS-X-0-101-09-084-SU		
Sample ID :		MY31A0 D1			MY31A1			MY31A2			MY31A3			MY31A4 D2			MY31A5 D2		
Collection Date :		2/10/2006			2/10/2006			2/10/2006			2/10/2006			2/10/2006			2/10/2006		
PARAMETER	Result	Val	Com																
ARSENIC	3.0			45.6			35.4			36.6			26.9			25.9			
COPPER	122			9290			6510			7750			6550			6210			
LEAD	21.9	J	A	642	J	A	1300	J	A	1060	J	A	626	J	A	633	J	A	
Percent Solids	97.8%			98.5%			98.9%			98.7%			99.1%			98.9%			

Station Location :		HSS-F-0-101-09-084-SU			HSS-G-0-101-09-084-SU			HSS-H-0-101-09-084-RE			HSS-I-0-101-09-084-SU			HSS-J-1-101-09-084-SU			HSS-A-0-101-07-035AN-RE		
Sample ID :		MY31A6			MY31A7			MY31A8			MY31A9			MY31B0			MY31B1		
Collection Date :		2/10/2006			2/10/2006			2/10/2006			2/10/2006			2/10/2006			2/9/2006		
PARAMETER	Result	Val	Com	Result	Val	Com													
ARSENIC	35.0			19.9			34.3			28.9			8.5			17.3			
COPPER	7710			4490			7100			7930			707			19100			
LEAD	1100	J	A	511	J	A	1840	J	A	951	J	A	152	J	A	48.1	J	A	
Percent Solids	98.5%			98.7%			98.8%			98.5%			97.4%			98.6%			

Station Location :		HSS-B-0-101-07-035AN-SU			HSS-X-0-101-07-035AN-SU			HSS-C-0-101-07-035AN-SU			HSS-E-0-101-09-088-SU			HSS-F-0-101-09-088-SU			HSS-H-0-101-09-088-SU		
Sample ID :		MY31B2 D3			MY31B3 D3			MY31B4			MY3194			MY3195			MY3197		
Collection Date :		2/9/2006			2/9/2006			2/9/2006			2/10/2006			2/10/2006			2/10/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	
ARSENIC	13.3			12.8			54.9			20.9			10.9			7.3			
COPPER	7640			7510			31100			4060			2220			1270			
LEAD	78.4	J	A	68.1	J	A	128	J	A	704	J	A	196	J	A	215	J	A	
Percent Solids	96.8%			96.6%			99.1%			97.5%			97.9%			97.7%			

Station Location :		HSS-I-0-101-09-088-SU			HSS-J-1-101-09-088-SU			MDL			CRQL								
Sample ID :		MY3198			MY3199 D1			MDL			CRQL								
Collection Date :		2/10/2006			2/10/2006														
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	
ARSENIC	7.5			3.1			0.44			1.0									
COPPER	761			123			0.12			2.5									
LEAD	117	J	A	23.6	J	A	0.45			1.0									
Percent Solids	97.6%			97.7%			N/A			N/A									

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit                      N/A - Not Applicable                      NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

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.: 00105046

DATE: March 6, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	35920
SDG No.:	MY31Q2
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 2, 6, and 7, 2006
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: Cynthia Gurley, 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.: 35920  
SDG No.: MY31Q2  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: March 6, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY31Q2 through MY31Q9, MY31R0 through  
MY31R9, MY31S0, and MY31S1  
Concentration and Matrix: Medium Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.3 and Modification Reference Number 1414.0  
Collection Date: February 2, 6, and 7, 2006  
Sample Receipt Date: November 28, 2006  
Preparation Date: November 30 and December 1, 2006  
Analysis Date: December 1, 13, and 14, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY31Q9 and MY31R0  
(D2): MY31S1 and MY31S2 (See Additional Comments)

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples  
listed above  
Matrix Spike: MY31R1S  
Duplicates: MY31R1D  
ICP Serial Dilution: MY31R1L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	December 1, 2006	December 13 and 14, 2006
Percent Solids	November 30, 2006	December 1, 2006

#### CLP PO Action

None.

## Sampling Issues

1. The Traffic Report/Chain of Custody (TR/COC) record forms specified two samples, MY31R1 and MY31S0, to be used for laboratory quality control (QC). The laboratory selected sample MY31R1 for QC analysis. The effect on data quality is not known.
2. The cooler containing samples MY31Q1 through MY31Q9 and MY31R0 through MY31R5 arrived at the laboratory with a temperature of 9.5°C. This temperature exceeds the temperature of 4°±2°C specified in the Statement of Work (SOW). Since these soil samples were only analyzed for arsenic, copper, and lead, no adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1414.0.

The recovery results for copper in CRQL Check Standard (CRI) CRI06 and CRI08 were 220 percent and 148 percent, respectively. These results exceed the 70-130 percent control limits specified in the SOW. However, Region 9 advised the laboratory that copper results that are greater than five times the CRQL (12.5 mg/kg) do not have to be re-analyzed and may be reported. See attached e-mails.

Samples MY31Q2, MY31R4, MY31R8, MY31R9, and MY31S0 were analyzed at a three-fold dilution due to copper concentrations that exceeded the instrument's linear range. No adverse effect on data quality is expected.

The results for sample MY31S2, the field duplicate of sample MY31S1, are included in Case: 35920 SDG: MY31S2.

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), Modification Reference Number: 1414.0, November 8, 2006;
- 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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	No	A
10.	Field Duplicate Sample Analysis	Yes	
11.	Sample Quantitation	Yes	
12.	Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

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

X Lead in all samples

The percent difference for the ICP serial dilution analysis of sample MY31R1L did not meet the 10% criterion for lead as shown below.

Analyte	% Difference
Lead	+13

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

*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.*



## 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.



Laboratory E-Mail Attachment (page 1 of 2)

Page 2 of 5

000194

**From:** Nebelsick.John@epamail.epa.gov [mailto:Nebelsick.John@epamail.epa.gov]  
**Sent:** Monday, December 11, 2006 10:55 AM  
**To:** Heather Bauer  
**Subject:** Fw: (12-6) | Case 35920 | Lab BONNER | Issue Laboratory problems

Heather, Was the response below ever sent to Bonner? He sent me another e-mail today requesting a response.

Thanks,

John Nebelsick  
Analytical Services Branch  
402-697-2572 (Omaha)  
703-603-8845 (D.C.)  
Sent by EPA Wireless E-Mail Services.

---

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

**From:** John Nebelsick  
**Sent:** 12/06/2006 04:20 PM  
**To:** Heather Bauer" <hbauer3@fedcsc.com>; Al Mayo" <amayo@fedcsc.com>  
**Subject:** Re: (12-6) | Case 35920 | Lab BONNER | Issue Laboratory problems

Heather, Please sent the response to the lab.  
Thanks,

John Nebelsick  
Analytical Services Branch  
402-697-2572 (Omaha)  
703-603-8845 (D.C.)  
Sent by EPA Wireless E-Mail Services.

---

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

**From:** "Bauer, Heather" [hbauer3@fedcsc.com]  
**Sent:** 12/06/2006 08:58 AM  
**To:** John Nebelsick; Mayo, Alfred" <amayo@fedcsc.com>  
**Subject:** (12-6) | Case 35920 | Lab BONNER | Issue Laboratory problems

John,

Following is the response from Region 9 to BONNER's issues with MA 1414.0. Please let me know if this response should be sent to BONNER.

Issue 1. Per BONNER, this flex regards about 450 samples from Region 9, analyzing for Cu, Pb and As. Currently the lab is diluting about 50 to 75% of all the samples for Cu, with a linear range of 80 ppm. (Typical dilutions are a 3X to 5X.) The lab is wondering due to the excessive levels; if the region would disregard two consecutive failed CRIs due to carry over. This is not happening all the

12/14/2006

000195

time but the lab has had to reanalyze about half of the SDGs so far. From the data they have looked at so far they don't think any samples have been below 10 to 20 ppm.

Region 9 Response: The Region presumes that this means that the CRI is above control limits due to carry over. The Region thinks that if the lab could apply the 5X rule, any samples that are at above 5X the value of the CRI can be reported regardless of whether the CRI passes or fails. Samples that are less than or equal to 5X the CRI that fails due to carryover should be reanalyzed.

Thanks,  
Heather

Heather Bauer  
CSC  
Environmental Coordinator  
(703) 818-4220  
hbauer3@fedcsc.com

-----  
This is a PRIVATE message. If you are not the intended recipient, please delete without copying and kindly advise us by e-mail of the mistake in delivery. NOTE: Regardless of content, this e-mail shall not operate to bind CSC to any order or other contract unless pursuant to explicit written agreement or government initiative expressly permitting the use of e-mail for such purpose.  
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**From:** Rudolph, Elizabeth  
**Sent:** Wednesday, December 06, 2006 8:27 AM  
**To:** Bauer, Heather  
**Cc:** Kramer, Caroline  
**Subject:** FW: (12-6) | Case 35920 | Lab BONNER | Issue Laboratory problems

Good morning Heather,

Please see Region 9's response below. Would you like me to pass this on to BONNER or wait for a response from John?

Thanks,  
Beth

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

**From:** Bauer.Richard@epamail.epa.gov [mailto:Bauer.Richard@epamail.epa.gov] **On Behalf Of** R9RSCC@epamail.epa.gov  
**Sent:** Tuesday, December 05, 2006 5:59 PM  
**To:** Rudolph, Elizabeth  
**Cc:** odonnell.mary@epa.gov; R9RSCC@epa.gov  
**Subject:** Re: (12-6) | Case 35920 | Lab BONNER | Issue Laboratory problems

I presume this means that the CRI is above control limits due to carry over. I think that the lab could apply the 5X rule, any samples that are at above 5X the value of the CRI can be reported regardless of whether the CRI passes or fails. Samples that are less than or equal to 5X the a CRI that fails due to carryover should be reanalyzed.

-----  
Region 9 RSCC  
EPA Region 9 Laboratory

12/14/2006

**ANALYTICAL RESULTS**

Case No. : 35920                      SDG No. : MY31Q2  
 Site : ASARCO  
 Lab : BONNER ANALYTICAL TESTING CO. (BONNER)  
 Reviewer : Stan Kott, ESAT/LDC  
 Date : March 6, 2007

**Table 1A**

**QUALIFIED DATA**  
**Concentration in mg/kg (Dry Weight)**

Analysis Type : Low Concentration Soil Samples  
 for Select Total Metals by ICP-AES

Station Location :		HSS-D-0-101-07-047-RE			HSS-E-0-101-07-047-SU			HSS-G-0-101-07-047-SU			HSS-H-0-101-07-047-SU			HSS-I-0-101-07-047-SU			HSS-J-1-101-07-047-SU		
Sample ID :		MY31Q2			MY31Q3			MY31Q4			MY31Q5			MY31Q6			MY31Q7		
Collection Date :		2/6/2006			2/6/2006			2/6/2006			2/6/2006			2/6/2006			2/6/2006		
PARAMETER	Result	Val	Com																
ARSENIC	65.3			22.3			31.6			22.8			42.1			11.5			
COPPER	16100			6120			9460			6550			9960			816			
LEAD	827	J	A	327	J	A	841	J	A	329	J	A	908	J	A	60.7	J	A	
Percent Solids	97.8%			98.5%			97.1%			98.1%			98.3%			96.6%			

Station Location :		HSS-A-0-101-07-062-SU			HSS-B-0-101-07-062-SU			HSS-X-0-101-07-062-SU			HSS-C-0-101-07-062-SU			HSS-D-0-101-07-062-SU			HSS-E-0-101-07-062-SU		
Sample ID :		MY31Q8			MY31Q9 D1			MY31R0 D1			MY31R1			MY31R2			MY31R3		
Collection Date :		2/2/2006			2/2/2006			2/6/2006			2/6/2006			2/6/2006			2/2/2006		
PARAMETER	Result	Val	Com																
ARSENIC	10.1			10.6			10.9			21.4			16.7			17.2			
COPPER	4330			2930			2780			8370			4420			5180			
LEAD	70.3	J	A	76.4	J	A	104	J	A	106	J	A	89.1	J	A	75.9	J	A	
Percent Solids	97.4%			98.6%			98.4%			98.5%			98.3%			97.9%			

Station Location :		HSS-G-0-101-07-062-RE			HSS-H-0-101-07-062-SU			HSS-I-0-101-07-062-SU			HSS-J-1-101-07-062-SU			HSS-C-0-101-07-089T-SU			HSS-D-0-101-07-089T-SU		
Sample ID :		MY31R4			MY31R5			MY31R6			MY31R7			MY31R8			MY31R9		
Collection Date :		2/2/2006			2/2/2006			2/2/2006			2/2/2006			2/7/2006			2/7/2006		
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com										
ARSENIC	54.7			18.8			13.9			11.6			64.2			63.6			
COPPER	13600			4750			3530			1320			19000			15500			
LEAD	253	J	A	94.1	J	A	72.2	J	A	47.5	J	A	176	J	A	189	J	A	
Percent Solids	98.4%			98.0%			98.7%			98.0%			99.2%			98.7%			

Station Location :		HSS-E-0-101-07-089T-SU			HSS-F-0-101-07-089T-SU			MDL			CRQL								
Sample ID :		MY31S0			MY31S1 D2														
Collection Date :		2/7/2006			2/7/2006														
PARAMETER	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com	
ARSENIC	32.4			60.6			0.25			1.0									
COPPER	15600			4300			0.11			2.5									
LEAD	109	J	A	90.2	J	A	0.31			1.0									
Percent Solids	99.0%			99.4%			N/A			N/A									

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable              NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) RF  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105091 Amendment 1

DATE: November 8, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	36715
SDG No.:	MY3F64
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 2 and 8, 2006
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: Cynthia Gurley, 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.: 36715  
SDG No.: MY3F64  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: November 8, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3F64 through MY3F70 and MY3H39 through MY3H51  
Concentration and Matrix: Low Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.4 and Modification Reference Number 1470.0  
Collection Date: February 2 and 8, 2006  
Sample Receipt Date: August 23, 2007  
Preparation Date: August 29, 2007  
Analysis Date: September 7 and 11, 2007

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY3F65 and MY3F70  
Field Duplicates (D2): MY3H39 and MY3H48  
Field Duplicates (D3): MY3H49 and MY3H58 (See Additional Comments)

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples listed above  
Matrix Spike: MY3H41S  
Duplicates: MY3H41D  
ICP Serial Dilution: MY3H41L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	August 29, 2007	September 7 and 11, 2007
Percent Solids	Not Prepared	Not Analyzed

#### CLP PO Action

None.

## Sampling Issues

Temperature indicator bottles were not present in the sample coolers. The sample temperature was determined by the laboratory to be 5°C. No adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1470.0.

The above MAR instructed the laboratory to dilute all samples prior to analysis. Samples MY3H50 and MY3H51 were analyzed at a two-fold dilution for arsenic, copper, and lead. Samples MY3F64 through MY3F69 and MY3H39 through MY3H49 were analyzed at a three-fold dilution for arsenic, copper, and lead. Sample MY3F70 was analyzed at a three-fold dilution for arsenic and lead and a ten-fold dilution for copper. No adverse effect on data quality is expected.

The laboratory was given permission by the Region to store the samples of this SDG in an unrefrigerated, dry secure area instead of a refrigerated storage area. The effect on data quality is not known.

The laboratory was instructed by the Region that total solids analysis was not required if the samples are dry. No adverse effect on data quality is expected.

The holding time for the soil samples in this SDG was approximately nineteen months. Holding time limits for soil samples have not been established and the effect on data quality is not known.

The results for sample MY3H58, the field duplicate of sample MY3H49, are included in Case 36715 SDG MY3H52.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1470.0, August 15, 2007;
- *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration* ILM05.4, December 2006; and
- *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	
5. ICP Interference Check Sample (ICS)	Yes	
6. Laboratory Control Sample (LCS)	Yes	
7. Duplicate Sample Analysis	Yes	
8. Matrix Spike Sample Analysis	No	A
9. ICP Serial Dilution Analysis	No	B
10. Field Duplicate Sample Analysis	No	C
11. Sample Quantitation	Yes	
12. Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

- A. The following results are estimated and flagged "J" in Table 1A because a matrix spike recovery result is outside method QC limits.

- Arsenic in all samples

Matrix spike recovery for arsenic in QC sample MY3H41S did not meet the 75-125% criteria for accuracy. The percent recovery and possible percent bias for arsenic are presented below and are based on an ideal recovery of 100%.

Analyte	% Recovery	% Bias
Arsenic	142	+42

Results above the MDL are considered quantitatively uncertain. Results reported for arsenic in all samples may be biased high.

According to the inorganic SOW, when the pre-digestion spike recovery results for ICP analytes (except silver) 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 for sample MY3H41A was obtained.

Analyte	Post-Digestion Spike, % Recovery
Arsenic	122

Since the post-digestion spike recovery was acceptable, the high pre-digestion spike recovery result (142%) obtained for arsenic may indicate sample non-homogeneity, poor laboratory technique or matrix effects which may interfere with accurate analysis, enhancing the analytical result.

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

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

- Lead in all samples

The percent difference for the ICP serial dilution analysis of sample MY3H41L did not meet the 10% criterion for lead as shown below.

Analyte	% Difference
Lead	-26

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

C. Relative percent differences (RPDs) of 81 and 158 were obtained for arsenic and copper, respectively, in the analysis of field duplicate pair samples MY3F65 and MY3F70. Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\pm 35$  RPD criterion 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, sample non-homogeneity, or poor sampling or laboratory technique.*

**ANALYTICAL RESULTS**  
**Table 1A**

SDG No. : MY3F64

Case No. : 36715

Site : ASARCO  
Lab : BONNER ANALYTICAL TESTING CO. (BONNER)  
Reviewer : Stan Kott, ESAT/LDC  
Date : November 8, 2007

Analysis Type : Low Concentration Soil Samples for  
Select CLP Total Metals by ICP-AES

**QUALIFIED DATA**  
Concentration in mg/Kg (Dry Weight)

Station Location :	HSS-E-0-101-07-089Q		HSS-F-0-101-07-089Q		HSS-G-0-101-07-089Q		HSS-H-0-101-07-089Q		HSS-I-0-101-07-089Q		HSS-J-1-101-07-089Q	
	Sample ID :	Collection Date :										
	MY3F64	2/2/2006	MY3F65	2/2/2006	MY3F66	2/2/2006	MY3F67	2/2/2006	MY3F68	2/2/2006	MY3F69	2/2/2006
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>
ARSENIC	4.7	J	A	10.1	J	AC	58.6	J	A	17.9	J	A
COPPER	3930		C	5400		C	10600		C	6380		C
LEAD	24.7	J	B	51.5	J	B	156	J	B	45.2	J	B
Percent Solids	100.0%			100.0%			100.0%			100.0%		

Station Location :	HSS-X-0-101-07-089Q		HSS-A-0-101-09-137		HSS-B-0-101-09-137		HSS-C-0-101-09-137		HSS-D-0-101-09-137		HSS-E-0-101-09-137	
	Sample ID :	Collection Date :	Sample ID :	Collection Date :	Sample ID :	Collection Date :	Sample ID :	Collection Date :	Sample ID :	Collection Date :	Sample ID :	Collection Date :
	MY3F70	2/2/2006	MY3H39	2/8/2006	MY3H40	2/8/2006	MY3H41	2/8/2006	MY3H42	2/8/2006	MY3H43	2/8/2006
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>
ARSENIC	23.7	J	AC	23.1	J	A	24.7	J	A	17.4	J	A
COPPER	45900		C	4530		C	1710		C	3690		C
LEAD	65.4	J	B	80.6	J	B	48.3	J	B	78.8	J	B
Percent Solids	100.0%			100.0%			100.0%			100.0%		

Station Location :	HSS-F-0-101-09-137		HSS-H-0-101-09-137		HSS-I-0-101-09-137		HSS-J-1-101-09-137		HSS-X-0-101-09-137		HSS-A-0-101-09-140	
	Sample ID :	Collection Date :										
	MY3H44	2/8/2006	MY3H45	2/8/2006	MY3H46	2/8/2006	MY3H47	2/8/2006	MY3H48	2/8/2006	MY3H49	2/8/2006
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>
ARSENIC	25.9	J	A	41.1	J	A	32.0	J	A	12.1	J	A
COPPER	5390		C	7370		C	8140		C	2160		C
LEAD	91.2	J	B	131	J	B	158	J	B	113	J	B
Percent Solids	100.0%			100.0%			100.0%			100.0%		

Station Location :	HSS-B-0-101-09-140		HSS-C-0-101-09-140		MDL		CRQL	
	Sample ID :	Collection Date :	Sample ID :	Collection Date :	Sample ID :	Collection Date :	Sample ID :	Collection Date :
	MY3H50	2/8/2006	MY3H51	2/8/2006				
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>
ARSENIC	7.1	J	A	34.0	J	A	1.0	
COPPER	1850		C	9040		C	2.5	
LEAD	30.4	J	B	140	J	B	1.0	
Percent Solids	100.0%			100.0%			N/A	

Val - Validity. Refer to Data Qualifiers in Table 1B.  
Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
MDL - Method Detection Limit  
N/A - Not Applicable  
NA - Not Analyzed  
D1, D2, etc. - Field Duplicate Pairs  
FB - Field Blank, EB - Equipment Blank  
TB - Trip Blank, BG - Background Sample  
CRQL - Contract Required Quantitation Limit



## 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.





**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105091 Amendment 1

DATE: November 8, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not provided
Case No.:	36715
SDG No.:	MY3GB6
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	January 31 and February 1, 2006
Reviewer:	Dennis Mayugba, 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: Cynthia Gurley, CLP PO USEPA Region 4  
Steve Remaley, CLP PO USEPA Region 9

CLP PO:  FYI  Action

SAMPLING ISSUES:  Yes  No

Attachment

00105091-8575/36715/MY3GB6RPT.doc



## Data Validation Report

Case No.: 36715  
SDG No.: MY3GB6  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Dennis Mayugba, ESAT/LDC  
Date: November 8, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3GB6 through MY3GB9, MY3GC0 through MY3GC9, and MY3GD0 through MY3GD5  
Concentration and Matrix: Total Metals Low Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.4 and Modification Reference Number 1470.0  
Collection Date: January 31 and February 1, 2006  
Sample Receipt Date: August 22, 2007  
Preparation Dates: August 28, 2007  
Analysis Date: September 5 and 6, 2007

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY3GC2 and MY3GC3  
Field Duplicates (D2): MY3GD2 and MY3GD3

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples listed above  
Matrix Spike: MY3GC7S  
Duplicates: MY3GC7D  
ICP Serial Dilution: MY3GC7L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	August 28, 2007	September 5 and 6, 2007
Percent Solids	Not Prepared	Not Analyzed

#### CLP PO Action

None.

## Sampling Issues

1. Temperature blanks were not present in the shipment of samples received on August 22, 2007 for Case number 36715. The lab was instructed to note the issue and the method used to determine temperature in the SDG narrative, and proceed with the analysis. No adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1470.0.

The above MAR instructed the laboratory to dilute all samples prior to analysis. Samples MY3GB6 through MY3GB8 and MY3GC0 through MY3GD3 were analyzed at a three-fold dilution for arsenic, copper, and lead. No adverse effect on data quality is expected.

The laboratory was given permission by the Region to store the samples of this SDG in an unrefrigerated, dry secure area instead of a refrigerated storage area. The effect on data quality is not known.

The laboratory was instructed by the Region that total solids analysis was not required if the samples are dry. No adverse effect on data quality is expected.

The holding time for the soil samples in this SDG was approximately nineteen months. Holding time limits for soil samples have not been established and the effect on data quality is not known.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1470.0, August 15, 2007;
- *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration ILM05.4*, December 2006; and
- *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)	NA	
4.	Blanks	Yes	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	No	B
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	No	C
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 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.*

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

- Arsenic in all samples

The percent difference for the ICP serial dilution analysis of sample MY3GC7L did not meet the 10% criterion for the analyte shown below.

Analyte	% Difference
Arsenic	-12

Results reported for arsenic 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 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.*

- C. A relative percent difference (RPD) of 60 was obtained for lead in the analysis of field duplicate pair samples MY3GC2 and MY3GC3. An RPD of 44 was obtained for arsenic in the analysis of field duplicate pair samples MY3GD2 and MY3GD3. Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a 35 RPD criterion 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, sample non-homogeneity, or poor sampling or laboratory technique.*

**ANALYTICAL RESULTS**  
Table 1A

Case No.: 36715  
 Site: ASARCO  
 Lab: BONNER ANALYTICAL TESTING CO. (BONNER)  
 Reviewer: Dennis Mayugba, ESAT/LDC  
 Date: November 8, 2007

Concentration in mg/Kg (Dry Weight)  
 Analysis Type: Low Concentration Soil Samples for  
 Select CLP Total Metals by ICP-AES

Station Location: Sample ID: Collection Date:	HSS-J-1-101-07-111			HSS-E-0-101-07-111			HSS-F-0-101-07-111			HSS-G-0-101-07-111			HSS-H-0-101-07-111			HSS-I-0-101-07-111		
	Result	Val	Com															
MY3GB6 2/1/2006	6.9	J	B	15.2	J	B	4.9	J	B	3.0	J	B	11.0	J	B	9.7	J	B
ARSENIC	3320			6020			2820			1220			2200			5580		
COPPER	130			150			30.1			29.2			55.9			71.4		
LEAD	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		
Percent Solids																		

Station Location: Sample ID: Collection Date:	HSS-A-0-101-07-129			HSS-X-0-101-07-129			HSS-J-1-101-07-129			HSS-B-0-101-07-129			HSS-C-0-101-07-129			HSS-E-0-101-07-129		
	Result	Val	Com															
MY3GC2 2/1/2006	16.8	J	B	17.7	J	B	5.1	J	B	33.7	J	B	28.4	J	B	24.3	J	B
ARSENIC	5220			5980			1150			9620			6060			4310		
COPPER	320		C	172		C	94.2			348			347			121		
LEAD	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		
Percent Solids																		

Station Location: Sample ID: Collection Date:	HSS-F-0-101-07-129			HSS-G-0-101-07-129			HSS-H-0-101-07-129			HSS-I-0-101-07-129			HSS-A-0-101-07-142A			HSS-X-0-101-07-142A		
	Result	Val	Com	Result	Val	Com	Result	Val	Com									
MY3GC8 2/1/2006	18.8	J	B	19.1	J	B	5.1	J	B	24.0	J	B	7.5	J	BC	11.7	J	BC
ARSENIC	6520			3120			3350			4830			1460			1960		
COPPER	187			242			71.9			139			108			136		
LEAD	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		
Percent Solids																		

Station Location: Sample ID: Collection Date:	HSS-B-0-101-07-142A			HSS-C-0-101-07-142A			CRQL			MDL		
	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
MY3GD4 1/31/2006	0.51L	J	AB	0.98L	J	AB	3.5			1.0		
ARSENIC	307			408			1.1			2.5		
COPPER	23.5			17.1			3.8			1.0		
LEAD	100.0%			100.0%			N/A			N/A		
Percent Solids												

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable NA - Not Analyzed  
 D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



## 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.





**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105091 Amendment 1

DATE: November 9, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not provided
Case No.:	36715
SDG No.:	MY3H63
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 10, 21, and 22, 2006
Reviewer:	Dennis Mayugba, 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: Cynthia Gurley, CLP PO USEPA Region 4  
Steve Remaley, CLP PO USEPA Region 9

CLP PO:  FYI  Action

SAMPLING ISSUES:  Yes  No

Attachment

00105091-8576/36715/MY3H63RPT.doc



## Data Validation Report

Case No.: 36715  
SDG No.: MY3H63  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Dennis Mayugba, ESAT/LDC  
Date: November 9, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3H63 through MY3H69, MY3H70, MY3H71, MY3H72, MY3H74 through MY3H79, and MY3H80 through MY3H83  
Concentration and Matrix: Low Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.4 and Modification Reference Number 1470.0  
Collection Date: February 10, 21, and 22, 2006  
Sample Receipt Date: August 25, 2007  
Preparation Dates: August 31, 2007  
Analysis Date: September 13, 2007

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY3H69 and MY3H70  
Field Duplicates (D2): MY3H79 and MY3H80

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples listed above  
Matrix Spike: MY3H74S  
Duplicates: MY3H74D  
ICP Serial Dilution: MY3H74L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	August 31, 2007	September 13, 2007
Percent Solids	Not Prepared	Not Analyzed

#### CLP PO Action

None.

## Sampling Issues

1. Temperature blanks were not present in the shipment of samples received on August 25, 2007 for Case number 36715, SDG MY3H63. The lab was instructed to note the issue and the method used to determine temperature in the SDG narrative, and proceed with the analysis. No adverse effect on data quality is expected.

## Additional Comments

- The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1470.0.

The above MAR instructed the laboratory to dilute all samples prior to analysis. Samples were analyzed for arsenic, copper, and lead at the following dilutions:

3-fold: MY3H68, MY3H74, MY3H75, MY3H78, MY3H82, and MY3H83;

6-fold: MY3H65, MY3H79, MY3H80, and MY3H81;

8-fold: MY3H64;

10-fold: MY3H72;

12-fold: MY3H76;

15-fold: MY3H66, MY3H67, and MY3H71;

20-fold: MY3H69 and MY3H70;

30-fold: MY3H63 and MY3H77. No adverse effect on data quality is expected.

The laboratory was given permission by the Region to store the samples of this SDG in an unrefrigerated, dry secure area instead of a refrigerated storage area. The effect on data quality is not known.

The laboratory was instructed by the Region that total solids analysis was not required if the samples are dry. No adverse effect on data quality is expected.

The holding time for the soil samples in this SDG was approximately nineteen months. Holding time limits for soil samples have not been established and the effect on data quality is not known.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1470.0, August 15, 2007;
- *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration ILM05.4*, December 2006; and

- 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	
5. ICP Interference Check Sample (ICS)	Yes	
6. Laboratory Control Sample (LCS)	Yes	
7. Duplicate Sample Analysis	Yes	
8. Matrix Spike Sample Analysis	Yes	
9. ICP Serial Dilution Analysis	Yes	
10. ICP-MS Internal Standards	N/A	
11. Field Duplicate Sample Analysis	Yes	
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 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.*



**ANALYTICAL RESULTS**  
Table 1A

SDG No.: MY3H63

Case No.: 36715

Site: ASARCO

Lab: BONNER ANALYTICAL TESTING CO. (BONNER)

Reviewer: Dennis Mayugba, ESAT/LDC

Date: November 9, 2007

Analysis Type: Low Concentration Soil Samples for  
Select CLP Total Metals by ICP-AES

**QUALIFIED DATA**  
Concentration in mg/Kg (Dry Weight)

Station Location: Sample ID: Collection Date:	KS-11-SED-0 MY3H63 2/21/2006			KS-12-SED-0 MY3H64 2/21/2006			KS-13-SED-0 MY3H65 2/21/2006			KS-14-SED-0 MY3H66 2/10/2006			KS-15-SED-0 MY3H67 2/21/2006			PCON-01-SED-1 MY3H68 2/21/2006		
	Result	Val	Com	Result	Val	Com												
ARSENIC	106			54.2			38.5			90.7			70.2			30.1		
COPPER	66800			31400			9420			48600			19800			7170		
LEAD	172			242			179			450			254			67.7		
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Station Location: Sample ID: Collection Date:	PCON-02-SED-1 MY3H69 2/21/2006			PCON-X1-SED-1 MY3H70 2/21/2006			PCON-03-SED-1 MY3H71 2/21/2006			PCON-04-SED-1 MY3H72 2/21/2006			PCON-08-SED-0 MY3H74 2/22/2006			PCON-09-SED-0 MY3H75 2/22/2006		
	Result	Val	Com															
ARSENIC	19.2L	J	A	24.6			65.4			43.5			5.3			8.5		
COPPER	40100			41100			19100			20700			3540			3800		
LEAD	48.5			50.2			259			117			18.8			37.5		
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Station Location: Sample ID: Collection Date:	PCON-10-SED-0 MY3H76 2/22/2006			PCON-12-SED-0 MY3H77 2/22/2006			PCON-13-SED-0 MY3H78 2/22/2006			PCON-15-SED-0 MY3H79 2/22/2006			PCON-X2-SED-0 MY3H80 2/22/2006			PCON-16-SED-0 MY3H81 2/22/2006		
	Result	Val	Com															
ARSENIC	34.8			68.1			27.8			18.7			21.2			13.4		
COPPER	24900			69300			5740			10900			11200			9510		
LEAD	72.6			163			84.3			62.2			51.6			47.4		
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Station Location: Sample ID: Collection Date:	PCON-17-SED-0 MY3H82 2/22/2006			PCON-18-SED-0 MY3H83 2/22/2006			MDL			CRQL		
	Result	Val	Com	Result	Val	Com	Result	Val	Com	Result	Val	Com
ARSENIC	12.9			10.5			0.31			1.0		
COPPER	7530			2440			0.071			2.5		
LEAD	21.3			38.9			0.26			1.0		
Percent Solids	100.0%			100.0%			N/A			N/A		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
MDL - Method Detection Limit  
N/A - Not Applicable  
NA - Not Analyzed  
D1, D2, etc. - Field Duplicate Pairs  
FB - Field Blank, EB - Equipment Blank  
TB - Trip Blank, BG - Background Sample  
CRQL - Contract Required Quantitation Limit



## 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.





**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105091 Amendment 1

DATE: November 13, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	Not provided
Case No.:	36715
SDG No.:	MY3HC7
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 20, 21, and 22, 2006
Reviewer:	Dennis Mayugba, 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: Cynthia Gurley, CLP PO USEPA Region 4  
Steve Remaley, CLP PO USEPA Region 9

CLP PO:  FYI  Action

SAMPLING ISSUES:  Yes  No

Attachment

00105091-8577/36715/MY3HC7RPT.doc



## Data Validation Report

Case No.: 36715  
SDG No.: MY3HC7  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Dennis Mayugba, ESAT/LDC  
Date: November 13, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3HC7, MY3HC9, MY3HD0 through MY3HD9,  
MY3HE0 through MY3HE7  
Concentration and Matrix: Low Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.4 and Modification Reference Number 1470.0  
Collection Date: February 20, 21, and 22, 2006  
Sample Receipt Date: August 25, 2007  
Preparation Dates: August 28, 2007  
Analysis Date: September 14 and 15, 2007

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY3HD0 and MY3HD1  
Field Duplicates (D2): MY3HD9 and MY3HE0

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples  
listed above  
Matrix Spike: MY3HD5S  
Duplicates: MY3HD5D  
ICP Serial Dilution: MY3HD5L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	August 28, 2007	September 14 and 15, 2007
Percent Solids	Not Prepared	Not Analyzed

#### CLP PO Action

None.

## Sampling Issues

1. Temperature blanks were not present in the shipment of samples received on August 25, 2007 for Case number 36715, SDG MY3HC7. The lab was instructed to note the issue and the method used to determine temperature in the SDG narrative, and proceed with the analysis. No adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1470.0.

The above MAR instructed the laboratory to dilute samples prior to analysis. Samples were analyzed for arsenic, copper, and lead at the following dilutions:

2-fold: MY3HD2 through MY3HD5, MY3HD7, MY3HD8, MY3HE0, MY3HE1, MY3HE5, MY3HE6, and MY3HE7;

3-fold: MY3HD0, MY3HD1, MY3HD6, MY3HE3, and MY3HE4;

4-fold: MY3HC7 and MY3HC9. No adverse effect on data quality is expected.

The laboratory was given permission by the Region to store the samples of this SDG in an unrefrigerated, dry secure area instead of a refrigerated storage area. The effect on data quality is not known.

The laboratory was instructed by the Region that total solids analysis was not required if the samples are dry. No adverse effect on data quality is expected.

The holding time for the soil samples in this SDG was approximately nineteen months. Holding time limits for soil samples have not been established and the effect on data quality is not known.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1470.0, August 15, 2007;
- *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration ILM05.4*, December 2006; and
- *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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	Yes	
10.	ICP-MS Internal Standards	N/A	
11.	Field Duplicate Sample Analysis	Yes	
12.	Sample Quantitation	Yes	
13.	Overall Assessment	Yes	

N/A = Not Applicable

## III. OVERALL ASSESSMENT OF DATA

All of the method requirements specified in the USEPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) have been met. Reported results for total arsenic, copper, and lead in all of the samples were appropriate and correctly calculated.



**ANALYTICAL RESULTS**  
Table 1A

SDG No.: MY3HC7

Case No.: 36715

Site: ASARCO

Lab: BONNER ANALYTICAL TESTING CO. (BONNER)

Reviewer: Dennis Mayugba, ESAT/LDC

Date: November 13, 2007

Analysis Type: Low Concentration Soil Samples for  
Select CLP Total Metals by ICP-AES

**QUALIFIED DATA**  
Concentration in mg/Kg (Dry Weight)

PARAMETER	UPA-08-SED-0		UPA-09-SED-0		WSC-01-SED-0		WSC-X1-SED-0		WSC-02-SED-0		WSC-03-SED-0	
	Result	Com	Val	Com	Result	Com	Val	Com	Result	Com	Result	Com
ARSENIC	11.1		6.4		14.9		13.4		3.4		4.7	
COPPER	5160		7250		1170		1080		727		730	
LEAD	17.7		12.0		35.1		35.5		19.3		29.8	
Percent Solids	100.0%		100.0%		100.0%		100.0%		100.0%		100.0%	

PARAMETER	WSC-04-SED-0		WSC-05-SED-0		WSC-06-SED-0		WSC-07-SED-0		WSC-09-SED-0		WSC-10-SED-0	
	Result	Com	Val	Com	Result	Com	Val	Com	Result	Com	Result	Com
ARSENIC	4.5		6.8		7.4		5.8		3.0		2.5	
COPPER	392		876		1210		216		77.8		58.2	
LEAD	16.0		20.6		42.5		25.2		9.0		8.0	
Percent Solids	100.0%		100.0%		100.0%		100.0%		100.0%		100.0%	

PARAMETER	WSC-X2-SED-0		WSC-11-SED-0		WSC-12-SED-0		WSC-13-SED-0		WSC-14-SED-0		WSC-15-SED-0	
	Result	Com	Val	Com	Result	Com	Val	Com	Result	Com	Result	Com
ARSENIC	3.6		3.5		2.3		4.1		4.5		3.1	
COPPER	62.3		58.0		306		311		198		62.1	
LEAD	8.2		7.3		20.3		22.1		13.6		6.5	
Percent Solids	100.0%		100.0%		100.0%		100.0%		100.0%		100.0%	

PARAMETER	WSC-16-SED-0		WSC-17-SED-0		CRQL		MDL	
	Result	Com	Val	Com	Result	Com	Result	Com
ARSENIC	2.8		3.2		1.0		0.31	
COPPER	20.0		60.5		2.5		0.071	
LEAD	3.4		6.4		1.0		0.26	
Percent Solids	100.0%		100.0%		N/A		N/A	

Val - Validity. Refer to Data Qualifiers in Table 1B.  
Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
MDL - Method Detection Limit  
N/A - Not Applicable NA - Not Analyzed

D1, D2, etc. - Field Duplicate Pairs  
FB - Field Blank, EB - Equipment Blank,  
TB - Trip Blank, BG - Background Sample  
CRQL - Contract Required Quantitation Limit



## 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.





**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105091 Amendment 1

DATE: November 9, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	36715
SDG No.:	MY3HR3
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 13, 2006
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: Cynthia Gurley, 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.: 36715  
SDG No.: MY3HR3  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: November 9, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3HR3 through MY3HR9, MY3HS0 through MY3HS9, and MY3HT0 through MY3HT2  
Concentration and Matrix: Low Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.4 and Modification Reference Number 1470.0  
Collection Date: February 13, 2006  
Sample Receipt Date: August 30, 2007  
Preparation Date: September 17, 2007  
Analysis Date: September 21, 2007

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY3HR8 and MY3HR9  
Field Duplicates (D2): MY3HS8 and MY3HS9

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples listed above  
Matrix Spike: MY3HS3S  
Duplicates: MY3HS3D  
ICP Serial Dilution: MY3HS3L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	September 17, 2007	September 21, 2007
Percent Solids	Not Prepared	Not Analyzed

#### CLP PO Action

None.

## Sampling Issues

Temperature indicator bottles were not present in the sample coolers. The sample temperature was determined by the laboratory to be 4°C. No adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1470.0.

The laboratory was given permission by the Region to store the samples of this SDG in an unrefrigerated, dry secure area instead of a refrigerated storage area. The effect on data quality is not known.

The laboratory was instructed by the Region that total solids analysis was not required if the samples are dry. No adverse effect on data quality is expected.

The holding time for the soil samples in this SDG was approximately nineteen months. Holding time limits for soil samples have not been established and the effect on data quality is not known.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1470.0, August 15, 2007;
- *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration ILM05.4*, December 2006; and
- *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	
5.	ICP Interference Check Sample (ICS)	Yes	
6.	Laboratory Control Sample (LCS)	Yes	
7.	Duplicate Sample Analysis	Yes	
8.	Matrix Spike Sample Analysis	Yes	
9.	ICP Serial Dilution Analysis	Yes	
10.	Field Duplicate Sample Analysis	Yes	
11.	Sample Quantitation	Yes	
12.	Overall Assessment	Yes	

N/A = Not Applicable

## III. OVERALL ASSESSMENT OF DATA

All of the method requirements specified in the USEPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) have been met. Reported results for total arsenic, copper, and lead in all of the samples were appropriate and correctly calculated.



**ANALYTICAL RESULTS**  
Table 1A

SDG No.: MY3HR3

Case No.: 36715

Site: ASARCO  
Lab: BONNER ANALYTICAL TESTING CO. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: November 9, 2007

Analysis Type: Low Concentration Soil Samples for  
Select CLP Total Metals by ICP-AES

**QUALIFIED DATA**  
Concentration in mg/Kg (Dry Weight)

Station Location :	WSS-D-0-101-12-073			WSS-E-0-101-12-073			WSS-G-0-101-12-073			WSS-H-0-101-12-073			WSS-I-0-101-12-073			WSS-A-0-101-12-075		
	Sample ID :	Result	Com															
Collection Date :	MY3HR3	2/13/2006		MY3HR4	2/13/2006		MY3HR5	2/13/2006		MY3HR6	2/13/2006		MY3HR7	2/13/2006		MY3HR8	2/13/2006	
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>															
ARSENIC	1.4			3.0			5.1			5.2			3.0			2.8		
COPPER	191			353			507			677			313			461		
LEAD	39.8			87.7			101			134			58.1			63.2		
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Station Location :	WSS-X-0-101-12-075			WSS-B-0-101-12-075			WSS-C-0-101-12-075			WSS-D-0-101-12-075			WSS-E-0-101-12-075					
	Sample ID :	Result	Com															
Collection Date :	MY3HR9	2/13/2006		MY3HS0	2/13/2006		MY3HS1	2/13/2006		MY3HS2	2/13/2006		MY3HS3	2/13/2006		MY3HS4	2/13/2006	
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>												
ARSENIC	3.2			1.6			5.6			1.1			3.0			1.8		
COPPER	444			170			796			181			313			547		
LEAD	59.2			98.1			211			17.5			72.7			36.6		
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Station Location :	WSS-F-0-101-12-075			WSS-G-0-101-12-075			WSS-I-0-101-12-075			WSS-A-0-101-12-093			WSS-X-0-101-12-093			WSS-C-0-101-12-093		
	Sample ID :	Result	Com															
Collection Date :	MY3HS5	2/13/2006		MY3HS6	2/13/2006		MY3HS7	2/13/2006		MY3HS8	2/13/2006		MY3HS9	2/13/2006		MY3HT0	2/13/2006	
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>															
ARSENIC	5.4			3.3			1.9			3.0			3.3			4.3		
COPPER	768			541			248			665			635			837		
LEAD	146			54.9			47.2			92.1			79.9			73.1		
Percent Solids	100.0%			100.0%			100.0%			100.0%			100.0%			100.0%		

Station Location :	WSS-D-0-101-12-093			WSS-E-0-101-12-093		
	Sample ID :	Result	Com	Sample ID :	Result	Com
Collection Date :	MY3HT1	2/13/2006		MY3HT2	2/13/2006	
<b>PARAMETER</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>	<b>Result</b>	<b>Val</b>	<b>Com</b>
ARSENIC	4.2			3.1		
COPPER	393			511		
LEAD	50.6			66.6		
Percent Solids	100.0%			100.0%		

Val - Validity. Refer to Data Qualifiers in Table 1B.  
Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
MDL - Method Detection Limit  
N/A - Not Applicable  
NA - Not Analyzed  
CRQL - Contract Required Quantitation Limit  
D1, D2, etc. - Field Duplicate Pairs  
FB - Field Blank, EB - Equipment Blank,  
TB - Trip Blank, BG - Background Sample  
CRQL - Contract Required Quantitation Limit



## 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.





**ICF International / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
Private Site/DOE Section, SFD-8-2

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*  
Quality Assurance (QA) Program, MTS-3

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

ESAT Contract No.: EP-W-06-041  
Technical Direction Form No.: 00105091 Amendment 1

DATE: November 9, 2007

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	None Provided
Case No.:	36715
SDG No.:	MY3J55
Laboratory:	Bonner Analytical Testing Co. (BONNER)
Analysis:	Total Arsenic, Copper, and Lead by ICP-AES
Samples:	20 Soil Samples (see Case Summary)
Collection Date:	February 13, 15, and 24, 2006
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: Cynthia Gurley, 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.: 36715  
SDG No.: MY3J55  
Site: Asarco Hayden  
Laboratory: Bonner Analytical Testing Co. (BONNER)  
Reviewer: Stan Kott, ESAT/LDC  
Date: November 9, 2007

### I. CASE SUMMARY

#### Sample Information

Samples: MY3J55 through MY3J59, MY3J60 through MY3J69,  
and MY3J70 through MY3J74  
Concentration and Matrix: Low Concentration Soil  
Analysis: Total Arsenic, Copper, and Lead by ICP-AES  
SOW: ILM05.4 and Modification Reference Number 1470.0  
Collection Date: February 13, 15, and 24, 2006  
Sample Receipt Date: August 31, 2007  
Preparation Date: September 19, 2007  
Analysis Date: September 21 and 22, 2007

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): MY3J62 and MY3J63  
Field Duplicates (D2): MY3J72 and MY3J73

#### Laboratory QC

Method Blanks & Associated Samples: Preparation Blank- Solid (PBS) and samples  
listed above  
Matrix Spike: MY3J60S  
Duplicates: MY3J60D  
ICP Serial Dilution: MY3J60L

Analysis: Total Arsenic, Copper, and Lead by ICP-AES

<u>Analyte</u>	<u>Sample Preparation and Digestion Date</u>	<u>Analysis Date</u>
ICP-AES Metals	September 19, 2007	September 21 and 22, 2007
Percent Solids	Not Prepared	Not Analyzed

#### CLP PO Action

None.

## Sampling Issues

Temperature indicator bottles were not present in the sample coolers. The sample temperature was determined by the laboratory to be 1°C. No adverse effect on data quality is expected.

## Additional Comments

The samples in this SDG were analyzed for total arsenic, copper, and lead by ICP-AES under Modified Analysis Request (MAR), Modification Reference Number 1470.0.

The laboratory was given permission by the Region to store the samples of this SDG in an unrefrigerated, dry secure area instead of a refrigerated storage area. The effect on data quality is not known.

The laboratory was instructed by the Region that total solids analysis was not required if the samples are dry. No adverse effect on data quality is expected.

The holding time for the soil samples in this SDG was approximately nineteen months. Holding time limits for soil samples have not been established and the effect on data quality is not known.

All method requirements specified in the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) 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:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Request for Quote for Modified Analysis* (SOW flexibility clause), Modification Reference Number: 1470.0, August 15, 2007;
- *USEPA Contract Laboratory Program Statement of Work For Inorganic Analysis Multi-Media, Multi-Concentration ILM05.4*, December 2006; and
- *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	
5. ICP Interference Check Sample (ICS)	Yes	
6. Laboratory Control Sample (LCS)	Yes	
7. Duplicate Sample Analysis	Yes	
8. Matrix Spike Sample Analysis	Yes	
9. ICP Serial Dilution Analysis	Yes	
10. Field Duplicate Sample Analysis	Yes	
11. Sample Quantitation	Yes	A
12. Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

- A. Results above the method detection limit (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.*



**ANALYTICAL RESULTS**  
Table 1A

SDG No. : MY3J55

Case No. : 36715

Site : ASARCO

Lab : BONNER ANALYTICAL TESTING CO. (BONNER)

Reviewer : Stan Kott, ESAT/LDC

Date : November 9, 2007

**QUALIFIED DATA**  
Concentration in mg/Kg (Dry Weight)

Analysis Type : Low Concentration Soil Samples for  
Select CLP Total Metals by ICP-AES

Station Location :	WSS-D-0-101-12-150			WSS-E-0-101-12-150			WSS-F-0-101-12-150			WSS-H-0-101-12-150			WSS-I-0-101-12-150			WSS-K-0-101-12-150		
	Sample ID :	Result	Com															
Collection Date :	MY3J55	30.2		MY3J56	52.0		MY3J57	3.2		MY3J58	4.7		MY3J59	6.2		MY3J60	22.4	
PARAMETER																		
ARSENIC		5710			6790			703			5680			1550			3230	
COPPER		673			517			98.7			59.5			110			112	
LEAD		100.0%			100.0%			100.0%			100.0%			100.0%			100.0%	
Percent Solids																		

Station Location :	WSS-L-0-101-12-150			WSS-A-0-101-12-154A			WSS-X-0-101-12-154A			WSS-C-1-101-12-154A			WSS-C-0-101-12-154A			WSS-D-0-101-12-154A		
	Sample ID :	Result	Com	Sample ID :	Result	Com	Sample ID :	Result	Com	Sample ID :	Result	Com	Sample ID :	Result	Com	Sample ID :	Result	Com
Collection Date :	MY3J61	55.9		MY3J62	4.6		MY3J63	5.6		MY3J64	0.61J	A	MY3J65	3.6		MY3J66	3.1	
PARAMETER																		
ARSENIC		4720			918			1030			39.4			487			293	
COPPER		1160			154			167			11.0			90.7			62.9	
LEAD		100.0%			100.0%			100.0%			100.0%			100.0%			100.0%	
Percent Solids																		

Station Location :	WSS-E-0-101-12-154A			WSS-F-0-101-12-154A			WSS-G-0-101-12-154A			WSS-H-0-101-12-154A			WSS-I-0-101-12-154A			WSS-A-0-101-12-155		
	Sample ID :	Result	Com	Sample ID :	Result	Com												
Collection Date :	MY3J67	2.9		MY3J68	4.0		MY3J69	6.3		MY3J70	2.7		MY3J71	2.8		MY3J72	6.5	
PARAMETER																		
ARSENIC		307			361			1070			484			482			746	
COPPER		83.7			78.9			107			102			100			1250	
LEAD		100.0%			100.0%			100.0%			100.0%			100.0%			100.0%	
Percent Solids																		

Station Location :	WSS-X-0-101-12-155			WSS-B-0-101-12-155			CRQL			MDL		
	Sample ID :	Result	Com	Sample ID :	Result	Com	Sample ID :	Result	Com	Sample ID :	Result	Com
Collection Date :	MY3J73	7.1		MY3J74	1.3							
PARAMETER												
ARSENIC		899			264							
COPPER		1510			96.3							
LEAD		100.0%			100.0%							
Percent Solids												

Val - Validity. Refer to Data Qualifiers in Table 1B.  
 Com - Comments. Refer to the Corresponding Section in the Narrative for each letter.  
 MDL - Method Detection Limit  
 N/A - Not Applicable NA - Not Analyzed  
 D1, D2, etc. - Field Duplicate Pairs  
 FB - Field Blank, EB - Equipment Blank,  
 TB - Trip Blank, BG - Background Sample  
 CRQL - Contract Required Quantitation Limit



## 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.





**ICF Consulting / Laboratory Data Consultants**

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

MEMORANDUM

TO: John Hillenbrand, Remedial Project Manager  
CWA Compliance Office, WTR-7

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.: 00905091 Amendment 1

DATE: May 25, 2006

SUBJECT: Review of Analytical Data, Tier 3

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

Site:	Asarco Hayden
Site Account No.:	09 JS LA00
CERCLIS ID No.:	NA
Case No.:	35104
SDG No.:	Y2ES5
Laboratory:	A4 Scientific, Inc. (A4)
Analysis:	Volatiles
Samples:	7 Water Samples (see Case Summary)
Collection Date:	March 2, 3, and 6, 2006
Reviewer:	Calvin Tanaka, ESAT/Laboratory Data Consultants

This report has been reviewed by the EPA TOPO for the ESAT contract, whose signature appears above.

Table 1A is not provided with this report. Please contact Rose Fong (QA Program/EPA) at (415) 972-3812 if Table 1A is needed.

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.: 35104  
SDG No.: Y2ES5  
Site: Asarco Hayden  
Laboratory: A4 Scientific, Inc.  
Reviewer: Calvin Tanaka, ESAT/LDC  
Date: May 25, 2006

### I. CASE SUMMARY

#### Sample Information

Samples: Y2ER9, Y2ES1, Y2ES3, Y2ES5, Y2ET2, Y2ET3, and Y2ET4  
Concentration and Matrix: Trace Concentration Water  
Analysis: Volatiles  
SOW: SOM01.1  
Collection Date: March 2, 3, and 6, 2006  
Sample Receipt Date: March 8, and 9, 2006  
Extraction Date: Not Applicable  
Analysis Date: March 13, 15, and 16, 2006

#### Field QC

Field Blanks (FB): Not Provided  
Equipment Blanks (EB): Y2ET4  
Background Samples (BG): Not Provided  
Field Duplicates (D1): Not Provided

#### Laboratory QC

Method Blanks & Associated Samples:  
VBLK17: Y2ET4  
VBLK23: Y2ER9, Y2ES1, Y2ES3, Y2ES5, and Y2ET2  
VBLK25: Y2ET3, Y2ET3MS, Y2ET3MSD, storage blank  
VHBLK01

#### Tables

1B: Data Qualifier Definitions for Organic Data Review  
2: Calibration Summary

#### CLP PO Action

1. Nondetected results for 1,4-dioxane are qualified as rejected (R) due to very low response factors (<0.01) in the initial and continuing calibrations (see Comment A).
2. Nondetected result for vinyl chloride in sample Y2ET2 is qualified as rejected (R) due to a very low deuterated monitoring compound (DMC) recovery (see Comment B).

### CLP PO Attention

1. Detected results for acetone, methylene chloride, and chloroform are qualified as nondetected and estimated (U,J) due to method blank and equipment blank contamination (see Comment D).
2. Results for some analytes are qualified as estimated (J) due to calibration problems (see Comment E).
3. Results for some analytes are qualified as estimated (J) due to deuterated monitoring compound (DMC) recovery problems (see Comment F).
4. Results for some analytes in samples Y2ER9, Y2ES1, Y2ES3, Y2ES5, and Y2ET2 are qualified as estimated (J) due to internal standard (IS) area problems (see Comment G).

### Sampling Issues

1. Detected results for acetone and chloroform are qualified as nondetected and estimated (U,J) due to equipment blank contamination (see Comment D).
2. For sample Y2ET3, two vials were broken when received by the laboratory.
3. Sampler signature is missing on the traffic report & chain of custody records (attached, p. 6 and 7 in data package).

### Additional Comments

Other than laboratory and field artifacts (approximate retention times of 3.6, 4.5, 8.2, 12.5, 14.2, and 14.4 minutes), tentatively identified compounds (TICs) were found in the samples Y3ER9, Y2ET2, and Y2ET3 (see attached Form 1Js).

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 Guidelines for Suprtfund Organic Methods Data Review, January 2005.

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

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

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

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

Relative response factors (RRFs) below 0.01 were reported for the analyte listed above in the initial and continuing calibrations (see Table 2). These values are well below the 0.05 validation criterion. Since results are nondetected, false negatives may exist.

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

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

- B. The nondetected result for the following analyte should be qualified as rejected due to a very low DMC recovery (<20%) and should be flagged AR@.

{ Vinyl Chloride-d3 }

X Vinyl chloride in sample Y2ET2

A recovery of 3% was reported for the DMC vinyl chloride-d3 in sample Y2ET2, which is well below the QC limit of 65-131%. Since the result is nondetected, false negative may exist. The sample was 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.*

- C. The following results should be denoted with an AL@ qualifier, qualified as estimated, and flagged AJ@.

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.*

- D. The following results should be qualified as nondetected and estimated due to method blank and equipment blank contamination and should be flagged AU,J@.

X Methylene chloride in all samples and storage blank VHBLK01

X Acetone in sample Y2ET2

X Chloroform in samples Y2ET2 and Y2ET3

Methylene chloride was found in all method blanks and acetone and chloroform were found in equipment blank Y2ET4. 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.

*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.*

- E. Results for the following analytes should be qualified as estimated due to large percent differences (%Ds) in continuing calibrations and should be flagged AJ@.

X Carbon tetrachloride and bromoform in samples Y2ET3, Y2ET3MS, and Y2ET3MSD; method blank VBLK25; and storage blank VHBLK01

%Ds exceeded the  $\forall$ 30.0% validation criterion for the analytes listed above in the 03/15/06 continuing calibration (see Table 2).

The DMC chloroethane-d also had a %D that exceeded the  $\forall$ 30.0% validation criterion in the 03/15/06 continuing calibration (see Table 2). Quantitation of the analytes associated with this DMC may have been affected by the high %D (see attached Table 9 from the Functional Guidelines).

*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 should be qualified as estimated due to DMC recoveries outside QC limits and should be flagged AJ@.

{Toluene-d8}

X Trichloroethene, toluene, tetrachloroethene, ethylbenzene, o-xylene, m,p-xylenes, styrene, and isopropylbenzene in sample Y2ET2

{trans-1,3-Dichloropropene-d4}

X cis-1,3-dichloropropene, trans-1,3-dichloropropene, and 1,1,2-trichloroethane in sample Y2ET2

{1,4-Dioxane-d8}

X 1,4-Dioxane in samples Y2ET2, method blank VBLK25, and storage blank VHBLK01

{1,1,2,2-Tetrachloroethane-d2}

X 1,1,2,2-tetrachloroethane and 1,2-dibromo-3-chloropropane in sample Y2ET3MS

The DMC recoveries outside QC limits are shown below.

<u>Sample</u>	<u>DMC</u>	<u>% Recovery</u>	<u>QC Limits</u>
---------------	------------	-------------------	------------------

Y2ET2	Vinyl chloride-d3	3	65-131
Y2ES1	Chloroethane-d5	137	71-131
<u>Sample</u>	<u>DMC</u>	<u>% Recovery</u>	<u>QC Limits</u>
Y2ET3MS	1,1-Dichloroethene-d2	112	55-104
Y2ET3MSD	1,1-Dichloroethene-d2	106	55-104
Y2ET2	Toluene-d8	30	77-121
Y2ET2	t-1,3-Dichloropropene-d4	65	73-121
Y2ET3	1,4-Dioxane-d8	40	50-150
VBLK25	1,4-Dioxane-d8	48	50-15
VHBLK01	1,4-Dioxane-d8	48	50-150
Y2ET3MS	1,1,2,2,-Tetrachloroethane-d2	72	73-125

Detected results for affected analytes where DMC recoveries fell below QC limits may be biased low; where results are nondetected, false negatives may exist. For DMC recoveries that exceeded QC limits, only detected results for associated analytes are qualified. Recoveries for DMCs chloroethane-d5 and 1,1-dichloroethene-d2 exceeded QC limits but results were not qualified because they were nondetects. The samples were not reanalyzed.

It should be noted that the result for vinyl chloride in sample Y2ET2 was previously qualified as rejected (see Comment B).

- G. Results for the following analytes should be qualified as estimated due to low internal standard areas and should be flagged AJ@.

{ 1,4-Dichlorobenzene-d4 }

X Bromoform, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, 1,2-dibromo-3-chloropropane, 1,2,4-trichlorobenzene, and 1,2,3-trichlorobenzene in samples Y2ER9, Y2ES1, Y2ES3, Y2ES5, and Y2ET2

Internal standard areas outside QC limits are shown below.

<u>Sample</u>	<u>Internal Standard</u>	<u>Area</u>	<u>QC Limits</u>
Y2ER9	1,4-Dichlorobenzene-d4	7472	7571 - 17667
Y2ES1	1,4-Dichlorobenzene-d4	6732	7571 - 17667
Y2ES3	1,4-Dichlorobenzene-d4	7190	7571 - 17667
Y2ES5	1,4-Dichlorobenzene-d4	6698	7571 - 17667
Y2ET2	1,4-Dichlorobenzene-d4	7136	7571 - 17667

Detected results and quantitation limits for the affected analytes are considered quantitatively questionable. Where results are nondetected, false negatives may exist. The samples were not reanalyzed.

*Internal standards, introduced into every calibration standard, blank, sample, and QC sample, monitor changes in analyte response due to matrix effects and fluctuations in instrument sensitivity throughout the analytical sequence. Internal*

*standards are used to quantitate the concentration of target analytes and surrogate standards.*

**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.

Table 2  
Calibration Summary

Case No.: 35104  
 SDG No.: Y2ES5  
 Site: Asarco Hayden  
 Laboratory: A4 Scientific, Inc.  
 Reviewer: Calvin Tanaka, ESAT/LDC  
 Date: May 25, 2006

RELATIVE RESPONSE FACTORS (RRF)

	<u>RRF</u>	<u>RRF</u>	<u>RRF</u>	<u>RRF</u>
Analysis date:	3/13/06	3/7/06	3/13/06	3/13/06
Analysis time:	11:32-15:19	10:33-12:40	12:34	22:47
GC/MS I.D.:	C-5973	F-5973	C-5973	C-5973
<u>Analyte</u>	<u>Init.</u>	<u>Init.</u>	<u>Cont.</u>	<u>Cont.</u>
1,4-Dioxane	0.010	0.007	0.009	0.008
1,4-Dioxane-d8	0.010	0.006	0.007	0.008

	<u>RRF</u>	<u>RRF</u>	<u>RRF</u>	<u>RRF</u>
Analysis date:	3/15/06	3/15/06	3/15/06	3/16/06
Analysis time:	08:07	15:26	23:44	08:26
GC/MS I.D.:	C-5973	C-5973	C-5973	C-5973
<u>Analyte</u>	<u>Cont.</u>	<u>Cont.</u>	<u>Cont.</u>	<u>Cont.</u>
1,4-Dioxane	0.006	0.007	0.007	0.007
1,4-Dioxane-d8	0.007	0.008	0.007	0.006

PERCENT DIFFERENCES (%D)

	<u>%D</u>
Analysis Date:	3/15/06
Analysis Time:	23:44
GC/MS I.D.:	C-5973
<u>Analyte</u>	<u>Cont.</u>
Carbon tetrachloride	+39.0
Bromoform	+35.0
Chloroethane-d5	+30.5

- = RRF biased low; + = RRF biased high.

## ASSOCIATED SAMPLES AND METHOD BLANKS

Initial 3/13/06: All samples, method blanks, and storage blank VHBLK01  
Cont., 3/13/06 (12:34): Sample Y2ET4, method blank VBLK17  
Cont., 3/13/06 (22:47): Sample Y2ET4, method blank VBLK17  
Cont., 3/15/06 (08:07): Samples Y2ER9, Y2ES1, Y2ES3, Y2ES5, and Y2ET2, method blank VBLK23  
Cont., 3/15/06 (15:26): Samples Y2ER9, Y2ES1, Y2ES3, Y2ES5, and Y2ET2, method blank VBLK23  
Cont., 3/15/06 (23:44): Samples Y2ET3, Y2ET3MS, and Y2ET3MSD, storage blank VHBLK01, method blank VBLK25  
Cont., 3/16/06 (08:26): Samples Y2ET3, Y2ET3MS, and Y2ET3MSD, storage blank VHBLK01, method blank VBLK25