

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY ENVIRONMENTAL SCIENCE CENTER

ENVIRONMENTAL SCIENCE CENTER 701 MAPES ROAD FORT MEADE, MD 20755-5350



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DATE : June 30, 2003

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- SUBJECT: Region III Data 🕰 Review
- FRCM : Fredrick Foremark Region III ESA: RPO (3ES20)
- TO : Marjarie Easten Regional Project Manager (3HS-1)

Attached is the inorganic data validation report for the Pip Veh.'s Salvade/Hoult Road Site (Case 31620; SDG#: MCCEIE, MCCEIE, MCCEIE, MCCEIE, MCCEIE, MCCEIE, Completed by the Region III Environmental Services Assistance Tear. (ESAT) contractor under the direction of Region III FST.

If you have any questions regarding this review or the charge in format, please call me at(410) 305-2639.

 ${\tt Attachment}$

cc: Suddha Graves (E & E Wheeling)

TO #: 0011 TDE #: 0633

ANALYTICAL SERVICES AND QUALITY ASSURANCE BRANCH



DATE:	June 24, 2003	
SUBJECT:	Inorganic Data Validation (Case: 31620 SDGs: MC0E15, MC0E17 Site: Big John Salvage-Hou	IM2 Level) It Road
FROM:	Reginald Howard ^{१भ} Inorganic Data Reviewer	می Mahboobeh Mecanic می Senior Oversight Chemist
TO:	Fredrick Foreman ESAT Region 3 Project Off	ĩcer

OVERVIEW

Case 31620, Sample Delivery Groups (SDGs) MC0E15 and MC0E17, consisted of three (3) soil samples analyzed for total metals by CompuChem a Division of Liberty Analytical (LIBRTY). The sample set included one (1) field duplicate pair. Samples were analyzed in accordance with Contract Laboratory Program (CLP) Statement of Work (SOW) ILM04.1 through Routine Analytical Services (RAS) program.

SUMMARY

All samples were successfully analyzed for all Target Analyte List (TAL) parameters with exception of thallium (Tl) in sample MC0E17 (SDG MC0E17). Areas of concern with respect to data usability are listed below.

Data in this case have been impacted by outliers present in the laboratory blanks, matrix spike, laboratory duplicate, serial dilution, and Contract Required Detection Limit (CRDL) standard analyses. Details of these outliers are discussed under "Major and Minor Problems"; specific samples affected are outlined in "Table 1A" and qualified analytical results for all samples are summarized on Data Summary Forms (DSFs).

MAJOR PROBLEM

The CRDL percent recovery was extremely low (<50%) for thallium (Tl) in SDG MC0E17. The quantitation limit for this analyte in this SDG has been rejected and qualified "R" on the DSF.

MINOR PROBLEMS

The preparation blank had a reported result greater than Instrument Detection Limit (IDL) for sodium (Na) in SDG MC0E17. The detected result for this analyte in this SDG which is less than five times (<5X) blank concentration may be biased high and has been qualified "B" on the DSF.

The preparation blank had a negative value greater than the absolute value of the IDL for Tl in SDG MC0E17. The "UL" qualifier has been superseded by "R" on the DSF for this analyte in this SDG.

CRDL standard recoveries were low (<90% but >50%) for selenium (Se) and Tl in SDG MC0E15 as well as for silver (Ag) in SDG MC0E17. Low recoveries may indicate negative biases for results detected near the detection limit due to an unstable baseline. The reported result which is less than two times (<2X) CRDL and quantitation limits for these analytes in these SDGs may be biased low and have been qualified "L" and "UL", respectively, on DSFs.

Matrix spike recoveries were high (>125%) for arsenic (As) and Se in SDG MC0E17. Detected results for these analytes in this SDG may be biased high and have been qualified "K" on the DSF.

Matrix spike recoveries were low (<75% but >30%) regarding analytes listed below. Low recoveries may be attributed to matrix interferences or analyte lost during digestion process. Positive results and quantitation limits in affected samples may be biased low and have been qualified "L" and "UL", respectively, on the DSF.

<u>SDG</u>	Analytes
MC0E15	antimony (Sb), lead (Pb), manganese (Mn), Tl

MC0E17 Sb, Mn, Hg

The matrix spike recovery was extremely low (<30%) for mercury (Hg) in SDG MC0E15. Reported results for this analyte in this SDG may be biased extremely low. The "L" qualifier for this outlier has been superseded by "J" on the DSF for this analyte in this SDG.

The Relative Percent Difference (RPD) for the laboratory duplicate analysis was outside control limits (35% RPD for soil) for Hg in SDG MC0E15. Detected results are estimated and have been qualified "J" on the DSF.

Percent Differences (%Ds) for ICP serial dilution analysis were outside control limits (10%) for potassium (K) and zinc (Zn) in SDG MC0E15. Detected results for these analytes in this SDG are estimated due to possible matrix interferences and have been qualified "J" on the DSF.

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NOTES

The RPD for laboratory duplicate analysis was outside the contractual control limit $(20\% RPD, \pm CRDL)$ for aluminum (Al) and Pb in SDG MC0E17. However, RPDs for these analytes were within Region 3 established control limits (35% RPD, ±2XCRDL) for soil sample analysis. No data were qualified for these analytes based on laboratory duplicate imprecision.

Results for field duplicate pairs MC0E15 / MC0E16 did not agree well for Pb and Hg. All results for remaining analytes in this field duplicate pair were comparable.

The cooler chest used to transport samples had an interior temperature of 7.3 °C (SDG MC0E15) which is outside control limit ($4^{\circ}C \pm 2^{\circ}C$). Due to thermostability of metals, no data were qualified for this slightly elevated cooler temperature.

The laboratory has failed to comply with the rounding guidelines as instructed in SOW regarding analytes listed below. The Form Is were corrected by data reviewer due to these rounding errors.

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<u>SDG</u> MC0E15	<u>Sample #</u> MC0E15	<u>Affected Analyties</u> As
	MC0E16	barium (Ba), beryllium (Be)
MC0E17	MC0E17	Pb

Data for Case 31620, SDG MC0E15 and MC0E17, were reviewed in accordance with National Functional Guidelines for Evaluating Inorganic Analyses with Modification for use within Region III.

ATTACHMENTS

INFORMATION REGARDING REPORT CONTENT

Table 1A is a summary of qualifiers applied to the laboratory-generated results during data validation.

TABLE 1A	SUMMARY OF QUALIFIERS ON DATA SUMMARY FORMS AFTER
	DATA VALIDATION
TABLE 1B	CODES USED IN COMMENTS COLUMN OF TABLE 1A
APPENDIX A	GLOSSARY OF DATA QUALIFIER CODES
APPENDIX B	DATA SUMMARY FORM(S)
APPENDIX C	CHAIN OF CUSTODY RECORD(S)
APPENDIX D	LABORATORY CASE NARRATIVE(S)

DCN:31620.IM2

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TABLE 1ASUMMARY OF QUALIFIERS ON DATA SUMMARYFORM AFTER DATA VALIDATION

RAS: 31620 SDG#: MC0E15

	SAMPLES	POSITIVE	NON- DETECTED		
<u>ANALYTE</u>	AFFECTED	<u>VALUES</u>	VALUES	<u>BIAS</u>	COMMENTS*
Sb	MC0E15, MC0E16	L	UL	Low	MSL(65.4° o)
Рb	MC0E15, MC0E16	L		Low	MSL(63.6° ö)
Mn	MC0E15, MC0E16	L		Low	MSL(57.3%)
Hg	MC0E15, MC0E16	J			DUP(74.5%) MSE(-17.4%)
К	MC0E15, MC0E16	J		t .	ISD(14.3%)
Se	MC0E15, MC0E16	L	UL	Low	CRL(83.1%)
Tl	MC0E15, MC0E16		UL	Low	MSL(51.7%) CRL(87.4%, 86.2%)
Zn	MC0E15, MC0E16	J			ISD(11.0%)

* See explanation of Comments on Table 1B.

Page 2 of 2

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TABLE 1ASUMMARY OF QUALIFIERS ON DATA SUMMARYFORM AFTER DATA VALIDATION

RAS: 31620 SDG#: MC0E17

ANALYTE	SAMPLES AFFECTED	POSITIVE <u>VALUES</u>	NON- DETECTED VALUES	BIAS	<u>COMMENTS*</u>
Sb	MC0E17		UL	Low	MSL(47.9%)
As	MC0E17	К		High	MSH(134.7%)
Mn	MC0E17	L		Low	MSL(45.5%)
Hg	MC0E17	L		Low	MSL(61.1%)
Se	MC0E17	К		High	MSH(143.6%)
Ag	MC0E17		UL	Low	CRL(88.7%)
Na	MC0E17	В		High	PB(70.829 mg/Kg)
ΤI	MC0E17		R	Extr./ Low	CRE(46.7%) PBN(-1.632 mg/Kg)

* See explanation of Comments on Table 1B.

TABLE 1BCODES USED IN COMMENTS COLUMN

- MSL = Matrix spike recoveries were low (<75% but >30%) [% recoveries are in parenthesis]. Reported results and quantitation limits may be biased low.
- DUP = The Relative Percent Differences (RPD) for the laboratory duplicate analysis was outside control limits (±2XCRDL, 35%RPD) [the RPD is in parenthesis]. Detected results are estimated.
- MSE = The matrix spike recovery was extremely low (<30%) [% recovery is in parenthesis].Detected results may be extremely low.
- ISD = Percent Differences (%Ds) for the serial dilution analysis were outside control limit (10%) [%Ds are in parenthesis]. Detected results are estimated.
- MSH = The matrix spike recovery was high (>125%) [% recovery is in parenthesis]. The detected result may be biased high.
- CRL = The CRDL standard recovery was low (<90% but >50%) [% recovery is in parenthesis]. The quantitation limit may be biased low.
- PB = The preparation blank had a result greater than the Instrument Detection Limit (IDL) [the result is in parenthesis]. The reported result which less than five times (<5X) the blank concentration may be biased high.
- PBN = The preparation blank had a negative result with absolute value >IDL (the result is in parenthesis). The quantitation limit may be biased low.

APPENDIX A

GLOSSARY OF

DATA QUALIFIER CODES

GLOSSARY OF DATA QUALIFIER CODES (INORGANIC)

CODES RELATED TO IDENTIFICATION

(confidence concerning presence or absence of analytes):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

- B = Not detected substantially above the level reported in laboratory or field blanks.
- R = Unusable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

CODES RELATED TO QUANTITATION

(can be used for both positive results and sample quantitation limits):

- J = Analyte Present. Reported value may not be accurate or precise.
- K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.
- L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.
- [] = Analyte present. As values approach the IDL the quantitation may not be accurate.
- UJ = Not detected, quantitation limit may be inaccurate or imprecise.
- UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

APPENDIX B

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DATA SUMMARY FORMS

DATA SUMMARY FORM: INORGANIC

Case # 31620 SDG . MC0E15 Site : LIBRTY Lab.

BIG JOHN'S HOULT RD

- Number of Soil Samples 2
- Number of Water Samples 0

Sample Number		MC0E15		MC0E16							
Sampling Location		BJSD47		BJSD48							
Field QC		Field Dup of	f	Field Dup of	f						
		MC0E16		MC0E15							
Matrix		Soil		Soul							
Units :		mg/Kg		mg/Kg							
Date Sampled :	1	04/14/2003		04/14/2003							
Time Sampled		13.25		13 35							
%Solids		70.8		700							
Dilution Factor		1.0		· 0							
ANALYTE	CRDL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ALUMINUM	40	5970		6730							
ANTIMONY	12	[0.93]	L		UL						
ARSENIC	2	5.3		6.6							
BARIUM	40	78.5		89.5							
BERYLLIUM	1	[0.48]		[0 53]							
CADMIUM	1	[0.59]		1.4							
CALCIUM	1000	1550	[2440							
CHROMIUM	2	10.9		12.7							
COBALT	10	[12.1]		[10-4]							
COPPER	5	16.0	1	22.5							
IRON	20	19100		228 00							
•LEAD	0.6	19.8	L	33.8	L			,			
MAGNESIUM	1000	[1180]	Ì	[1220]					1		
MANGANESE	3	308	L	296	L						
MERCURY	0.1	0 74	J	14	J						
NICKEL	8	14.8		15 3							
POTASSIUM	1000	[837]	J	[904]	J						
SELENIUM	1	[1.1]	L		UL						
SILVER	2						1				
SODIUM	1000	[225]		[145]				i i i i i i i i i i i i i i i i i i i			
THALLIUM	2		UL		UL						
VANADIUM	10	14.5		17.2							
ZINC	4	40.5	J	53 .0	J						

CRDL = Contract Required Quantitation Limit

To calculate sample quantitation limits: (CRDL * Dilution Factor) / (%Solids/ 100)

SEE NARRATIVE FOR CODE DEFINITIONS

Revised 09/99

DATA SUMMARY FORM: INORGANIC

Case #. 31620	SDG : MC0E17
Site :	BIG JOHN'S HOULT RD
Lab. :	LIBRTY

Number of Soil Samples . 1

Number of Water Samples : 0

Sample Number		MC0E17									
Sampling Location		BJSD49								:	
Field QC											
Matrix :		Soil									
Units :		mg/Kg									
Date Sampled :		04/29/2003									
Time Sampled :		13:30									
%Solids :		79.6									
Dilution Factor:		1.0									
ANALYTE	CRDL	Result	Flag	Result	Flag	Result	Fag	Result	Flag	Result	Flag
ALUMINUM	40	7150									
ANTIMONY	12		UL								
ARSENIC	2	12.3	к								
BARIUM	40	58.1									
BERYLLIUM	1	[0.64]									
CADMIUM	1										
CALCIUM	1000	4520									
CHROMIUM	2	16.8									
COBALT	10	[9.4]									
COPPER	5	16.4									
IRON	20	32400									
*LEAD	0.6	28.5									
MAGNESIUM	1000	1520									
MANGANESE	3	300	L								
MERCURY	0.1	0.22	L								
NICKEL	8	15.5									
POTASSIUM	1000	[1230]									
SELENIUM	1	3.3	к								
SILVER	2		UL								
SODIUM	1000	[212]	в				1				
THALLIUM	2		R								
VANADIUM	10	21.8									
ZINC	4	96.3									
			1						1		

CRDL = Contract Required Quantitation Limit

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To calculate sample quantitation limits: (CRDL * Dilution Factor) / (%Solids/ 100)

SEE NARRATIVE FOR CODE DEFINITIONS

Revised 09/99

APPENDIX C

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CHAIN-OF-CUSTODY RECORDS

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1. Project Code	2. A	ccount Ologi	Code 2 2 5 371	000T03	3. R	legion	No. S	Sampling Co. E + E	, Inc.	5. Date 4-15	Shipped	Carrier F	edEx			7. Matrix (Enter in Column A)		8. Preservative (Enter in Column D)
Regional Informa	ation				Sam	pler (N 1	ame)	·		Airbill Nu	imber	010	770			1. Surface W	ater	1. HCl
Jupe	SKru	INP			1 Com	lour		BURHA	N	8 <u>24</u>	521	060	110			3. Leachate	3. NaOH	
										o. Ship to. Liberty Anglistal					4. Field 5. Soil/Sedim	ent	4. H ₂ SO ₄ 5. K ₂ CB ₂ O ₇	
Site Name	<u> </u>	~			4. P	urpose	*	Early Action	Long-Term	1	501		dison Avor	140		6. Oil (High o	nly)	6. loe only
Biaz	Sohn	\geq	ilvag	e	L	ed Zhec			FS	{	100	, n		513		8. Other (spe	cify in	in Column D)
City, State		Site	Spill ID		1 1						Car	γ, \prime				Column A)		N. Not Preserved
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CLP Sample Numbers (from labels)	A Matrix (from <u>Box 7)</u> Other:	B Conc.: Low Med High	C Sample Type: Comp./ Grab	D Preser- vative (from <u>Box 8)</u> Other:	Dies. Metala Total Metala	RA SI LON CON CON			Regior Tracki or Tag	F nal Specifik ng Numbe Numbers	c r		G Station Location Identifier	H Mo/Da Year/Tii Samp Collecti	y/ me le ion	l Corresponding CLP Organic Sample No.	J Sampler Initials	K Field QC Qualifier B = Blank S = Spike D = Duplicate R = Rineate PE = Perform Eval - = Not a QC Sample
MCOE15	5	4	G	6	X			3	-3046	973	_		BJSD47	4/14/03	1325	COF23	W	
maril	5	1_	G	6	X			3	- 3046	3976			BJSD48	4/14/03	1335	COF24	N	Dot MODELS
									Temo	ont	10	To	licadar -					
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Relinquished by:	(Signatu	ire)		Da	te / Tir	ne	Re	ceived by: (Sig	gnature)		Relinquis	shed by:	(Signature)	Date	/ Time	Received by: (S	Signeture)	
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\$EP/	United States Envin Contract L	onmental Protection Agency aboratory Program	inorga & Chain (For Inc	nlc Traffic Report of Custody Record rganic CLP Analysis)	Case No. 31621	0
1. Project Corle 2. Ac Non-Superfund Program Site Name Big John City State	Salvage	2	 Matrix (Enter In Column A) Surface Water Ground Water Leachate Field Soil/Sediment Oil (High only) Waste (High only) Other (specify in Column A) 	8. Preservative (Enter In Column D) 1. HCl 2. HNO3 3. NaOH 4. H2SO4 5. K2CR2O7 6. Ice only 7. Other (specify in Column D) N. Not Preserved		
Fairment WV Fairment WV CLP A Sample Matrix (from labels) Numbers (from labels) Box 7) Other	B C D Conc.: Sample Preser- Low Type: vative Med Comp./ (from High Grab Box 8) Other:	E - RAS Analysis E - RAS Analysis Only Only Only Tracking State of State	ATTN: Alice Evans F I Specific Number Number Numbers I Specific Number I Specific Number I Specific Number I Specific Number	H Mo/Day/ Year/Time Sample Collection	I J Corresponding Sample CLP Organic Initials Sample No.	er Field QC s Qualifier B = Blank S = Spike D = Ouplicate R = Rinsate PE = Perform Eval. Not a OC Sermie
MCOE17 5		X 3-303555	0 BISD4	1 4/29/03 137 dirator	COF26 SC	
Shipment for Case Complete? (YN)	Page Sample(s)	to be Used for Laboratory QC VCDEIT	Additional Sampler Signatures	Chai	n of Custody Seal Number((s)

Chain of Custody Record

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (S	Signatur o)	Date /	Time	Received by: (Signature)
Suddha haves	4/29/23 1600						
Relinquished by: (Signature),	Date / Time	Received by: (Signature)	Relinquished by: (S	Signature)	Date /	Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by:	Date / Time	Remarks: Is	custody seal ir	tact? Y/I	N/none
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APPENDIX D

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LABORATORY CASE NARRATIVE

U.S. EPA - CLP COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name:		COMPUCHEM		Contract: 68W00082				
Lab	Code:	LIBRTY	Case No.:	31620	SAS No.:		SDG No.:	MC0E15
SOW	No.:	ILM04.1						
		EPA	Sample No.		Lab Samp	ole ID.		
		MC0E	15		MC0E15-	1	_	
		MCOE	15D		WG23902	-2	_	
		MC0E	155		WG23902	-1	-	

MC0E15-2

Were ICP interelement corrections applied?	Yes/No	YES
Were ICP background corrections applied? If yes -were raw data generated before	Yes/No	YES
application of background corrections?	Yes/No	NO

Comments:	THE FOLLOWING ANALYTES HAVE BEEN FLAGGED WITH AN "E" TO INDICATE SERIAL
	DILUTION RESULTS WHICH ARE NOT WITHIN CONTROL LIMITS:
	POTASSIUM, ZINC
	·

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

Title:

Signature:	Thomas R. Cole	Name:
Date:	April 28, 2003	Title

MC0E16

Date Reviewer IT 3 AR115148

Date:

ILM04.1

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CompuChem

a Division of Liberty Analytical Corp. 501 Madison Avenue Cary, NC 27513

SDG NARRATIVE CASE # 31620 SDG # MC0E15 CONTRACT # 68W00082

The indicated Sample Delivery Group (SDG) consisting of two (2) soil samples was received into the laboratory information management system (LIMS) on April 16, 2003; intact and in good condition with Chains of Custody (COC) Records in order, unless otherwise noted in any attachments or Quality Assurance Notices. Sample ID's reported in this data package are noted by the receiving department on the COC if they differ from those listed by the samplers on the COC.

The samples were analyzed, in accordance with EPA - CLP Statement of Work (SOW) document ILM04.1 for CLP TAL total metals.

The correlation coefficient for the mercury analytical run is confirmed to be ≥ 0.9950 .

The cooler temperature bottle was present with samples received on April 16, 2003; and sample temperature was 7.3 degrees Celsius.

EQUATIONS FOR SOLID SAMPLE CALCULATIONS:

Client sample MC0E15 is used for illustration.

Any sample result that is < the instrument detection limit (IDL) will be entered at the IDL for that analyte.

ICP Equation:

Equation for obtaining metals sample results in mg/Kg as presented on FORM I data sheets from ICP instrument acquired results in μ g/L (ppb).

 $\begin{array}{rcl} C & x & D & x & V \\ Concentration (\% solids) & (mg/Kg) & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & \\ & & & & \\ & & & \\ & & & & \\ & & & & \\ & & & \\ & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & &$

Mercury Equation:

Equation for obtaining mercury sample results in mg/Kg as presented on FORM I data sheets from instrument acquired results in μ g/L (ppb).

A x D x F B x E Where $A = \mu g/L Hg$ B = wet weight of sample D = dilution factor to bring sample into analysis range E = % solids/100 F = final volume in liters (0.1 L)Example: mercury result $\mu g/L$ to mg/Kg1.049 $\mu g/L$ (A) x 1 (D) x 0.1 (F) 0.2 g (B) x 0.708 (E) = 0.741 mg/Kg reported as 0.74 mg/Kg

SAMPLE IDs:

The following customer IDs are associated with this SDG:

MC0E15 MC0E16

INSTRUMENTAL QUALITY CONTROL:

All calibration verification solutions (ICV & CCV), blanks (ICB, & CCB), and interference check samples (ICSA & ICSAB) associated with this data were confirmed to be within EPA CLP allowable limits.

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SAMPLE PREPARATION QUALITY CONTROL:

The sample preparation procedure verifications (LCSS & PBS) were found to be within acceptable ranges and all field samples were prepared and analyzed within the contract specified holding times.

MATRIX RELATED QUALITY CONTROL:

The sample matrix spike, CCN = WG23902-1 (MC0E15S) was found to be outside CLP control limits for antimony, lead, manganese, mercury, and thallium. The reported concentrations for these analytes are flagged with an "N" on all associated Form 1 and on Form 5a.

An "N" indicates a matrix-related interference in the sample preparation procedure &/or analysis for the flagged analyte. This is normally the consequence of a relatively high anionic content in the sample or (for some sediments) an inconsistent sample matrix relative to that analyte.

CLP control limits for matrix spike recoveries are set at 75% to 125% of the analyte quantity added unless original sample concentrations exceed the true values of these "spikes" by a factor of four or more. In this case, affected analytes are not flagged even if recoveries are outside percentage recovery control limits.

Post-digestion spikes are mandatory for analytes demonstrating unsatisfactory matrix spike recoveries during ICP analysis (excluding silver). The results of such spikes are presented on Form 5b.

Unsatisfactory recovery of post-digestion spikes of this type do not have bearing upon the aforementioned "N" flags, but may indicate interference during analysis &/or a solution matrix which is hostile to the analyte in question.

Satisfactory recovery of an analyte in a post-digestion spike of this type implies interference by the required preparation procedure or in the sample matrix itself. Lack of uniformity for an analyte in sediments will also result in satisfactory recovery of post-digestion spikes after failure in the related matrix spike.

The sample matrix duplicate, CCN = WG23902-1 (MC0E15D) was outside CLP control limits for mercury. The reported concentration for this analyte is flagged with a "*" on all associated Form 1 and on Form 6.

A "*" indicates a non-homogeneous sample matrix in regard to the flagged analyte. This is normally the consequence of a relatively coarse texture or of a mixed-matrix in sediment samples.

CLP control limits for duplicate determinations are +/- 20% Relative Percent Difference (RPD) for concentrations greater than or equal to five times the CRDL in both the original and duplicate samples, and +/- the CRDL for concentrations less than five times the CRDL. The RPD is not calculated if both the original and duplicate values fall below the IDL.

A five-fold serial dilution of sample, CCN = MC0E15-1 (MC0E15L) was performed in accordance with CLP requirements for ICP analysis.

The adjusted sample concentrations were outside CLP control limits for potassium and zinc, which are flagged with an "E" on all associated Form 1, the Cover Page and Form 9.

An "E" indicates that a chemical or physical interference effect was encountered during the analysis of the flagged analyte. As a result of this interference, all values for the analyte in the same matrix must be considered to be estimated quantities.

CLP control limits for serial dilution are defined as a deviation less than or equal to 10% in the dilutionadjusted concentrations from the original values for all analyte concentrations with values greater than fifty (50) times their respective Instrument Detection Limit (IDL) in the original sample.

The laboratory manager or his designee, as verified by the following signature has authorized release of the data contained in this hard copy data package.

nR. Cite

Thomas R. Cole Data Reviewer II April 28, 2003

CompuChem

a Division of Liberty Analytical Corp. 501 Madison Avenue Cary, NC 27513

DATA REPORTING QUALIFIERS FOR INORGANICS

On Form I, under the column labeled "C" for concentration qualifier and "Q" for qualifier, each result is flagged with the specific data reporting qualifiers listed below, as appropriate. Up to five qualifiers may be reported on Form 1 for each analyte

The C (concentration) qualifiers used are:

- U: This flag indicates the analyte was analyzed for but not detected. This reported value was obtained to malreading that was less than the Instrument Detection Limit (IDL). The IDL will be adjusted to reflect any dilution and, for soils, the percent moisture.
- B: This flag indicates the analyte was analyzed for and the reported value was obtained from a reading that was less than the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL)

The Q qualifiers used are:

- E: This flag indicates an estimated value. This flag is used:
 - When the serial dilution (a five fold dilution for CLP and a five fold dilution for SW-846 method 5010B) results are not within 10%. The analyte concentration must be sufficiently high (minimally a factor of 50N above the IDL in the original sample).
- N: This flag indicates the sample spike recovery is outside of control limits:
- *: This flag is used for duplicate analysis when the sample and the sample duplicate results are not within control limits.

The extensions: D, S, SD, L. A, added to the end of the client ID represent as follows:

- D: matrix duplicate
- S: matrix spike
- SD: matrix spike duplicate
- L: serial dilution
- A post digestion spike

Method Codes:

- P: ICP PLASMA
- CV: MERCURY COLD VAPOR AA
- CA: MIDI-DISTILLATION SPECTROPHOTOMETRIC

U. S. EPA - CLP **COVER PAGE - INORGANIC ANALYSES DATA PACKAGE**

Lab	Name:	COMPUCHEM		Contract: <u>68W00082</u>	
Lab	Code:	LIBRTY	Case No.: 31620	SAS No.:	SDG No.: MCOE17
SOW	No.:	ILM 04.1			
		EPA :	Sample No.	Lab Sample ID.	
	MC0E17		17	MC0E17-1	

MC0E17D	WG24134-2
MC0E17S	WG24134-1

Were ICP interelement corrections applied?	Yes/No	YES
Were ICP background corrections applied? If yes-were raw data generated before	Yes/No	YES
application of background corrections?	Yes/No	NO

Comments:

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

signature: <u>May 9, 2003</u>

Name:

Title:

Thomas R. Cole Linta Reviewer II AR115153 _____.

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COVER PAGE - IN

CompuChem

a Division of Liberty Analytical Corp. 501 Madison Avenue Cary, NC 27513

SDG NARRATIVE CASE # 31620 SDG # MC0E17 CONTRACT # 68W00082

The indicated Sample Delivery Group (SDG) consisting of one (1) soil samples was received into the laboratory information management system (LIMS) on April 30, 2003; intact and in good condition with Chains of Custody (COC) Records in order, unless otherwise noted in any attachments or Quality Assurance Notices. Sample ID's reported in this data package are noted by the receiving department on the COC if they differ from those listed by the samplers on the COC.

The sample was analyzed, in accordance with EPA - CLP Statement of Work (SOW) document ILM04.1 for CLP TAL total metals.

The correlation coefficient for the mercury analytical run is confirmed to be ≥ 0.9950

The cooler temperature bottle was present with samples received on April 30, 2003; and sample temperature was 6.0 degrees Celsius.

EQUATIONS FOR SOLID SAMPLE CALCULATIONS:

Client sample MC0E17 is used for illustration.

Any sample result that is < the instrument detection limit (IDL) will be entered at the IDL for that analyte.

ICP Equation:

Equation for obtaining metals sample results in mg/Kg as presented on FORM I data sheets from ICP instrument acquired results in μ g/L (ppb).

 $\begin{array}{rcl} C & x & D & x & V \\ Concentration (\% solids) & (mg/Kg) & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & \\ & & & & \\ & & & & \\ & & & \\ & & & & \\ & & & \\ & & & & \\ & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & &$

UUL 5

Mercury Equation:

Equation for obtaining mercury sample results in mg/Kg as presented on FORM I data sheets from instrument acquired results in μ g/L (ppb).

SAMPLE IDs:

The following customer IDs are associated with this SDG:

MC0E17

INSTRUMENTAL QUALITY CONTROL:

All calibration verification solutions (ICV & CCV), blanks (ICB, & CCB), and interference check samples (ICSA & ICSAB) associated with this data were confirmed to be within EPA CLP allowable limits.

SAMPLE PREPARATION QUALITY CONTROL:

The sample preparation procedure verifications (LCSS & PBS) were found to be within acceptable ranges and all field samples were prepared and analyzed within the contract specified holding times.

MATRIX RELATED QUALITY CONTROL:

The sample matrix spike, CCN = WG24134-1 (MC0E17S) was found to be outside CLP control limits for antimony, arsenic, manganese, selenium, and mercury. The reported concentrations for these analytes are flagged with an "N" on all associated Form 1 and on Form 5a.

An "N" indicates a matrix-related interference in the sample preparation procedure &/or analysis for the flagged analyte. This is normally the consequence of a relatively high anionic content in the sample or (for some sediments) an inconsistent sample matrix relative to that analyte.

CLP control limits for matrix spike recoveries are set at 75% to 125% of the analyte quantity added unless original sample concentrations exceed the true values of these "spikes" by a factor of four or more. In this case, affected analytes are not flagged even if recoveries are outside percentage recovery control limits.

Post-digestion spikes are mandatory for analytes demonstrating unsatisfactory matrix spike recoveries during ICP analysis (excluding silver). The results of such spikes are presented on Form 5b.



Unsatisfactory recovery of post-digestion spikes of this type do not have bearing upon the aforementioned "N" flags, but may indicate interference during analysis &/or a solution matrix which is hostile to the analyte in question.

Satisfactory recovery of an analyte in a post-digestion spike of this type implies interference by the required preparation procedure or in the sample matrix itself. Lack of uniformity for an analyte in sediments will also result in satisfactory recovery of post-digestion spikes after failure in the related matrix spike.

The sample matrix duplicate, CCN = WG24134-2 (MC0E17D) was outside CLP control limits for aluminum and lead. The reported concentrations for these analytes are flagged with a "*" on all associated Form 1 and on Form 6.

A "*" indicates a non-homogeneous sample matrix in regard to the flagged analyte. This is normally the consequence of a relatively coarse texture or of a mixed-matrix in sediment samples.

CLP control limits for duplicate determinations are +/- 20% Relative Percent Difference (RPD) for concentrations greater than or equal to five times the CRDL in both the original and duplicate samples, and +/- the CRDL for concentrations less than five times the CRDL. The RPD is not calculated if both the original and duplicate values fall below the IDL.

A five-fold serial dilution of sample, CCN = MC0E17-1 (MC0E17L) was performed in accordance with CLP requirements for ICP analysis.

The adjusted sample concentrations were inside CLP control limits for the requested analytes.

CLP control limits for serial dilution are defined as a deviation less than or equal to 10% in the dilutionadjusted concentrations from the original values for all analyte concentrations with values greater than fifty (50) times their respective Instrument Detection Limit (IDL) in the original sample.

The laboratory manager or his designee, as verified by the following signature has authorized release of the data contained in this hard copy data package.

Thomas R. Cole Data Reviewer II May 9, 2003



CompuChem

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DATA REPORTING QUALIFIERS FOR INORGANICS

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- **B:** This flag indicates the analyte was analyzed for and the reported value was obtained from a reading that was less than the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL)

The Q qualifiers used are:

- E: This flag indicates an estimated value. This flag is used
 - 1. When the serial dilution (a five fold dilution for CLP and a five fold dilution for SW-846 method 6010B) results are not within 10%. The analyte concentration must be sufficiently high (minimally a factor of 50N above the IDE in the original sample).
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The extensions: D, S, SD, L. A, added to the end of the client ID represent as follows:

- D: matrix duplicate
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Method Codes:

- P: ICP PLASMA
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