Appendix FData Validation Reports – October through December2012



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May 8, 2013

Mr. Christopher Ralston Program Administrator Oil Control Program Maryland Department of the Environment 1800 Washington Boulevard, Suite 620 Baltimore, Maryland 21230-1719

RE: Laboratory Data Validation **Monrovia BP/Former Green Valley Citgo** 11791 Fingerboard Road Monrovia, Frederick County, MD 21770 MDE-OCP Case No. **2005-0834FR** Remedial Management Services Contract CGS Project No. CG-12-0788.05

Dear Mr. Ralston:

Chesapeake GeoSciences, Inc. (CGS) is pleased to provide you with the attached Data Validation Report for the Monrovia BP/Former Green Valley Citgo site in Monrovia, MD. The samples that were validated include water samples collected from domestic supply wells at residences near the former Green Valley Citgo from October through December 2012 and on March 12, 2013. The samples were analyzed by Enviro-Chem Laboratories, Inc. for total and dissolved chromium and lead, and for hexavalent chromium (chromate).

CGS contracted Laboratory Data Consultants, Inc. to perform the third party data validation. The analytical data for this project were validated according to review procedure IM2 guidelines for inorganics, as described in *EPA Region III Innovative Approaches to Data Validation (EPA, 1995)*. The attached table summarizes the qualified sample results, defines the qualifiers, and gives the reason for the qualifier. All instances of reported qualifiers are based on advisory/technical validation criteria and not protocol/contractual deviations (i.e. samples being analyzed outside of the allowable holding time). The qualified hexavalent chromium sample results listed were due to matrix spike and matrix spike duplicate sample percentage of recoveries being below the acceptable limit. This indicates that the results may be biased low (or that the detection/reporting limit may be higher than noted). One dissolved chromium sample result was qualified due to the laboratory (internal) duplicate sample result being greater than the allowable difference between the sample and duplicate results.

All of the validated laboratory data were determined to be usable for their intended purpose. The minor outliers identified above did not result in any data being rejected. The data fall within the limits of precision and accuracy prescribed in each analytical method and the EPA Region III Guidelines.A copy of the data validation report is attached.

Please contact me at (410) 740-1911 (x102) or via electronic mail at <u>sdaniel@cgs.us.com</u> if you have any questions regarding this submittal or the project itself. Thank you.

Sincerely,

Chesapeake GeoSciences, Inc.

n

Sean P. Daniel Operations Manager

Attachments: Data Validation Results Summary Laboratory Data Consultants, Data Validation Report 29503

Monrovia BP/Former Green Valley Citgo MDE Case No. **2005-0834FR** Data Validation Results Summary – Qualified Results Only

Total & Dissolved Lead & Chromium (EPA 200.8) and Hexavalent Chromium/Chromate (EPA 218.7)

Sample ID	Flagged Analysis Reported Concentration (ug/L)	Flagged Analysis	Validation Qualifiers	Reason for Qualifier
3997Farm-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3997Farm-Flushed	0.152	Method 218.7 Hexavalent Chromium	L/A	1
3740Blueberry-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3995Farm-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3995Farm-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
11712Serene-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
11712Serene-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3996Farm-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3996Farm-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
11712Serene-FlushedDB	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3993Farm-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3993Farm-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3998Rye-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3998Rye-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3998Rye-FlushedDB	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3985Farm-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	2
3985Farm-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	2
3933Rosewood-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	2
3933Rosewood-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	2
11894Barley-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	2
11894Barley-Flushed	0.026	Method 218.7 Hexavalent Chromium	L/A	2
3991Farm-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	2
3991Farm-Flushed	0.023	Method 218.7 Hexavalent Chromium	L/A	2
57711 arm-1 fushed	6.3	Method 200.8 Dissolved Chromium	J/A	3
3833Greenridge-FirstDraw	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	4
3833Greenridge-Flushed	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	4

Table Notes:

J - Indicates an estimated value.

L - Indicates the reported value may be biased low.

UL - Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher. **A** -Indicates the finding is based upon technical validation criteria.

U - Analyte Not Detected Above Specified Reporting Limit

Bold - Detected analyte concentration

ug/L - micrograms per liter or parts per billion (ppb)

1 - Matrix spike/Matrix spike duplicate (Percents of recovery (%R) of 68.6%/70.5% are below acceptable limit of 85%)

2 - Matrix spike/Matrix spike duplicate (%R of 71%/69.3% are below the acceptable limit of 85%)

3 - Laboratory duplicate sample analysis result (difference of 2.0 mg/L is greater than 1.0 mg/L acceptable limit)

4 - Matrix spike/Matrix spike duplicate (%R of 54.4%/57.6% are below the acceptable limit of 85%)



Laboratory Data Consultants, Inc.

7750 El Camino Real, Ste. 2L Carlsbad, CA 92009

Phone 760.634.0437 Web www.lab-data.com Fax 760.634.0439

Chesapeake GeoScience, Inc. 5405 Twin Knolls Rd, Suite 1 Columbia, MD 21045 ATTN: Mr. Sean P. Daniel

April 26, 2013

SUBJECT: Green Valley Citgo Project, Data Validation

Dear Mr. Daniel,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on April 10, 2013. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 29503:

SDG

Fraction

Metals

ECL026876/ECL026908/ECL026926/ECL026927 ECL026945/ECL026962/ECL026972/ECL026973/ECL026987 ECL027052/ECL027072/ECL027120/ECL027121 ECL027154/ECL027163 ECL027334 ECL028223

The data validation was performed under EPA Region III, Level IM2. The analyses were validated using the following documents, as applicable to each method:

EPA Region III Innovative Approaches for Data Validation, EPA June 1995

Please feel free to contact us if you have any questions.

Sincerely,

Pei Gena **Project Manager/Senior Chemist**

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LDC	SDG#	DATE REC'D	(3) DATE DUE	Cr, (200	Pb).8)	Di: Cr, (200	ss Pb).8)	Dis Cr ^v (218	ss VI 1.7)					<u>,</u>																							
Matrix	: Water/Soil-Prod	uct		W	s	W	S	W	S	W	S	w	s	w	s	W	s	w	s	w	s	w	S.	w	S	W	S	w	s	w	s	W	S.	W	<u>s</u>	W	s
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С	ECL027052/ ECL027072/ ECL027120/ ECL027121	04/10/13	05/01/13	15	0	15	0	15	0																												
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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	October 16 through October 19, 2012
LDC Report Date:	April 30, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL026876/ECL026908/ECL026926/ECL026927

Sample Identification

11711 Serene-First Draw 11711 Serene-Flushed 3923 Rosewood-First Draw 3923 Rosewood-Flushed 4016 Middleton-First Draw 4016 Middleton-Flush 3989 Farm-First Draw 3989 Farm-Flushed 3989 Farm-FB 11711 Serene-First DrawF 11711 Serene-FlushedF 3923 Rosewood-First DrawF 3923 Rosewood-FlushedF 4016 Middleton-First DrawF 4016 Middleton-FlushF 3989 Farm-First DrawF 3989 Farm-FlushedF 3989 Farm-FBF 3989 Farm-FBFDUP 3989 Farm-FBFMS

Samples appended with "F" were analyzed as dissolved

Introduction

This data review covers 20 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 200.8 for Chromium and Lead.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B. Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Calibration

The initial and continuing calibrations were performed at the required frequency.

The calibration standards criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No chromium or lead contaminants were found in the initial, continuing and preparation blanks.

Sample 3989 Farm-FB and 3989 Farm-FBF was identified as field blanks. No chromium or lead contaminants were found.

V. ICP Interference Check Sample (ICS) Analysis

ICP interference check sample (ICS) analysis was not required by the method.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

IX. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

XII. Sample Result Verification

All sample result verifications were acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Green Valley Citgo Project

Chromium & Lead - Data Qualification Summary - SDG ECL026876/ECL026908/ECL026926/ECL026927

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL026876/ECL026908/ECL026926/ECL026927

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL026876/ECL026908/ECL026926/ECL026927

No Sample Data Qualified in this SDG

LDC #: 29503A4

VALIDATION COMPLETENESS WORKSHEET

SDG #: <u>ECL026876/ECL026908/ECL026926/ECL02692@vel IV</u> Laboratory: <u>Enviro-Chem Laboratories</u>, Inc. <u>ECL026927</u> Date: <u>4-18-</u>13 Page: <u>1 of 1</u> Reviewer: <u>MG</u> 2nd Reviewer: <u>____</u>

METHOD: Chromium & Lead (EPA Method 200.8)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
Ι.	Technical holding times	A	Sampling dates: 10-16-12 through 10-19-12
١١.	ICP/MS Tune	A	0
(.	Calibration	A	
iV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	2	not required
VI.	Matrix Spike Analysis	A	MS
VII.	Duplicate Sample Analysis	A	DUP
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
Х.	Furnace Atomic Absorption QC	2	not utilized
XI.	ICP Serial Dilution	N	not performed
XII.	Sample Result Verification	A	•
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	N	
xv	Field Blanks	ND	FB=9,16

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

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11	11711 Serene-First Draw	11 I	3923 Rosewood-First DrawF	21	31
21	11711 Serene-Flushed	12 l	3923 Rosewood-FlushedF	22	32
3 l	3923 Rosewood-First Draw	132	4016 Middleton-First DrawF	23	33
4 l	3923 Rosewood-Flushed	14 2	3989 Farm-First DrawF	24	34
5 2	4016 Middleton-First Draw	152	3989 Farm-FlushedF	_25	35
6 2	4016 Middleton-Flush	16 2	3989 Fam-FBF	26	36
7 2	3989 Farm-First Draw	17 2	3989 Farm-FBDUP	27	37
8 2	3989 Farm-Flushed	18 2	#16 MS	28	38
, 1	3989 Farm-FB	₁₉ I	11711 Serene-Flushed F	291 PBWI	39
10 l	11711 Serene-First DrawF	20 J	4016 Middleton-Flush	F307 PBW2	40

Notes: Samples appended with "F" were analyzed as dissolved

VALIDATION FINDINGS CHECKLIST

	Page:_	Lof 2
	Reviewer:	MG
2nd	Reviewer:	-v-

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Method:Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	, <u> </u>			
All technical holding times were met.				
Cooler temperature criteria was met.	\checkmark			
II. ICP/MS Tune				
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	7			
Were %RSD of isotopes in the tuning solution ≤5%?	J			
III. Calibration				
Were all instruments calibrated daily, each set-up time?	. 🗸			
Were the proper number of standards used?	\checkmark			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?	/			
Were all initial calibration correlation coefficients > 0.995?	/			
IV. Blanks				-
Was a method blank associated with every sample in this SDG?	\checkmark			· · · · · · · · · · · · · · · · · · ·
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		1		· ·
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?		\checkmark		
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/			
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were \leq 5X the RL, including when only one of the duplicate sample values were \leq 5X the RL.	/			
VII. Laboratory control samples				
Was an LCS anaylzed for this SDG?				·
Was an LCS analyzed per extraction batch?	~			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	~			

LDC #: 29503A4

VALIDATION FINDINGS CHECKLIST



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Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			\checkmark	
Do all applicable analysies have duplicate injections? (Level IV only)				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?				
IX. ICP Serial Dilution		4. °		
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		/		
Were all percent differences (%Ds) < 10%?			\checkmark	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/	
X, Internal Standards (EPA SW 846 Method 6020/EPA 200.8)				
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/			
If the %Rs were outside the criteria, was a reanalysis performed?			~	
XI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		\checkmark		
Were the performance evaluation (PE) samples within the acceptance limits?			~	
XII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	\checkmark			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		
Target analytes were detected in the field duplicates.			/	
XV. Field blanks		• • • •		
Field blanks were identified in this SDG.	\checkmark			
Target analytes were detected in the field blanks.		<u> </u>		

LDC #: 29503A4

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VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: ____of ____ Reviewer: ______ 2nd reviewer: ______ Ĭ.

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All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
19,20	w	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb)Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN [*] ,
QC 17,18	\mathbf{b}	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN ⁻ ,
•		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN [*] ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
· · ·		Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
ICP-MS	W	Al, Sb, As, Ba, Be, Cd, Ca Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN [*] ,
GEAA		ALSE As Ba Be Cd Ca Cr Co Cu Fe Pb Mg Mn Hg Ni K Se Ag Na TLV Zn Mo B Si CN

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Comments: Mercury by CVAA if performed

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VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: I of I Reviewer: MG 2nd Reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

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An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source Where, %R = <u>Found</u> x 100 True

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
1340 TCV	(CP/MS (Initial calibration)	Cr	103.80	001	103.80	103.80	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
1601 CKS	ICP/MS (Continuing calibration)	٩٩	201.200	900	100.60	100.60	~
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

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Percent recoveries	(%R) for an ICP interferer	nce check	: sample, a laboratory con	irol sample and a mat	ix spike sample were	recalculated using th	e following formula:
%R = <u>Found</u> x 100 True	Where, Found = Conc True	centration of Found = Conce	each analyte <u>measured</u> in the a = SSR (spiked sample result) ntration of each analyte in the sc	nalysis of the sample. For t SR (sample result). มเrce.	ne matrix spike calculation,		
A sample and dupli	cate relative percent differ	rence (RF	D) was recalculated using	the following formula			
RPD = <u>[S-D]</u> × 100 (S+D)/2	Where, S=(D=L	Original sam Duplicate sa	tple concentration imple concentration				
An ICP serial dilutio	n percent difference (%D) was rec	alculated using the followi	ng formula:			
%D = <u> -SDR </u> x 100 	Where, I = Ir. SDR = Serial C	nitial Sample Dilution Resu	s Result (mg/L) ult (mg/L) (Instrument Reading x	5)			
					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R / RPD / %D	%R / RPD / %D	Acceptable (Y/N)
1	ICP interference check	١	I		1	1	1
LCS 4459	Laboratory control sample	Pه	51.750 (mg/)	20 (mg)	103.5	103.5	\ \ \
1627 18	Matrix spike	٢	(1/bm) 068.05	20 (mg)	101.6	9.001	
he91 / 1091	Duplicate	د ن	< 1.0 (mg/L)	16m) 0.1 ×	0	NC	>

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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ICP serial dilution

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VALIDATION FINDINGS WORKSHEET **Sample Calculation Verification**

Page:_	of
Reviewer:	MG_
2nd reviewer:	-1

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METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>ON N/A</u> <u>ON N/A</u> Have results been reported and calculated correctly? Are results within the calibrated range of the instruments and within the linear range of the ICP? ON N/A Are all detection limits below the CRDL? 井1, Ph Detected analyte results for _____ were recalculated and verified using the following equation: Recalculation: Concentration = (RD)(FV)(Dil) (In. Vol.) (3.601 Mg/L) (0.050 L) Raw data concentration RD = = 3.601 mg/, Final volume (ml) FV = Initial volume (ml) or weight (G) In, Vol. = 0.050 L Dil = Dilution factor Calculated Reported Concentration Concentration Acceptable (Mg/L) (\dot{Y}/N) Analyte # Sample ID 3.6 3.6 96 4.6 Pb 2 П 4.6 , ~ • Note:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	October 16 through October 19, 2012
LDC Report Date:	May 1, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.

Sample Delivery Group (SDG): ECL026876/ECL026908/ECL026926/ECL026927

Sample Identification

11711 Serene-First Draw 11711 Serene-Flushed 3923 Rosewood-First Draw 3923 Rosewood-Flushed 4016 Middleton-First Draw 4016 Middleton-Flush 3989 Farm-First Draw 3989 Farm-Flushed 3989 Farm-FB 11711 Serene-First DrawMS 11711 Serene-First DrawMSD

Introduction

This data review covers 11 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 218.7 for Dissolved Chromate as Chromium.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. Initial Calibration

All criteria for the initial calibration were met.

III. Calibration verification

Calibration verification frequency and analysis criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No dissolved chromate as chromium was found in the initial, continuing and preparation blanks.

Sample 3989 Farm-FB was identified as a field blank. No dissolved chromate as chromium was found.

V. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits.

VI. Duplicates

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VII. Laboratory Control Samples

Laboratory control sample (LCS) analysis was not required by the method.

VIII. Sample Result Verification

All sample result verifications were acceptable.

IX. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

X. Field Duplicates

No field duplicates were identified in this SDG.

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Green Valley Citgo Project

Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL026876/ECL026908/ECL026926/ECL026927

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL026876/ECL026908/ECL026926/ECL026927

No Sample Data Qualified in this SDG

Green Valley Citgo Project Dissolved Chromate as Chromium - Field Blank Data Qualification Summary -SDG ECL026876/ECL026908/ECL026926/ECL026927

No Sample Data Qualified in this SDG

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VALIDATION COMPLETENESS WORKSHEET

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SDG #: ECL026876/ECL026908/ECL026926/ECL02692tevel IV Laboratory: Enviro-Chem Laboratories, Inc. ECL026927

Date: 4-18-13
Page:of
Reviewer: MG
2nd Reviewer:
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Dissolved Chromate as Cr

METHOD: Chromate (EPA Method 218.7)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 10-16-12 through 10-19-12
11	Initial calibration	A	V
111.	Calibration verification	A	
IV	Blanks	A	
v	Matrix Spike/Matrix Spike Duplicates	A	MS/MSD
VI.	Duplicates	N	
VII.	Laboratory control samples	N	not: required
VIII.	Sample result verification	A	
IX.	Overall assessment of data	A	
Х.	Field duplicates	N	
Xi	Field blanks	ND	FB=7

Note: A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

	all worldt				
1	11711 Serene-First Draw	11	11711 Serene-Flushed	21	31
2 .	3923 Rosewood-First Draw	12	4016 Middleton-Flus	122	32
3	3923 Rosewood-Flushed	13		23 .	33
4	4016 Middleton-First Draw	14		24	34
5	3989 Farm-First Draw	15		25	35
6	3989 Farm-Flushed	16		26	36
7	3989 Farm-FB	17		27	37
8	11711 Serene-First DrawMS	18		28	38
9	11711 Serene-First DrawMSD	19		29	39
10	-3989 Farm FBMS	20		30	40

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Notes:

LDC #: 29503A6

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2 Reviewer: <u>MG</u> 2nd Reviewer:

Method:Inorganics (EPA Method 218.7)				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	1			
Cooler temperature criteria was met.	\checkmark			
II. Calibration				
Were all instruments calibrated dally, each set-up time?	/			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients > 0.995?	/			•
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/			
Were titrant checks performed as required? (Level IV only)			\checkmark	
Were balance checks performed as required? (Level IV only)			\checkmark	
III. Blanks				
Was a method blank associated with every sample in this SDG?	\checkmark			(LRBS)
Was there contamination in the method blanks? If yes, please see the Blanks		\checkmark		
W. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/			
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.	1			
V. Laboratory control samples	,			
Was an LCS anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?				
VI. Regional Quality Assurance and Quality Control	·····			······································
Were performance evaluation (PE) samples performed?		1		
Were the performance evaluation (PE) samples within the acceptance limits?				

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LDC #: 29503A6

VALIDATION FINDINGS CHECKLIST



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Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	~			
Were detection limits < RL?				
VIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.		\checkmark		
Target analytes were detected in the field duplicates.				
X. Field blanks				
Field blanks were identified in this SDG,	\checkmark			
Target analytes were detected in the field blanks.		\checkmark		

LDC # 29503	46	Initial an	VALIDATION F	INDINGS WORKS dibration Calcula	sHEET tion Verification		Page: <u>Lof L</u> Reviewer: <u>MG</u>
METHOD: Inorganics	i, Method	18.7					
The correlation coeffi	cient (r) for the c	alibration of $\underline{\mathcal{C}}$	O 4-Cr was recald	culated. Calibration da	te: 10-9-12	1	
An initial or continuinç	g calibration verifi	ication percent rec	overy (%R) was rec	alculated for each type	e of analysis using th	le following formula:	
%R = <u>Found</u> x 100 True	Where, Found True =	= concentration of eau = concentration of eacl	ch analyte <u>measured</u> in th h analyte in the ICV or CC	e analysis of the ICV or CC X source	V solution		
					Recalculated	Reported	
Type of Analysis	Analyte	Standard ID	しじゅC Found (units)	True (units)	r or %R	r or %R	Acceptable (Y/N)
Initial calibration		Blank	1	1			
<u>.</u>		Standard 1	1/8m) E0.0	0.0541			
		Standard 2	0.05 (1)	0.1069			
		Standard 3	0.10 ()	0.2209		, , , , , , , , , , , , , , , , , , ,	
	Cr 04	Standard 4	0.25 ()	0.5474	r 7=0.9999	8665.0= 1	>
	as	Standard 5	0.50 ()	1.1031			
	ڑ د	Standard 6	() ()	2.1934			
		Standard 7	5.0 ()	11.1459			
Calibration verification	Croy-Cr	CCC - LOW	0.0166 (mg/1)	(7°m) 20.0	83	83	
Calibration verification	1		ł		ł	1	ł
Calibration verification	1)))			
Comments: Refer to recalculate.	Calibration Verifi	cation findings wo	rksheet for list of qual	lifications and associat	ed samples when re	ported results do not a	gree within 10.0% of the
					•		
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LDC #. 39503A6

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

৻৸ Page: 1 of 2nd Reviewer: Reviewer.

> 7.812 METHOD: Inorganics, Method

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

Found = concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). True = concentration of each analyte in the source. Where, %R = Found x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

Original sample concentration ະ 11 ແ 11 ແ Where, $RPD = |S-D| \times 100$ (S+D)/2

Duplicate sample concentration

			1		
	Acceptable (Y/N)			-	->
Reported	%R / RPD		4.96		3.7
Recalculated	%R / RPD		98.4		3.7
<u>,,,,,,,,,,,,,,,,,,,,,,,,</u>	True / D (units))	(7/Bm) 1		1.0538 (mg/L)
	Found / S (units)	1	(ssr-sr) (a. 9844 (mg/L)		1.0158 (mg/r)
	Element	١	Cr04-Cr		Cr Oy - Cr
	Type of Analysis	Laboratory control sample	Matrix spike sample	Duplicate sample	
	Sample ID)	8	ı	6/3

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC #	29503A6 VALIDATION FINDINGS WORKSHEET Page: Sample Calculation Verification Reviewer: 2nd reviewer:					
METHOD: Inorganics, Method						
Please N Comp recalc Concer y: when	e see qualifications below <u>N/A</u> Have results by <u>N/A</u> Are results with <u>N/A</u> Are all detection ound (analyte) results for ulated and verified using tration = $m \times + b$ m = 2.2173 b = 0.0040	w for all questions answered "N". Not app been reported and calculated correctly? thin the calibrated range of the instrument on limits below the CRQL? or <u># 1 Chromate</u> the following equation: 0.158 = 2.2173 0.069 = X M	licable questions are $C \sim repondent (x) + 0.00 g/L$	e identified as "N/. orted with a positiv 4 O	4". /e detect were	
#	Sample ID	Analyte	Reported Concentration	Calculated Concentration (Mg (L)	Acceptable (Y/N)	
		Chromate - Cr	0.069	0.069	Y	
	·					
 						
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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	October 23 through October 25, 2012
LDC Report Date:	April 25, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL026945/ECL026962/ECL026972/ECL026973/ ECL026987

Sample Identification

3997 Farm-First Draw	3995 Farm-First DrawF
3997 Farm-Flushed	3995 Farm-FlushedF
3740 Blueberry-First Draw	3993 Farm-First DrawF
3740 Blueberry-Flushed	3993 Farm-FlushedF
3995 Farm-First Draw	11712 Serene - First DrawF
3995 Farm-Flushed	11712 Serene - FlushedF
3993 Farm-First Draw	3996 Farm - First DrawF
3993 Farm-Flushed	3996 Farm - FlushedF
11712 Serene - First Draw	11712 Serene - Flushed DBF
11712 Serene – Flushed	3998 Rye - First DrawF
3996 Farm - First Draw	3998 Rye - FlushedF
3996 Farm - Flushed	3998 Rye - Flushed DBF
11712 Serene - Flushed DB	3997 Farm-First DrawMS
3998 Rye - First Draw	3997 Farm-First DrawDUP
3998 Rye - Flushed	3996 Farm - First DrawDUP
3998 Rye - Flushed DB	11712 Serene - FlushedFDUP
3997 Farm-First DrawF	11712 Serene - FlushedFMS
3997 Farm-FlushedF	3998 Rye - Flushed DBFMS
3740 Blueberry-First DrawF	3998 Rye - Flushed DBFDUP
3740 Blueberry-FlushedF	3997 Farm-First DrawMS

Samples appended with "F" were analyzed as dissolved

Introduction

This data review covers 40 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 200.8 for Chromium and Lead.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Calibration

The initial and continuing calibrations were performed at the required frequency.

The calibration standards criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No chromium or lead contaminants were found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. ICP Interference Check Sample (ICS) Analysis

ICP interference check sample (ICS) analysis was not required by the method.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

IX. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

XII. Sample Result Verification

All sample result verifications were acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

Samples 11712 Serene – Flushed and 11712 Serene - Flushed DB, samples 3998 Rye – Flushed and 3998 Rye - Flushed DB, samples 11712 Serene – FlushedF and 11712 Serene - Flushed DBF, and samples 3998 Rye – FlushedF and 3998 Rye - Flushed DBF were identified as field duplicates. No chromium or lead was detected in any of the samples with the following exceptions:

	Concer	Concentration (ug/L)			
Analyte	11712 Serene - Flushed	11712 Serene - Flushed DB	RPD		
Lead	323	129	86		

	Concentration (ug/L)					
Analyte	11712 Serene - FlushedF	11712 Serene - Flushed DBF	RPD			
Lead	76.1	80.4	5			

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDG ECL026945/ECL026962/ECL026972/ECL026973/ECL026987

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL026945/ECL026962/ECL026972/ECL026973/ECL026987

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL026945/ECL026962/ECL026972/ECL026973/ECL026987

No Sample Data Qualified in this SDG

LDC #: 29503B4

VALIDATION	COMPLETENESS	WORKSHEET

SDG #: ECL026945/ECL026962/ECL026972/ECL026973/ECL026987 Laboratory: Enviro-Chem Laboratories, Inc. Level IV



METHOD: Chromium & Lead (EPA Method 200.8)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
Ι.	Technical holding times	A	Sampling dates: 10-23-12 through 10-25-12
11.	ICP/MS Tune	A	l l
111.	Calibration	Ă	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	N	Not required
VI.	Matrix Spike Analysis	A	MS
VII.	Duplicate Sample Analysis	A	hul
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
Х.	Furnace Atomic Absorption QC	2	not utilized
XI.	ICP Serial Dilution	N	Not performed
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	D= 10+13 D=15+16 D= 26+29 D=31+32*
xv	Field Blanks	N	

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet

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 \neq = ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

	all water							ส
11	3997 Farm-First Draw	112	3996 Farm - First Draw	21	3995 Farm-First DrawF	₃₁ 2	3998 Rye - FlushedF	
21	3997 Farm-Flushed	122	3996 Farm - Flushed	221	3995 Farm-FlushedF	32 7	3998 Rye - Flushed DBF	
3 l	3740 Blueberry-First Draw	13 2	11712 Serene - Flushed DB	23	3993 Farm-First DrawF	₃₃ 1	3997 Farm-First DrawMS	
4 l	3740 Blueberry-Flushed	142	3998 Rye - First Draw	24	3993 Farm-FlushedF	₃₄ 1	3997 Farm-First DrawDUP	
5 l	3995 Farm-First Draw	152	3998 Rye - Flushed	25 l	11712 Serene - First DrawF	35 2	3996 Farm - First DrawDUP	
6 l	3995 Farm-Flushed	16 7	3998 Rye - Flushed DB	26 l	11712 Serene - FlushedF	36	11712 Serene - FlushedDUP	m
71	3993 Farm-First Draw	17	3997 Farm-First DrawF	27 2	3996 Farm - First DrawF	37	F 11712 Serene - FlushedMS	
₈ 1	3993 Farm-Flushed	18	3997 Farm-FlushedF	28 2	3996 Farm - FlushedF	38 2	3998 Rye - Flushed DBMS	
91	11712 Serene - First Draw	19 I	3740 Blueberry-First DrawF	29 2	11712 Serene - Flushed DBF	39 7	3998 Rye - Flushed DBDUP	I 1
10	11712 Serene - Flushed	20 l	3740 Blueberry-FlushedF	30 2	3998 Rye - First DrawF	40 2	#11 MS	

Notes: Samples appended with "F" were analyzed as dissolved

VALIDATION FINDINGS CHECKLIST



A.

Method:Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments		
I. Technical holding times						
All technical holding times were met.	\checkmark					
Cooler temperature criteria was met.	/					
II. ICP/MS Tune						
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	/					
Were %RSD of isotopes in the tuning solution ≤5%?	/					
III. Calibration						
Were all instruments calibrated daily, each set-up time?	\checkmark					
Were the proper number of standards used?	/					
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?	\checkmark					
Were all initial calibration correlation coefficients <a> 0.995?	1					
IV. Blanks						
Was a method blank associated with every sample in this SDG?	/					
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.						
V. ICP Interference Check Sample						
Were ICP interference check samples performed daily?		\checkmark				
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				ł		
VI. Matrix spike/Matrix spike duplicates			·			
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	1					
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/					
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were \leq 5X the RL, including when only one of the duplicate sample values were \leq 5X the RL.	/					
VII. Laboratory control samples						
Was an LCS anaylzed for this SDG?	1					
Was an LCS analyzed per extraction batch?						
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	<					
LDC #: 29503B4

VALIDATION FINDINGS CHECKLIST

Page: 2 of	2
Reviewer: M	G
2nd Reviewer:	\leq

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Validation Area	Yes	No	NA	Findings/Comments	
VIII. Furnace Atomic Absorption QC					
If MSA was performed, was the correlation coefficients > 0.995?			\checkmark		
Do all applicable analysies have duplicate injections? (Level IV only)					
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)					
Were analytical spike recoveries within the 85-115% QC limits?			\checkmark		
IX. ICP Serial Dilution					
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		/			
Were all percent differences (%Ds) < 10%?			\checkmark		
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			1		
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)			r		
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/				
If the %Rs were outside the criteria, was a reanalysis performed?					
XI. Regional Quality Assurance and Quality Control					
Were performance evaluation (PE) samples performed?		/			
Were the performance evaluation (PE) samples within the acceptance limits?).				
XII. Sample Result Verification		r	1		
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/				
XIII. Overall assessment of data					
Overall assessment of data was found to be acceptable.	1				
XIV. Field duplicates				1	
Field duplicate pairs were identified in this SDG.			<u> </u>		
Target analytes were detected in the field duplicates.	/				
XV. Field blanks					
Field blanks were identified in this SDG.		1			
Target analytes were detected in the field blanks.		1			

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VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1->32	\sim	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
QC 33→4	> ↓	Al, Sb, As, Ba, Be, Cd, Ca Cr, Co, Cu, Fe, Pb Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
ICP-MS	W	Al, Sb, As, Ba, Be, Cd, Ca, Cr)Co, Cu, Fe, DMg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
GEAA		ALSD AS Ba Be Cd Ca Cr Co Cu Fe Pb Mg Mn Hg Ni K Se Ag Na TL V Zn Mo B Si CN
Comments:	Mercury	v by CVAA if performed

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VALIDATION FINDINGS WORKSHEET Field Duplicates



METHOD: Metals (EPA Method 6010B/7000)

Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentra			
Analyte	10	13	RPD	
Lead	323	129	86	

V:\FIELD DUPLICATES\FD_inorganic\29503B4.WPD

	Concentra			
Analyte	26	29		
Lead	76.1	80.4	5	

V:\FIELD DUPLICATES\FD_inorganic\29503B4.WPD

LDC # 29503 B4

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: 1 of Reviewer: 3 C

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration (in ug/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source %R = <u>Found</u> x 100 True

	Acceptable (Y/N)		\succ							
Reported	%R		107.60			99.55				
Recalculated	%R		107.60			99.55				
	True (ug/L)		001			900				
	Found (ug/L)		107.600			199.100				
	Element		٩d			Cr				
	Type of Analysis	ICP (Initial calibration)	ICP/MS (Initial calibration)	CVAA (Initial calibration)	ICP (Continuing calibration)	ICP/MS (Continuing calibration)	CVAA (Continuing calibration)	GFAA (Initial calibration)	GFAA (Continuing calibation)	
	Standard ID		H C V			L X S				

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

 Where,
 Found =
 Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,
 Found =
 SR (spiked sample result) - SR (sample result).
 True =
 Concentration of each analyte in the source.
 %R = Found x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

 RPD = [S-D]
 x 100
 Where, S = Original sample concentration

 (S+D)/2
 D = Duplicate sample concentration

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = [I-SDR] x 100

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

	Acceptable (Y/N)	l	>		>	l
Reported	%R / RPD / %D	1	100.3	97.3	6.1	1
Recalculated	%R / RPD / %D	l	100.3	98.0	1.9)
	True / D / SDR (units)	ł	50 (mg/L)	20 (mg/r)	74.630(mg/L)	
	Found / S / I (units)	I	50.140 (mg/1)	(SSR-SR) 49.020 (mg/)	76.070 (mg/l)	
	Element		C r	C C	٩d)
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID		1324 LCS 4474	1310 37	1305 / 1308	1

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #	<u>+ 29503</u> B4	VALIDATION FINDINGS	VALIDATION FINDINGS WORKSHEET Page: 1_of Sample Calculation Verification Reviewer: MG 2nd reviewer: V				
METH	IOD: Trace Metals (EP	A SW 846 Method 6010/6020/7000)					
Please YN YN YN	e see qualifications belo <u>N/A</u> Have results <u>N/A</u> Are results w <u>N/A</u> Are all detect	bw for all questions answered "N". Not app been reported and calculated correctly? within the calibrated range of the instrument tion limits below the CRDL?	blicable questions are	e identified as "N// ar range of the IC	A". ₽?		
Detec equati	ted analyte results for _ ion:	#10, Pb	were recalcu	lated and verified	using the following		
Concen	ntration = <u>(RD)(FV)(Dil)</u> (In. Vol.)	Recalculation:					
RD FV In. Vol. Dil	 Raw data conce Final volume (m Initial volume (m Dilution factor 	entration (323.000 $\mu g / $ 1) or weight (G) 0.05)(0.050L) 0 L	= 323.0	, pm 000		
#	Sample ID	Analyte	Reported Concentration (Mg (L)	Calculated Concentration	Acceptable (Y/N)		
		РЬ	14.9	14.9	Ý		
		R	E 0				
2	2	Cr Ph	2.7	5.0			
3	10	Рь	323	323			
4	29	РЬ	80.4	<i>8</i> 0.५			
Note:			<u> </u>				

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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	October 23 through October 25, 2012
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.

Sample Delivery Group (SDG): ECL026945/ECL026962/ECL026972/ECL026987

Sample Identification

3997 Farm-First Draw 3997 Farm-Flushed 3740 Blueberry-First Draw 3740 Blueberry-Flushed 3995 Farm-First Draw 3995 Farm-Flushed 3993 Farm-First Draw 3993 Farm-Flushed 11712 Serene - First Draw 11712 Serene - Flushed 3996 Farm - First Draw 3996 Farm - Flushed 11712 Serene - Flushed DB 3998 Rye - First Draw 3998 Rye - Flushed 3998 Rye - Flushed DB 3997 Farm-First DrawMS 3997 Farm-First DrawMSD

Introduction

This data review covers 18 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 218.7 for Dissolved Chromate as Chromium.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. Initial Calibration

All criteria for the initial calibration were met.

III. Calibration verification

Calibration verification frequency and analysis criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No dissolved chromate as chromium was found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
3997 Farm-First DrawMS/MSD (All samples in SDG ECL026945/ ECL026962/ ECL026972/ ECL026987)	Dissolved chromate as chromium	68.8 (85-115)	70.5 (85-115)	-	L (all detects) UL (all non-detects)	A

VI. Duplicates

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VII. Laboratory Control Samples

Laboratory control sample (LCS) analysis was not required by the method.

VIII. Sample Result Verification

All sample result verifications were acceptable.

IX. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

X. Field Duplicates

Samples 11712 Serene – Flushed and 11712 Serene - Flushed DB and samples 3998 Rye – Flushed and 3998 Rye - Flushed DB were identified as field duplicates. No dissolved chromate as chromium was detected in any of the samples.

Green Valley Citgo Project Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL026945/ECL026962/ECL026972/ECL026987

SDG	Sample	Analyte	Flag	A or P	Reason
ECL026945/ ECL026962/ ECL026972/ ECL026987	3997 Farm-First Draw 3997 Farm-Flushed 3740 Blueberry-First Draw 3740 Blueberry-Flushed 3995 Farm-First Draw 3993 Farm-Flushed 3993 Farm-Flushed 11712 Serene - First Draw 3996 Farm - First Draw 3996 Farm - Flushed 11712 Serene - Flushed 11712 Serene - Flushed 11712 Serene - Flushed DB 3998 Rye - Flushed 3998 Rye - Flushed DB	Dissolved chromate as chromium	L (all detects) UL (all non-detects)	A	Matrix spike/Matrix spike duplicate (%R)

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Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL026945/ECL026962/ECL026972/ECL026987

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Field Blank Data Qualification Summary - SDG ECL026945/ECL026962/ECL026972/ECL026987

No Sample Data Qualified in this SDG

LDC #: 29503B6 VALIDATION	COMPLETENESS WORKSHEET
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SDG #: ECL026945/ECL026962/ECL026972/ECL026973/ECL026987

Laboratory: Enviro-Chem Laboratories, Inc. Level IV

Date: 4-19-13 Page: ___of_ Reviewer: M 2nd Reviewer:

Dissolved Chromate as Cr

METHOD: Ghromate (EPA Method 218.7)

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The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

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	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 10-23-12 through 10-25-12
11	Initial calibration	A	
111.	Calibration verification	A	
IV	Blanks	A	
v	Matrix Spike/Matrix Spike Duplicates	รพ	MS/MSD
VI.	Duplicates	N	
VII.	Laboratory control samples	N	not required
VIII.	Sample result verification	A	
IX.	Overall assessment of data	A	
Х.	Field duplicates	ND	D = 10 + 13 $D = 15 + 16$
	Field blanks	N	

Note: A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank

EB = Equipment blank

Validated Samples:

all Water

1	3997 Farm-First Draw	11	3996 Farm - First Draw	21	31
2	3997 Farm-Flushed	12	3996 Farm - Flushed	22	32
3	3740 Blueberry-First Draw	13	11712 Serene - Flushed DB	23	33
4	3740 Blueberry-Flushed	14	3998 Rye - First Draw	24	34
5	3995 Farm-First Draw	15	3998 Rye - Flushed	25	35
6	3995 Farm-Flushed	16	3998 Rye - Flushed DB	26	36
7	3993 Farm-First Draw	17	3997 Farm-First DrawMS	27	37
8	3993 Farm-Flushed	18	3997 Farm-First DrawMSD	28	38
9	11712 Serene - First Draw	19		29	39
10	11712 Serene - Flushed	20		30	40

Notes:_

LDC #: 29503 B6

VALIDATION FINDINGS CHECKLIST

A . A

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Method:Inorganics (EPA Method <i>d</i>(5.()				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	1			
Cooler temperature criteria was met.	1			
II. Calibration				
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients > 0.995?	\checkmark			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	\checkmark			
Were titrant checks performed as required? (Level IV only)			1	
Were balance checks performed as required? (Level IV only)			1	
III. Blanks				
Was a method blank associated with every sample in this SDG?	<			LRBS
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		~		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		/		
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.				
V. Laboratory control samples				
Was an LCS anayized for this SDG?		\checkmark		
Was an LCS analyzed per extraction batch?		\checkmark		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?			\checkmark	
VI. Regional Quality Assurance and Quality Control			_	
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?			~	

VALIDATION FINDINGS CHECKLIST



Validation Area	Yes	No	NA	Findings/Comments							
VII. Sample Result Verification											
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/										
Were detection limits < RL?	\checkmark										
VIII. Overall assessment of data											
Overall assessment of data was found to be acceptable.	\checkmark										
IX. Field duplicates											
Field duplicate pairs were identified in this SDG.	\checkmark										
Target analytes were detected in the field duplicates.		/									
X. Field blanks	X. Field blanks										
Field blanks were identified in this SDG.		~									
Target analytes were detected in the field blanks.			~								

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VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

С Ч С ō Page: Reviewer: 2nd Reviewer:

J 18.7 METHOD: Inorganics, EPA Method_ Rease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Was a matrix spike analyzed for each matrix in this SDG? N N/A

Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken. AN NA Y (N/N/A

Were all duplicate sample relative percent differences (RPD) \leq 20% for water samples and \leq 35% for soil samples? -EVEL IV ONLY:

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. **WN NA**

#	UI USW/SW	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (I imits)	Associated Samples	Qualifications	
-	17/18	water	CrO4-Cr	68.8 (85-115)	70.5(85-115)		all	L /UL /A	
									Ι
									Γ
									Τ
									Τ
									T
									Τ
				-					T
┨									
Comn	nents:]

MSD.6

LDC # 29503B6

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

METHOD: Inorganics, Method 218.7

The correlation coefficient (r) for the calibration of Cr 04-Cr was recalculated. Calibration date: 10-9-12

An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Found = concentration of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source Where, %R = <u>Found</u> x 100 True

										11	1	12	7
	Acceptable (Y/N)					>	-			;	l		
Reported	r or %R					8666.0= 1				103.3	l	1	
Recalculated	r or %R					r]= 0.99999				103.3	Ì		
A	True (units)		0.0541	0.1069	0.2209	0.5474	1.1031	2.1934	11.1459	(7/Bm) 1	l	J	
, : e ,	しじょく Found (units)	1	1/8m) E0.0	0.05 (1)	0.10 ()	0.25 ()	0.50 ()	1.0 ()	5.0 ()	(1/ En) 8e20.1	1	l	
	Standard ID	Blank	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7	GIM-202	ł		
	Analyte					Cr 04	8	Č		Cr Oy-Cr))	
	Type of Analysis	Initial calibration								Calibration verification	Calibration verification	Calibration verification	

CALCLC.6

recalculated results.

Page: Lof

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18 J.C. 199

LDC # 39 503B6

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: 1 of 1 Reviewer: MG 2nd Reviewer: 1

METHOD: Inorganics, Method 2(8.7

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

Found = concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). True = concentration of each analyte in the source. Where, %R = Found x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

RPD = <u>[S-D]</u> × 100 Where, S = (S+D)/2 D =

Original sample concentration Duplicate sample concentration

-					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	%R / RPD	%R / RPD	Acceptable (Y/N)
	Laboratory control sample						
1		l	l	l		I	l
~	Matrix spike sample		(SSR-SR)				
17		Cro4-Cr	(n) [m] 2829.0	(7/bm) 1	68.8	68.89	7
	Duplicate sample						-
17/18		CrO4-Cr	0.6883 (mg/1)	0.7050 (mg/L)	2.4	2.4	>

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC #	<u>, </u>	Sample Calculation Ve	rification	Pa Revie 2nd revie	wer:(_of wer:(wer:(
METHOD: Inorganics, Method 218.7										
Please YN YN YN Comp recalc	e see qualifications belo <u>N/A</u> Have results i <u>N/A</u> Are results wi <u>N/A</u> Are all detection bound (analyte) results for culated and verified using	w for all questions answered "N". Not apple been reported and calculated correctly? thin the calibrated range of the instrument on limits below the CRQL? μ μ μ μ μ μ μ μ μ μ	plicable questions ar nts? repo	e identified as "N/ orted with a positi	'A''. ve detect we					
Concer	ntration =	Recalculation:								
mx	+ 6	0.340= 2.2173(x) + 0. 0040)						
ere	b = 0.0040	01515 491	- x							
di	l = lx									
#	Sample ID	Analyte	Reported Concentration	Calculated Concentration	Acceptable (Y/N)					
	2	Chromate - Cr	0.152	0.152	Ý					

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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	November 6 through November 9, 2012
LDC Report Date:	April 25, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL027052/ECL027072/ECL027120/ECL027121

Sample Identification

3806 Greenridge-First Draw 3806 Greenridge-Flushed 11789 Thomas Spring-First Draw 11789 Thomas Spring-Flushed 3984A Farm First Draw 3984A Farm Flushed 3990 Farm First Draw 3990 Farm Flushed 3994 Farm-First Draw 3994 Farm-Flushed 3994 Farm-Flushed DB 3992 Farm-First Draw 3992 Farm-Flushed 3987 Farm-First Draw 3987 Farm-Flushed 3806 Greenridge-First DrawF 3806 Greenridge-FlushedF 11789 Thomas Spring-First DrawF 11789 Thomas Spring-FlushedF 3984A Farm First DrawF

3984A Farm FlushedF 3990 Farm First DrawF 3990 Farm FlushedF 3994 Farm-First DrawF 3994 Farm-FlushedF 3994 Farm-Flushed DBF 3992 Farm-First DrawF 3992 Farm-FlushedF 3987 Farm-First DrawF 3987 Farm-FlushedF 3806 Greenridge-First DrawMS 3806 Greenridge-First DrawDUP 3994 Farm-Flushed DBMS 3994 Farm-Flushed DBDUP 3994 Farm-FlushedMSF 3994 Farm-FlushedDUPF

Samples appended with "F" were analyzed as dissolved

Introduction

This data review covers 36 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 200.8 for Chromium and Lead.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Calibration

The initial and continuing calibrations were performed at the required frequency.

The calibration standards criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No chromium or lead contaminants were found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. ICP Interference Check Sample (ICS) Analysis

ICP interference check sample (ICS) analysis was not required by the method.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

IX. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

XII. Sample Result Verification

All sample result verifications were acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

Samples 3994 Farm-Flushed and 3994 Farm-Flushed DB and samples 3994 Farm-FlushedF and 3994 Farm-Flushed DBF were identified as field duplicates. No chromium or lead was detected in any of the samples.

Green Valley Citgo Project

Chromium & Lead - Data Qualification Summary - SDG ECL027052/ECL027072/ECL027120/ECL027121

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL027052/ECL027072/ECL027120/ECL027121

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL027052/ECL027072/ECL027120/ECL027121

No Sample Data Qualified in this SDG

VALIDATION COMPLETENESS WORKSHEET LDC #: 29503C4 SDG #: ECL027052/ECL027072/ECL027120/ECL027121

Laboratory: Enviro-Chem Laboratories, Inc. Level IV

Date: 4-19-13 Page: | of Reviewer: MC 2nd Reviewer:

METHOD: Chromium & Lead (EPA Method 200.8)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
١.	Technical holding times	A	Sampling dates: 11-6-12 through 11-9-12
11.	ICP/MS Tune	A	
III. ⁻	Calibration	Α	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	N	not required
VI.	Matrix Spike Analysis	A	Ms
VII.	Duplicate Sample Analysis	A	DUP
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
X .	Furnace Atomic Absorption QC	N	not utilized
XI.	ICP Serial Dilution	N	not performed
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	ND	D = 10 + 11 $D = 25 + 26$
xv	Field Blanks	N	

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

Va	all water										
11	3806 Greenridge-First Draw	11 2	3994 Farm-Flushed DB	21 l	3984A Farm FlushedF	31 ¹	3806 Greenridge-First DrawMS				
21	3806 Greenridge-Flushed	ן 12 ג	3992 Farm-First Draw	22 l	3990 Farm First DrawF	32	3806 Greenridge-First DrawDUP				
31	11789 Thomas Spring-First Draw	13 2	3992 Farm-Flushed	231	3990 Farm FlushedF	33	3994 Farm-Flushed DBMS				
41	11789 Thomas Spring-Flushed	14 2	3987 Farm-First Draw	24 l	3994 Farm-First DrawF	34	3994 Farm-Flushed DBDUP				
₅ 1	3984A Farm First Draw	15 2	3987 Farm-Flushed	25 ¹	3994 Farm-FlushedF	35	3994 Farm-FlushedMSF				
61	3984A Farm Flushed	16	3806 Greenridge-First DrawF	26 2	3994 Farm-Flushed DBF	36	3994 Farm-FlushedDUPF				
71	3990 Farm First Draw	17 l	3806 Greenridge-FlushedF	27	3992 Farm-First DrawF	37					
81	3990 Farm Flushed	18 l	11789 Thomas Spring-First DrawF	28 2	3992 Farm-FlushedF	38					
9 l	3994 Farm-First Draw	19 1	11789 Thomas Spring-FlushedF	29 2	3987 Farm-First DrawF	39					
10	3994 Farm-Flushed	201	3984A Farm First DrawF	30 2	3987 Farm-FlushedF	40					
<u>.</u>		"+h "F	" were analyzed as discolved				PBWI				
N	otes: Samples appended w	nui r	were analyzed as dissolved				2 PBW2				

PBW2

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VALIDATION FINDINGS CHECKLIST

Page:___of_2 Reviewer:___<u>MG</u> 2nd Reviewer:____

Validation Area	Yes	No	NA	Findings/Comments			
I. Technical holding times							
All technical holding times were met.	\checkmark						
Cooler temperature criteria was met.	\checkmark						
II. ICP/MS Tune	r	·					
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	\checkmark						
Were %RSD of isotopes in the tuning solution ≤5%?	1						
III. Calibration							
Were all instruments calibrated daily, each set-up time?	/						
Were the proper number of standards used?	1						
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?	/						
Were all initial calibration correlation coefficients > 0.995?	/						
IV. Blanks							
Was a method blank associated with every sample in this SDG?	~						
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/					
V. ICP Interference Check Sample							
Were ICP interference check samples performed daily?		/					
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?							
VI. Matrix spike/Matrix spike duplicates							
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	~						
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/						
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were \leq 5X the RL, including when only one of the duplicate sample values were \leq 5X the RL.	/						
VII. Laboratory control samples	·····	,					
Was an LCS anaylzed for this SDG?							
Was an LCS analyzed per extraction batch?							
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?							

VALIDATION FINDINGS CHECKLIST

Page:	2 _{of}	2
Reviewer:	M	G
2nd Reviewer:	\mathcal{N}	Ζ

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC			······	
If MSA was performed, was the correlation coefficients > 0.995?			\checkmark	
Do all applicable analysies have duplicate injections? (Level IV only)			~	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)				
Were analytical spike recoveries within the 85-115% QC limits?				
IX. ICP Serial Dilution		n		
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		/		
Were all percent differences (%Ds) < 10%?			\checkmark	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.				
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)			r	
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/			
If the %Rs were outside the criteria, was a reanalysis performed?				
XI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		1		
Were the performance evaluation (PE) samples within the acceptance limits?				
XII. Sample Result Verification		r	,	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	~			
XIII. Overall assessment of data			-	
Overall assessment of data was found to be acceptable.	/		Ĺ	
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	1		ļ	·
Target analytes were detected in the field duplicates.			1	· · · · · · · · · · · · · · · · · · ·
XV. Field blanks				
Field blanks were identified in this SDG.		1		
Target analytes were detected in the field blanks.				

LDC #: 29503C4

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VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

.		
Sample ID	Matrix	
$1 \rightarrow 30$	W	Al, Sb, As, Ba, Be, Cd, Ca (Cr) Co, Cu, Fe (Pb) Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
⁶⁶ 31→36	/	Al, Sb, As, Ba, Be, Cd, Ca (Cr.)Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al. Sb. As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Pb Mg Mn Hg Ni K Se Ag Na Ti V Zn Mo B Si CN.
		Al Sb. As. Ba. Be. Cd. Ca. Cr. Co. Cu. Fe. Pb. Mg. Mn. Hg. Ni, K. Se, Ag. Na, Tl. V. Zn, Mo. B. Si. CN ⁻ .
		Al Sh As Ba Be Cd Ca Cr. Co Cu Fe Pb Ma Mn Ha Ni K Se Aa Na Ti V Zn Mo B Si CN.
		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Pb Ma Mn Ha Ni K Se Aa Na Ti V Zn Mo B Si CN
		Analysis Method
ICP		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Pb Mg Mn Hg Ni K Se Ag Na Ti V Zn Mo B Si CN
	W	Al Sh As Ba Be Cd Ca Cr Co Cu Fe PDMa Mn Ha Ni K Se Ag Na TI V Zn Mo B Si CN
		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Ma Mn Ha Ni K Se Ag Na TI V Zn Mo B Si CN

Comments: Mercury by CVAA if performed

LDC # 39503C4

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

z B Page: 1 of 1 Reviewer: 2nd Reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration (in ug/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source %R = Found x 100 True

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
10 V IC V	ICP/MS (Initial calibration)	r C	103.200	00	103.20	103.20	γ
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
1192 CKS	ICP/MS (Continuing calibration)	٩d	Jeo.000	000	100.00	100.00	>
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC # <u>29503</u>C4

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

 Where,
 Found =
 Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

 Found =
 SSR (spiked sample result) - SR (sample result).
 True =
 Concentration of each analyte in the source.
 %R = Found x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

 $RPD = \underline{|S-D|} \times 100$ Where, S = Original sample concentration (S+D)/2 D = Duplicate sample concentration

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = [I-SDR] x 100

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

	Acceptable (Y/N)	l	\succ		>	
Reported	%R / RPD / %D	١	101	99.2	Q	I
Recalculated	%R / RPD / %D)	101	99.H	0	ł
	True / D / SDR (units))	50 (mg/L)	50 (mg/)	2.318 (mg/n))
	Found / S / I (units))	50.490 (mg/L)	(SSR-SR) 49.680 (mg/L)	0.317 (mg/L)	
	Element)	გე	C.	ЪЬ	1
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID)	LCS 4501	1108 31	102 Los	

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

 $\{ r_{i}, r_{i} \}$

LDC #: 29503C4

VALIDATION FINDINGS WORKSHEET **Sample Calculation Verification**

Page:	of
Reviewer:	MG
2nd reviewer:	
	V

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METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Have results been reported and calculated correctly? ON N/AAre results within the calibrated range of the instruments and within the linear range of the ICP? ON N/A Are all detection limits below the CRDL? ON N/A

1, Pb Detected analyte results for were recalculated and verified using the following equation:

Concentration = (RD)(FV)(Dil) Recalculation:

RD FV In. Vol.	= = =	(In. Vol.) Raw data concentration Final volume (ml) Initial volume (ml) or weight (G)	(2.317 Mg/L) (0.050 L)	11	2.317	, pu
Dil	=	Dilution factor	0,050 L			

#	Sample ID	Analyte	Reported Concentration (#9.1L)	Calculated Concentration (Mg/L)	Acceptable (Y/N)
	1	Рь	2.3	2.3	Y
2	3	РЬ	2.5	2.5	
3	16	Pb	1.7	1.7	i
			· · · · · · · · · · · · · · · · · · ·	<u> </u>	
 					
				1	
<u> </u>			<u> </u>		
 					
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Note:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	November 6 through November 9, 2012
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL027052/ECL027072/ECL027120/EXL027121

Sample Identification

3806 Greenridge-First Draw 3806 Greenridge-Flushed 11789 Thomas Spring-First Draw 11789 Thomas Spring-Flushed 3984A Farm First Draw 3984A Farm Flushed 3990 Farm Flushed 3990 Farm-First Draw 3994 Farm-Flushed 3994 Farm-Flushed DB 3992 Farm-First Draw 3992 Farm-First Draw 3987 Farm-First Draw 3987 Farm-Flushed

Introduction

This data review covers 15 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 218.7 for Dissolved Chromate as Chromium.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. Initial Calibration

All criteria for the initial calibration were met.

III. Calibration verification

Calibration verification frequency and analysis criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No dissolved chromate as chromium was found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

VI. Duplicates

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VII. Laboratory Control Samples

Laboratory control sample (LCS) analysis was not required by the method.

VIII. Sample Result Verification

All sample result verifications were acceptable.

IX. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

X. Field Duplicates

Samples 3994 Farm-Flushed and 3994 Farm-Flushed DB were identified as field duplicates. No dissolved chromate as chromium was detected in any of the samples.

Green Valley Citgo Project

Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL027052/ECL027072/ECL027120/EXL027121

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL027052/ECL027072/ECL027120/EXL027121

No Sample Data Qualified in this SDG

Green Valley Citgo Project Dissolved Chromate as Chromium - Field Blank Data Qualification Summary -SDG ECL027052/ECL027072/ECL027120/EXL027121

No Sample Data Qualified in this SDG

LDC #: 29503C6 VALIDATION COMPLETENESS WORKSHEET	Date: 4-19-13
SDG #: ECL027052/ECL027072/ECL027120/ECL027121	Page: <u></u> of
Laboratory: Enviro-Chem Laboratories, Inc. Level IV	Reviewer: MG
The I Change Co	2nd Reviewer:

Dissolved Chromate as Cr

METHOD: Chromate (EPA Method 218.7)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

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	Validation Area		Comments			
I.	Technical holding times	A	Sampling dates: 11-6-12 through 11-9-12			
11	Initial calibration	A				
111.	Calibration verification	A				
IV	Blanks	A				
V	Matrix Spike/Matrix Spike Duplicates	2	client specified			
VI.	Duplicates	2	34 /1			
VII.	Laboratory control samples	N	not required			
VIII.	Sample result verification	A	V			
IX.	Overall assessment of data	A				
Х.	Field duplicates	DИ	D = 10 + 11			
	Field blanks	と				

Note: A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

1	3806 Greenridge-First Draw	11	3994 Farm-Flushed DB	21	 31	
2	3806 Greenridge-Flushed	12	3992 Farm-First Draw	22	 32	
3	11789 Thomas Spring-First Draw	13	3992 Farm-Flushed	23	33	
4	11789 Thomas Spring-Flushed	14	3987 Farm-First Draw	24	34	
5	3984A Farm First Draw	15	3987 Farm-Flushed	25	 35	
6	3984A Farm Flushed	16		26	 36	
7	3990 Farm First Draw	17		27	37	
8	3990 Farm Flushed	18		28	 38	
9	3994 Farm-First Draw	19		29	 39	
10	3994 Farm-Flushed	20		30	 40	

Notes:_
LDC #: 29503C6

VALIDATION FINDINGS CHECKLIST

Page: <u>lof</u> **2** Reviewer: <u>MG</u> 2nd Reviewer:

Method:Inorganics (EPA Method $\sigma(0, 0)$		<u></u>	1	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	1			
Cooler temperature criteria was met.	1		<u> </u>	
II. Calibration				
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients ≥ 0.995?	\checkmark			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/			
Were titrant checks performed as required? (Level IV only)				
Were balance checks performed as required? (Level IV only)				1
III. Blanks				
Was a method blank associated with every sample in this SDG?	/			LRBs
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil (Water)		/		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.			~	
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.			/	
V. Laboratory control samples				
Was an LCS anaylzed for this SDG?		/		
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?			/	
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		\checkmark		/
Were the performance evaluation (PE) samples within the acceptance limits?			\checkmark	

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VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: MG 2nd Reviewer:

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Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
Were detection limits < RL?	/			
VIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.	\checkmark			
Target analytes were detected in the field duplicates.		\checkmark		
X. Field blanks				
Field blanks were identified in this SDG.		1		
Target analytes were detected in the field blanks.			\checkmark	

LDC # 29503C6

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

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2nd Reviewer: <u>N</u>

Page: Lof

METHOD: Inorganics, Method 218.7

11-13-12

med.

1-13-13 The correlation coefficient (r) for the calibration of $\frac{cv \partial_{4} - cv}{cv \partial_{4} - cv}$ was recalculated. Calibration date: An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration of each analyte measured in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source %R = <u>Found</u> x 100 True

	Acceptable (Y/N)					~	_					1		
Reported	r or %R		r 2 = 1.0000											
Recalculated	r or %R				•	V 7= 1,0000					99.6	ł		
A 1.02	True (units)	l	0.0485	6611.0	7966.0	0.5841	1.1978	2.3853	11.9665		(7/8m) 1))	
60.07	Found (units)	1	0.02 (4g/L	0.05 ()	0.10 ()	0.25 ()	0.50 ()	1.00 ()	5.00 (J)		(18m) 0366.0)	ł	
	Standard ID	Blank	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7	5100	CCC-MID	1	l	
	Analyte			, ,	C + 0 4	2 2	J				Croy-Cr	1		
	Type of Analysis	Initial calibration								Calibration verification		Calibration verification	Calibration verification	

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC #	<u>* 2950</u> 3C6	VALIDATION FINDINGS We Sample Calculation Ver	ORKSHEET ification	Pa Revie 2nd revie	age:of wer: <u>M</u> (wer:\
METH	IOD: Inorganics, Metho	d 218.7			
Please ON ON Comp	e see qualifications bek <u>N/A</u> Have results <u>N/A</u> Are results w <u>N/A</u> Are all detec	by for all questions answered "N". Not app been reported and calculated correctly? within the calibrated range of the instrumen tion limits below the CRQL? for $\# \partial_{c}$ Chromate as Cr	licable questions an ts?repo	e identified as "N prted with a positi	/A". ve detect we
Concer		g the following equation:			
: mx	+ 6	$\partial . 173 = 2.3902(x)$	- 0.0012		
/heve - 2.3 0 (<u>- 1</u>	2 3902 . 0012 X	$0.0729 \text{ mg/}_{L} = \times$			
#	Sample ID	Analyte	Reported Concentration	Calculated Concentration	Acceptable (Y/N)
	2	CrOy-Cr	0.073	0.073	Y
2	ч	Cr04 - Cr	0.150	0.150	
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			·		
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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	November 13 through November 14, 2012
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL027154/ECL027163

Sample Identification

3985 Farm First Draw 3985 Farm Flushed 3933 Rosewood First Draw 3933 Rosewood Flushed 11894 Barley First Draw 11894 Barley Flushed 3991 Farm First Draw 3991 Farm Flushed 3985 Farm First DrawF 3985 Farm FlushedF 3933 Rosewood First DrawF 3933 Rosewood FlushedF 11894 Barley First DrawF 11894 Barley FlushedF 3991 Farm First DrawF 3991 Farm FlushedF 3985 Farm First DrawMS 3985 Farm First DrawDUP 3991 Farm FlushedFMS 3991 Farm FlushedFDUP

Samples appended with "F" were analyzed as dissolved

Introduction

This data review covers 20 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 200.8 for Chromium and Lead.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Calibration

The initial and continuing calibrations were performed at the required frequency.

The calibration standards criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No chromium or lead contaminants were found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. ICP Interference Check Sample (ICS) Analysis

ICP interference check sample (ICS) analysis was not required by the method.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Analyte	RPD (Limits)	Difference (Limits)	Flag	A or P
3991 Farm FlushedFDUP (3991 Farm FlushedF)	Chromium	-	2.0 mg/L (≤1.0)	J (all detects) UJ (all non-detects)	А

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

IX. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

XII. Sample Result Verification

All sample result verifications were acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDG ECL027154/ECL027163

SDG	Sample	Analyte	Flag	A or P	Reason
ECL027154/ ECL027163	3991 Farm FlushedF	Chromium	J (all detects) UJ (all non-detects)	A	Duplicate sample analysis (difference)

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL027154/ECL027163

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL027154/ECL027163

No Sample Data Qualified in this SDG

LDC #: VALIDATI	ON COMPLETENESS WORKSHEET	Date: <u>4-19-</u> 13
SDG #: ECL027154/ECL027163	Level IV	Page:of
Laboratory: Enviro-Chem Laboratories, Inc.		Reviewer: MG
		2nd Reviewer:

METHOD: Chromium & Lead (EPA Method 200.8)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments						
I.	Technical holding times	A	Sampling dates: 11-13-12 through 11-14-12						
11.	ICP/MS Tune	A	0						
III.	Calibration	A							
IV.	Blanks	A							
V.	ICP Interference Check Sample (ICS) Analysis	2	not required						
VI.	Matrix Spike Analysis	A	Ms						
VII.	Duplicate Sample Analysis	รพ	DUP						
VIII.	Laboratory Control Samples (LCS)	A	LCS						
IX.	Internal Standard (ICP-MS)	A							
Х.	Furnace Atomic Absorption QC	N	not utilized						
XI.	ICP Serial Dilution	ン	not performed						
XII.	Sample Result Verification	A	-						
XIII.	Overall Assessment of Data	A							
XIV.	Field Duplicates	2							
xv	Field Blanks	2							

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

1	3985 Farm First Draw	11	3933 Rosewood First DrawF	21		31	
2	3985 Farm Flushed	12	3933 Rosewood FlushedF	22		32	
3	3933 Rosewood First Draw	13	11894 Barley First DrawF	23		33	
4	3933 Rosewood Flushed	14	11894 Barley FlushedF	24		34	
5	11894 Barley First Draw	15	3991 Farm First DrawF	25		35	
6	11894 Barley Flushed	16	3991 Farm FlushedF	26		36	
7	3991 Farm First Draw	17	3985 Farm First DrawMS	27		37	
8	3991 Farm Flushed	18	3985 Farm First DrawDUP	28		38	
9	3985 Farm First DrawF	19	3991 Farm FlushedF MS	29		39	
10	3985 Farm FlushedF	20	3991 Farm FlushedFDUP	30	PBW	40	

Notes: Samples appended with "F" were analyzed as dissolved

VALIDATION FINDINGS CHECKLIST



Method:Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times			<u>.</u>	
All technical holding times were met.	\checkmark			
Cooler temperature criteria was met.	\checkmark			
II. ICP/MS Tune			. <u>.</u> .	
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	\checkmark			
Were %RSD of isotopes in the tuning solution ≤5%?	/			
III. Calibration				
Were all instruments calibrated daily, each set-up time?	\checkmark			
Were the proper number of standards used?	~			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?				
Were all initial calibration correlation coefficients ≥ 0.995?	/			
IV. Blanks				
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?		/		
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?			/	
VI. Matrix spike/Matrix spike duplicates	,			
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	~			
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were \leq 5X the RL, including when only one of the duplicate sample values were \leq 5X the RL.		/		
VII. Laboratory control samples				
Was an LCS anaylzed for this SDG?	1			
Was an LCS analyzed per extraction batch?	1			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?				

VALIDATION FINDINGS CHECKLIST

Page:_	20	of 2
Reviewer:	<u> </u>	١G
2nd Reviewer:		\swarrow

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC			·	
If MSA was performed, was the correlation coefficients > 0.995?				
Do all applicable analysies have duplicate injections? (Level IV only)			<u> </u>	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)				
Were analytical spike recoveries within the 85-115% QC limits?				
IX. ICP Serial Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		\checkmark	ļ	
Were all percent differences (%Ds) < 10%?			1	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.				
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)				
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	1			
If the %Rs were outside the criteria, was a reanalysis performed?				
XI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?				
XII. Sample Result Verification				1
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Overall assessment of data			_	
Overall assessment of data was found to be acceptable.	/		<u> </u>	
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.			<u> </u>	
Target analytes were detected in the field duplicates.		I		
XV. Field blanks	.	,		
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.				

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LDC #: 29503D4

VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:____of__ Reviewer:___ M G 2nd reviewer:

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All circled elements are applicable to each sample.

Sample ID	Matrix	
1-716	W	Al, Sb, As, Ba, Be, Cd, Ca Cr)Co, Cu, Fe, Cb Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN [*] ,
°¢ +71 → 20	1	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
	<u></u>	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
ICP-MS	W	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
GEAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mp, Hg, Ni, K, Se, Ag, Na, Tl, V, Zp, Mo, B, Si, CN ¹
ICP-MS GEAA	V	Ai, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Wg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN ⁻ , Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN ⁻ , Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN ⁻ ,

Comments: Mercury by CVAA if performed

LDC # 99503D4

VALIDATION FINDINGS WORKSHEET Duplicate Analysis

Page: Lof 2nd Reviewer. Reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N/A Was a duplicate sample analyzed for each matrix in this SDG?

Were all duplicate sample relative percent differences (RPD) < 20% for water samples and < 35% for soil samples? If no, see qualifications below. A control limit of ±R.L. (±2X R.L. for soil) was used for sample values that were <5X the R.L., including the case when only one of the duplicate sample values was <5X R.L. If field blanks were used for laboratory duplicates, note in the Overall Assessment. **ANNA**

TEVEL IV ONLY:

			 _		 		 				 	 	 	 	
	Oualifications	J/UJ/A												nt only.	
tions.	Associated Samples	* 91												o qual. pare	1
orksheet for recalculat	Difference (I imits)	(0.1 ≥) 7/8m 0.6												limit, s	-
I IV Recalculation W	RPD (l imits)									-				atch is in	
le? See Leve	Analyte	50												this bu	
sults acceptab	Matrix	water												 2C for	
Were recalculated res	Duplicate ID	90												* 2nd G	
Y'N N/A	# Date		 											 Comments:	

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LDC #. 39 50 3 DH

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: 1 of 1 Reviewer: MG

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration (in ug/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source %R = <u>Found</u> x 100 True

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
1003 ICV	ICP/MS (Initial calibration)	۹J	103.900	001	103.90	103.90	Y
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
1036 CKS	ICP/MS (Continuing calibration)	Cr	194.400	0,0 €	97.20	97.20	
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

đ Page: | 2nd Reviewer: Reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

 Where,
 Found =
 Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

 Found =
 SSR (spiked sample result) - SR (sample result).
 True =
 Concentration of each analyte in the source.
 %R = Found x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

S = Original sample concentration D = Duplicate sample concentration Where, RPD = <u>[S-D]</u> × 100 (S+D)/2

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = [I-SDR] x 100

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Resuft (mg/L) (Instrument Reading x 5)

	Acceptable (Y/N)	•	\checkmark			I
Reported	%R / RPD / %D		98.3	102.6	41.4	l
Recalculated	%R / RPD / %D	l	98.3	102.6	41.4	
	True / D / SDR (units)	l	20 (md/r)	50 (mg/r)	3.829 (mg/L)	
	Found / S / I (units)	l	(1/bm) 041.94	(SSR-SR) 51. 280 (mg/)	5.827 (mg/)	1
	Element	١	C	٩d	s U	1
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID	ł	1014 LCS 4518	193 19	1c11 / 8111	١

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 29503D4

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	MG-
2nd reviewer:	

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". (Y) N/A (Y)

Detected analyte results for <u># 4, C </u>were recalculated and verified using the following equation:

Concentration = (RD)(FV)(Dil) (in. Vol.) RD = Raw data concentration FV = Final volume (ml)

Recalculation: (7.165 mg/L)(0.050L) 0.050 L

= 7.165 Mg/L

In. Vol. = Initial volume (ml) or weight (G) Dil = Dilution factor

#	Sample ID	Analyte	Reported Concentration (#9. L)	Calculated Concentration (Mg/L)	Acceptable (Y/N)
	l	Ръ	3.3	3.3	Y
2	4	Cr	7.2	7.2	6
	· · · · · · · · · · · · · · · · · · ·				

Note:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	November 13 through November 14, 2012
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL027154/ECL027163

Sample Identification

3985 Farm First Draw 3985 Farm Flushed 3933 Rosewood First Draw 3933 Rosewood Flushed 11894 Barley First Draw 11894 Barley Flushed 3991 Farm First Draw 3991 Farm Flushed 3985 Farm First DrawMS 3985 Farm First DrawMSD

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Introduction

This data review covers 10 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 218.7 for Dissolved Chromate as Chromium.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. Initial Calibration

All criteria for the initial calibration were met.

III. Calibration verification

Calibration verification frequency and analysis criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No dissolved chromate as chromium was found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
3985 Farm First DrawMS/MSD (All samples in SDG ECL027154/ ECL027163)	Dissolved chromate as chromium	71 (85-115)	69.3 (85-115)	-	L (all detects) UL (all non-detects)	A

VI. Duplicates

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VII. Laboratory Control Samples

Laboratory control sample (LCS) analysis was not required by the method.

VIII. Sample Result Verification

All sample result verifications were acceptable.

IX. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

X. Field Duplicates

No field duplicates were identified in this SDG.

Green Valley Citgo Project Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL027154/ECL027163

SDG	Sample	Analyte	Flag	A or P	Reason
ECL027154/ ECL027163	3985 Farm First Draw 3985 Farm Flushed 3933 Rosewood First Draw 3933 Rosewood Flushed 11894 Barley First Draw 11894 Barley Flushed 3991 Farm First Draw 3991 Farm Flushed	Dissolved chromate as chromium	L (all detects) UL (all non-detects)	A	Matrix spike/Matrix spike duplicate (%R)

Green Valley Citgo Project

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL027154/ECL027163

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Field Blank Data Qualification Summary - SDG ECL027154/ECL027163

No Sample Data Qualified in this SDG

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Level IV

COMPLETENESS WORKSHEET

mK.

Date: 4- 22- 13 Page: Reviewer:_ 2nd Reviewer:

Laboratory: Enviro-Chem Laboratories, Inc.

Dissolved Chromate as Cr

METHOD: Chromate (EPA Method 218.7)

SDG #: ECL027154/ECL027163

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 11-13-12 +420094 11-14-12
11	Initial calibration	A	
- 111.	Calibration verification	A	
IV	Blanks	A	
v	Matrix Spike/Matrix Spike Duplicates	SW	MS/MSD
VI.	Duplicates	N	
VII.	Laboratory control samples	N	not required
VIII.	Sample result verification	A	V
IX.	Overall assessment of data	A	
Х.	Field duplicates	N	
XI	Field blanks	N	

Note: A = Acceptable N = Not provided/applicable SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples: ail

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	ail Water			<u></u>			
1	3985 Farm First Draw	11	3085 Farm First DrawDUP	21		31	
2	3985 Farm Flushed	12		22		32	
3	3933 Rosewood First Draw	13		23		33	
4	3933 Rosewood Flushed	14		24		34	
5	11894 Barley First Draw	15		25		35	
6	11894 Barley Flushed	16		26		36	
7	3991 Farm First Draw	17		27		37	
8	3991 Farm Flushed	18		28		38	
9	3985 Farm First DrawMS	19		29		39	
10	3985 Farm First DrawMSD	20		30	PBW	40	

Notes:_

LDC #: 29503D6

VALIDATION FINDINGS CHECKLIST

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Page: Lof 2 Reviewer: MG 2nd Reviewer: V

	T			
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	<i></i>		<u> </u>	· · ·
All technical holding times were met.	~			
Cooler temperature criteria was met.				
II. Calibration				
Were all instruments calibrated daily, each set-up time?				
Were the proper number of standards used?	/			
Were all initial calibration correlation coefficients > 0.995?	\checkmark			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	~			
Were titrant checks performed as required? (Level IV only)				
Were balance checks performed as required? (Level IV only)			\checkmark	
III. Blanks				
Was a method blank associated with every sample in this SDG?	1			LRBS
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		/		
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.	/			
V. Laboratory control samples				
Was an LCS anaylzed for this SDG?		\checkmark		
Was an LCS analyzed per extraction batch?		\checkmark		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?			\checkmark	
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			1	

VALIDATION FINDINGS CHECKLIST



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Validation Area	Yes	No	NA	Findings/Comments		
VII. Sample Result Verification						
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/					
Were detection limits < RL?	1					
VIII. Overall assessment of data						
Overall assessment of data was found to be acceptable.	\checkmark					
IX. Field duplicates						
Field duplicate pairs were identified in this SDG.		/				
Target analytes were detected in the field duplicates.			\checkmark			
X. Field blanks						
Field blanks were identified in this SDG.		\checkmark				
Target analytes were detected in the field blanks.			\checkmark			

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Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

218.7 METHOD: Inorganics, EPA Method_ Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Was a matrix spike analyzed for each matrix in this SDG? A/A

Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken. A)N/A AN N/A

Were all duplicate sample relative percent differences (RPD) \leq 20% for water samples and \leq 35% for soil samples? EVEL IV ONLY:

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. Y N N/A

Qualifications	L/UL/A											
Associated Samples	91											
RPD (Limits)												
MSD %Recoverv	69.3(85-115)											
MS %Recoverv	71 (85-115)											
Analyte	CrOy-Cr											
Matrix	water											
al asmism	9/10											
 		 -	 	 -	 \square	 	 _	\vdash	 	 	 	-

Comments:

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LDC #: 29503D6

Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Ť Page: Lof 2nd Reviewer: Reviewer:

7.816 METHOD: Inorganics, Method

11-13-12

++++3-13 mg The correlation coefficient (r) for the calibration of $\frac{cv \partial_4 - cv}{v}$ was recalculated. Calibration date:

An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

%R = <u>Found</u> x 100 True

Where, Found = concentration of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source

			(0 m c	4 60	Recalculated	Reported	
Type of Analysis	Analyte	Standard ID	Found (units)	True (units)	r or %R	r or %R	Acceptable (Y/N)
Initial calibration		Blank	1	l			
		Standard 1	1/8m) 20.0	0.0485			
	(Standard 2	0.05 ()	0.1199			
	Cr04	Standard 3	0.10 ()	7966.0	•		
	as	Standard 4	0.25 ()	0.5841	V 7= 1,0000	1 = 1. 0000	<u> </u>
	5 C	Standard 5	0.50 ()	1.1978			
		Standard 6	() 00.1	2.3853			
		Standard 7	5.00 (1)	11.9665			
Calibration verification	Croy- Cr	M07-222	(18m) pieco	(78m) 20.0	109.5	109.7	
Calibration verification	1	1	þ	ł		1	
Calibration verification))		1	l		

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recalculated results.

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the

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LDC #. 29503D6

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: <u>Lof L</u> Reviewer: <u>MG</u> 2nd Reviewer: <u>L</u>

METHOD: Inorganics, Method 2(8.7

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). Found = Where, %R = Found x 100 True

True = concentration of each analyte in the source.

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

 $RPD = \frac{|S-D|}{|S+D|/2} \times 100 \quad Where, S = Original sample concentration (S+D)/2 \quad D = Duplicate sample concentration$

-		and and					
	Acceptable (Y/N)	(607)	l		≻		
Reported	%R / RPD	047 KFD	١		12		2.5
Recalculated	%R/RPD		1		11		2.5
	True / D (units)		l		(~1)g~) 1		0.6925 (mg/L)
	Found / S (units)		١	(SSR-SR)	(1) (mg/r)		(1) 001L.0
	Element		1		Croy-CN		Cr04-Cr
	Type of Analysis	Laboratory control sample		Matrix spike sample		Duplicate sample	
	Sample ID)		o		9 /10

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC #: <u>29503D6</u> VALIDATION FINDINGS WORKSHEET Page: of Sample Calculation Verification Reviewer: 2nd reviewer:							
METHO	OD: Inorganics, Metho	a 218.7			Ū.		
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". W N/A Have results been reported and calculated correctly? Are results within the calibrated range of the instruments? Are all detection limits below the CRQL? Compound (analyte) results for #6 Dissolved Chromate as Cr recalculated and verified using the following equation:							
Concentr	liated and venified using	g the following equation:					
Wx t	+ b 2 39.0 2	0.062 = 2.3902	(x) - 0.00	12			
neve d:	b = -0.0012 1 = 1x	0.0264 Mg/L =	= X				
#	Sample ID	Analyte	Reported Concentration	Calculated Concentration	Acceptable (Y/N)		
	6	CrOy-Cr	0.026	0.026	Ý		
		· · · · · · · · · · · · · · · · · · ·					
		· · · · · · · · · · · · · · · · · · ·					

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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	December 1, 2012
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL027334

Sample Identification

3833 Greenridge First Draw
3833 Greenridge Flushed
3833 Greenridge First DrawF
3833 Greenridge FlushedF
3833 Greenridge First DrawMS
3833 Greenridge First DrawDUP

Samples appended with "F" were analyzed as dissolved

Introduction

This data review covers 6 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 200.8 for Chromium and Lead.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Calibration

The initial and continuing calibrations were performed at the required frequency.

The calibration standards criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No chromium or lead contaminants were found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. ICP Interference Check Sample (ICS) Analysis

ICP interference check sample (ICS) analysis was not required by the method.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

IX. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

XII. Sample Result Verification

All sample result verifications were acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDG ECL027334

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL027334

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL027334

No Sample Data Qualified in this SDG

LDC #: 29503E4	VALIDATION COMPLETENESS WORKSHEET	Date: <u>4-22-</u> 13
SDG #: ECL027334	Level IV	Page:of

Laboratory: Enviro-Chem Laboratories, Inc.

Reviewer: MG 2nd Reviewer:

METHOD: Chromium & Lead (EPA Method 200.8)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 12-1-12
II.	ICP/MS Tune	A	
111.	Calibration	<u>A</u>	
IV.	Blanks	_A	
V.	ICP Interference Check Sample (ICS) Analysis	N	not required
VI.	Matrix Spike Analysis	A	MS V
VII.	Duplicate Sample Analysis	<u>A</u>	DUP
VIII.	Laboratory Control Samples (LCS)	_A	LCS
IX.	Internal Standard (ICP-MS)	<u>A</u>	
X .	Furnace Atomic Absorption QC	N	not utilized
XI.	ICP Serial Dilution	N	not performed
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	N	
xv	Field Blanks	N	

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

	<u> </u>					
1	3833 Greenøridge First Draw	11		21	31	
2	3833 Green≱ridge Flushed	12		22	32	
3	3833 Green∳ridge First DrawF	13		23	33	
4	3833 Greenøridge FlushedF	14		24	34	
5	3833 Greenbridge First DrawMS	15		25	35	
6	3833 Greenkridge First DrawDUP	16		26	36	
7	Good Creenpinage i net brans en	17		27	37	
0	3 	18		28	38	
<u> </u>		10		29	39	
9		20	PRIN	30	40	
10	l	120				<u> </u>

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Samples appended with "F" were analyzed as dissolved Notes:

LDC #: 29503E4

VALIDATION FINDINGS CHECKLIST

Page: Lof_2 Reviewer: MG 2nd Reviewer: _____

Method:Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments					
I. Technical holding times									
All technical holding times were met.	\checkmark								
Cooler temperature criteria was met.	/								
II. ICP/MS Tune									
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	\checkmark								
Were %RSD of isotopes in the tuning solution ≤5%?	\checkmark								
III. Calibration									
Were all instruments calibrated daily, each set-up time?									
Were the proper number of standards used?	/								
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?	/								
Were all initial calibration correlation coefficients 2.995?	\checkmark								
IV. Blanks									
Was a method blank associated with every sample in this SDG?	/								
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/							
V. ICP Interference Check Sample									
Were ICP interference check samples performed daily?		\checkmark							
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?									
VI. Matrix spike/Matrix spike duplicates									
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/								
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/								
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were \leq 5X the RL, including when only one of the duplicate sample values were \leq 5X the RL.	/								
VII. Laboratory control samples									
Was an LCS anaylzed for this SDG?	1								
Was an LCS analyzed per extraction batch?									
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	<								
LDC #: 29503E4

VALIDATION FINDINGS CHECKLIST

Page: 2 of	2
Reviewer: M	3
2nd Reviewer:	\leq

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			\checkmark	·.
Do all applicable analysies have duplicate injections? (Level IV only)			\checkmark	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)				
Were analytical spike recoveries within the 85-115% QC limits?			\checkmark	·
IX. ICP Serial Dilution	r		,	
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		\checkmark		
Were all percent differences (%Ds) < 10%?				
Was there evidence of negative interference? If yes, professional judgement will be used to gualify the data.			1	
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)				
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	1			
If the %Rs were outside the criteria, was a reanalysis performed?			1	
XI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
XII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/		Í.	
XIV. Field duplicates		· · · · · · · · · · · · · · · · · · ·		• • • • • • • • • • • • • • • • • • •
Field duplicate pairs were identified in this SDG.		1		
Target analytes were detected in the field duplicates.				1
XV. Field blanks		· · · · · ·		·····
Field blanks were identified in this SDG.		1		
Target analytes were detected in the field blanks.			1	

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LDC #: 29503E4

IF

VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: ____of ____ Reviewer: _____MG 2nd reviewer: _____ ł

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All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
(→4	W	Al, Sb, As, Ba, Be, Cd, Ca(Cr)Co, Cu, Fe, Pb)Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
ac 5.6	r	Al, Sb, As, Ba, Be, Cd, Ca Cr Co, Cu, Fe Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Analysis Method.
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
ICP-MS	\sim	Al, Sb, As, Ba, Be, Cd, Ca Cr Co, Cu, Fe, Pb Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
GEAA		<u>Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V Zn Mo B Si CN</u>

Comments: Mercury by CVAA if performed

LDC # 29503E4

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: L of L Reviewer: MC 2nd Reviewer: //

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration (in ug/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source %R = <u>Found</u> x 100 True

Standard ID Type o					Recalculated	Reported	
	of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	libration)						
1106 ICP/MS (Initia	il calibration)	5	98.670	100	98.67	98.67	٢
CVAA (Initial (calibration)						
ICP (Continuit	ng calibration)						
CKS ICP/MS (Cont	tinuing calibration)	Pb	196. 800	000	98.40	98.40	
CVAA (Contin	uing calibration)						
GFAA (Initial	calibration)						
GFAA (Contin	uing calibation)						

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

Where, Found = Concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). True = Concentration of each analyte in the source. %R = Found x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

 $RPD = \frac{|S-D|}{|S+D|/2} \times 100$ Where, S = Original sample concentration
D = Duplicate sample concentration

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = [I-SDR] x 100

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

							Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / I (units)		True / D / SDR	(units)	%R / RPD / %D	%R / RPD / %D	Acceptable (Y/N)
ł	ICP interference check	1	1				ł		
LCS 4548	Laboratory control sample	Pp	48.890 (u	34)	50	(mg/r)	97.8	97.8	\succ
i ez	Matrix spike	د د	(SSR-SR) 48.050 (u	(⁷ /8	50	(mg/r)	96.1	1.96	
9 6611/0811	Duplicate	٩٩	5.144 (W	31,	5.305	(n g /r)	3.1	3.1	-7
	ICP serial dilution								

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #	29503 E4	VALIDATION FINDINGS WORKSHEET Page:of Sample Calculation Verification Reviewer:OG 2nd reviewer:O Page:o				
METH Please	OD: Trace Metals (EPA	A SW 846 Method 6010/6020/7000) ww for all questions answered "N". Not appl	licable questions are	e identified as "N/	A".	
	N/A Have results N/A Are results w N/A Are all detect	been reported and calculated correctly? ithin the calibrated range of the instrument tion limits below the CRDL?	ts and within the line	ear range of the IC	CP?	
Detect equati	ted analyte results for _ on:	# 2, Pb	were recalcu	lated and verified	using the following	
Concen RD FV In, Vol.	tration = <u>(RD)(FV)(Dil)</u> (In. Vol.) = Raw data conce = Final volume (m = Initial volume (m	Recalculation: intration i) ii) or weight (G) Recalculation: (39.300 Mg/L))(0.050) = 39	.300 Mg/	
Dil #	= Dilution factor	Analyte	Reported Concentration (Mg/L)	Calculated Concentration	Acceptable (Y/N)	
	2	Pb	39.3	39.3	Ý	
	·····					
					· · ·	
Note:			<u> </u>			

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LDC Report# 29503E6

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	December 1, 2012
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL027334

Sample Identification

3833 Greenridge First Draw3833 Greenridge Flushed3833 Greenridge First DrawMS3833 Greenridge First DrawMSD

Introduction

This data review covers 4 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 218.7 for Dissolved Chromate as Chromium.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. Initial Calibration

All criteria for the initial calibration were met.

III. Calibration verification

Calibration verification frequency and analysis criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No dissolved chromate as chromium was found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
3833 Greenridge First DrawMS/MSD (All samples in SDG ECL027334)	Dissolved chromate as chromium	54.4 (85-115)	57.6 (86-115)	-	L (all detects) UL (all non-detects)	A

VI. Duplicates

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VII. Laboratory Control Samples

Laboratory control sample (LCS) analysis was not required by the method.

VIII. Sample Result Verification

All sample result verifications were acceptable.

IX. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

X. Field Duplicates

No field duplicates were identified in this SDG.

Green Valley Citgo Project Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL027334

SDG	Sample	Analyte	Flag	A or P	Reason
ECL027334	3833 Greenridge First Draw 3833 Greenridge Flushed	Dissolved chromate as chromium	L (all detects) UL (all non-detects)	A	Matrix spike/Matrix spike duplicate (%R)

Green Valley Citgo Project

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL027334

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Field Blank Data Qualification Summary - SDG ECL027334

No Sample Data Qualified in this SDG

LDC #: 2950366	VALIDATION COMPLETENESS WORKSHEET	Date: <u>4-22-</u> 13
SDG #: ECL027334	Level IV	Page:of
Laboratory: Enviro-Chem	Laboratories, Inc.	Reviewer: MG
Dissolved C	Chromate as Cr and	2nd Reviewer:
METHOD: Chromoto /ED/	Method 218 7)	

METHOD: Ghromate (EPA Method 218.7)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 12-1-12
	Initial calibration	A	
Ш.	Calibration verification	Á	
IV	Blanks	A	
V	Matrix Spike/Matrix Spike Duplicates	SŴ	MS/MSD
VI.	Duplicates	N	
VII.	Laboratory control samples	N	not required
VIII.	Sample result verification	A	
IX.	Overall assessment of data	A	
Х.	Field duplicates	N	
XI	Field blanks	N	

A = Acceptable Note: N = Not provided/applicable SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples: ai

г		all water				<u> </u>	
	1	3833 Greenøridge First Draw	11		21	31	
	2	3833 Greenøridge Flushed	12		22	32	
	3	3833 Greenøridge First DrawMS	13		23	33	
	4	3833 Greenøridge First DrawMSD	14		24	34	
. 4	5	3833 Green First BrawDUP	15		25	35	<u></u>
Ā	6		16		26	36	
	7		17		27	37	
	8		18		28	38	
	9		19		29	39	
	10		20	PBW	30	40	

Notes:

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LDC #: 29503E6

VALIDATION FINDINGS CHECKLIST



Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	1			
Cooler temperature criteria was met.	1			
II. Calibration				
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients ≥ 0.995?	\checkmark			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	\checkmark			
Were titrant checks performed as required? (Level IV only)			1	
Were balance checks performed as required? (Level IV only)			1	
III. Blanks		<u></u>		
Was a method blank associated with every sample in this SDG?	V			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		1		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	\checkmark			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		/		
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.	/			
V. Laboratory control samples				
Was an LCS anaylzed for this SDG?		/		
Was an LCS analyzed per extraction batch?		/		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?			/	
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?			/	

Method: Inorganics (EPA Method 218.7)

VALIDATION FINDINGS CHECKLIST



Validation Area	Yes	No	NA	Findings/Comments						
VII. Sample Result Verification										
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/									
Were detection limits < RL?	/									
VIII. Overall assessment of data										
Overall assessment of data was found to be acceptable.	\checkmark									
IX. Field duplicates										
Field duplicate pairs were identified in this SDG.		\checkmark								
Target analytes were detected in the field duplicates.			\checkmark							
X. Field blanks										
Field blanks were identified in this SDG.		\checkmark		/						
Target analytes were detected in the field blanks.			/							

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DC #

Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

E Page: 1 of Reviewer:__ 2nd Reviewer:

B18.7 METHOD: Inorganics, EPA Method_ Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Was a matrix spike analyzed for each matrix in this SDG?

Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken. YON N/A ØN N/A

Were all duplicate sample relative percent differences (RPD) < 20% for water samples and <35% for soil samples? LEVEL IV ONLY:

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. Y)N N/A

				 	-	 				 	 	 	-	 	
Qualifications	L/UL/A														
Associated Samples	a11														
RPD (I imits)															
MSD %Recovery	57.6 <i>(8</i> 5-115														
MS %Recovery	54.4 (85-115)	•													
Analyte	Cr Oy-Cr	,													
Matrix	water				_										
UI USW/SW	3/4														
#		_	 	 	-	 _	_	-	-	 	 	 -			_

Comments:

MSD.6

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Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ð Page: Reviewer. 2nd Reviewer:

> 7.816 METHOD: Inorganics, Method _

11-13-12

++++3 -+3 -m4 The correlation coefficient (r) for the calibration of $\frac{cv \partial_{4} - cv}{cv \partial_{4} - cv}$ was recalculated. Calibration date:

An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source %R = <u>Found</u> x 100 True

	Acceptable (Y/N)					>					>		
Reported	r or %R				r 3 - 1 0000				94	1			
Recalculated	r or %R		_		¢	V == 1,0000					94	þ)
Area	True (units)	l	0.0485	0.1199	7966.0	0.5841	1.1978	2.3853	11.9665		0.02 (481)		J
Conc	Found (units)	1	0.02 (mg/L	0.05 ()	0.10 ()	0.25 ()	0.50 ()	1.00 ()	5.00 (1)		0.0138 (48/L)	1	ł
	Standard ID	Blank	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7	1147	200 - 20M	l)
	Analyte			ç	Cr04	as	ა				Croy- Cr)
	Type of Analysis	Initial calibration							-	Calibration verification		Calibration verification	Calibration verification

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

CALCLC.6

29503E6 LDC #:

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: Lof Reviewer: 2nd Reviewer:

> 7.812 METHOD: Inorganics, Method _

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

Found = concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). True = concentration of each analyte in the source. Where, %R = <u>Found</u> x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

။ ။ ပ ပ Where, $RPD = |S-D| \times 100$ (S+D)/2

Original sample concentration Duplicate sample concentration

	cceptable (Y/N)	(V/N)	1		~		>
	∢						
Reported	%R/RPD	%R / RPD	1		54.4		5.6
Recalculated	%R / RPD	%R / RPD	1		54.4		5. 6
	True / D (units)	(units)	1		(~1/Bm) 1		0.5755(mg/L)
	Found / S (units)	(units)	I	(SSR-SR)	0.5442 (mg/)		0.5443 (mg/L)
	Element	Liement	1		Cr 0+ - Cr		Croy-Cu
	Type of Analysis	type or Analysis Laboratory control sample		Matrix spike sample		Duplicate sample	
	Sample ID			1943	3	1943/1302	3/4

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC #: 2950	3E6 V	ALIDATION FIN	DINGS WOR	KSHEET ation	Page: _ l of Reviewer: 2nd reviewer:					
METHOD: Inorganics, Method										
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". N = N/A Have results been reported and calculated correctly? N = N/A Are results within the calibrated range of the instruments? N = N/A Are all detection limits below the CRQL?										
Compound (analyte	e) results forl	samples =	N.D.	-reported-	with a positive detect wore					

Concentration =

r;

Recalculation:

#	Sample ID	Analyte	Reported Concentration	Calculated Concentration (Mg/L)	Acceptable (Y/N)
		nane			

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Note:___

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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	March 12, 2013
LDC Report Date:	April 24, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.

Sample Delivery Group (SDG): ECL028223

Sample Identification

11712 Serene-PT1 Total 11712 Serene-PT1DB Total 11712 Serene-PT2 Total 11712 Serene-PT3 Total 11712 Serene-PT4 Total 11712 Serene-FB Total 11712 Serene-PT1 Dissolved 11712 Serene-PT1 DB Dissolved 11712 Serene-PT2 Dissolved 11712 Serene-PT3 Dissolved 11712 Serene-PT4 Dissolved 11712 Serene-FB Dissolved 11712 Serene-PT1 TotalMS 11712 Serene-PT1 TotalDUP 11712 Serene-FB TotalMS 11712 Serene-FB TotalDUP

Samples appended with "F" were analyzed as dissolved

Introduction

This data review covers 16 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 200.8 for Chromium and Lead.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Calibration

The initial and continuing calibrations were performed at the required frequency.

The calibration standards criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No chromium or lead contaminants were found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

V. ICP Interference Check Sample (ICS) Analysis

ICP interference check sample (ICS) analysis was not required by the method.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) analyses were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

IX. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

XII. Sample Result Verification

All sample result verifications were acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

Samples 11712 Serene-PT1 Total and 11712 Serene-PT1DB Total and samples 11712 Serene-PT1 Dissolved and 11712 Serene-PT1 DB Dissolved were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concen	Concentration (ug/L)							
Analyte	11712 Serene-PT1 Total	11712 Serene-PT1DB Total	RPD						
Lead	567	180	104						

	Concen	tration (ug/L)	
Analyte	11712 Serene-PT1 Dissolved	11712 Serene-PT1 DB Dissolved	RPD
Lead	82.1	85.5	4

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDGECL028223

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDGECL028223

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDGECL028223

No Sample Data Qualified in this SDG

	VALIDATION	COMPLE	ETENESS	WORKSH	IEET
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Level IV

SDG #: ______

LDC #: 29503F4

Laboratory: Enviro-Chem Laboratories, Inc.

Date: <u>4-22-13</u> Page: <u>1 of 1</u> Reviewer: <u>MG</u> 2nd Reviewer: <u>(</u>

METHOD: Chromium & Lead (EPA Method 200.8)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 3-12-13
II.	ICP/MS Tune	A	
111.	Calibration	<u> </u>	
1V.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	N	not required
VI.	Matrix Spike Analysis	A	MS
VII.	Duplicate Sample Analysis	A	DUP
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
Х.	Furnace Atomic Absorption QC	N	not utilized
XI.	ICP Serial Dilution	N	not performed
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A_	
XIV.	Field Duplicates	SW	D = 1 + 2, $D = 7 + 8$
xv	Field Blanks	ND	FB=6,12

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

11712 Serene-PT1 Total	11	11712 Serene-PT4 Dissolved	21		21	
11712 Serene-PT1DB Total					-131-	
	12	11712 Serene-FB Dissolved	22		32	·······
11712 Serene-PT2 Total	13	11712 Serene-PT1 TotalMS	23		33	
11712 Serene-PT3 Total	14	11712 Serene-PT1 TotalDUP	24		34	
11712 Serene-PT4 Total	15	11712 Serene-FB TotalMS	25		35	
11712 Serene-EB Total	16	11712 Serene-FB TotalDUP	26		36	
11712 Serence PT1 Dissolved	17		27		37	
T 11712 Selelle-PT1 Dissolved	10		28		38	
11712 Serene-PKT DB Dissolved	10		29		39	
11712 Serene-P12 Dissolved	119		30	PBW	40	
	1712 Serene-PT2 Total 1712 Serene-PT3 Total 1712 Serene-PT4 Total 1712 Serene-FB Total 1712 Serene-PT1 Dissolved 1712 Serene-PT1 DB Dissolved 1712 Serene-PT2 Dissolved 1712 Serene-PT3 Dissolved	1712 Serene-PT2 Total 13 1712 Serene-PT3 Total 14 1712 Serene-PT4 Total 15 1712 Serene-FB Total 16 1712 Serene-PT1 Dissolved 17 1712 Serene-PT1 Dissolved 17 1712 Serene-PT2 Dissolved 18 1712 Serene-PT2 Dissolved 19 1712 Serene-PT3 Dissolved 20	1712 Serene-PT2 Total 13 11712 Serene-PT1 TotalMS 1712 Serene-PT3 Total 14 11712 Serene-PT1 TotalDUP 1712 Serene-PT4 Total 15 11712 Serene-FB TotalMS 1712 Serene-FB Total 16 11712 Serene-FB TotalDUP 1712 Serene-PT1 Dissolved 17 1712 Serene-PT2 Dissolved 18 1712 Serene-PT2 Dissolved 19 1712 Serene-PT3 Dissolved 20	1712 Serene-PT2 Total 13 11712 Serene-PT1 Total/US 23 1712 Serene-PT3 Total 14 11712 Serene-PT1 Total/DUP 24 1712 Serene-PT4 Total 15 11712 Serene-FB Total/DUP 25 1712 Serene-FB Total 16 11712 Serene-FB Total/DUP 26 1712 Serene-PT1 Dissolved 17 27 1712 Serene-PT1 Dissolved 18 28 1712 Serene-PT2 Dissolved 19 29 1712 Serene-PT3 Dissolved 20 30	1712 Serene-PT2 Total 13 11712 Serene-PT1 TotalMIS 23 1712 Serene-PT3 Total 14 11712 Serene-PT1 TotalDUP 24 1712 Serene-PT4 Total 15 11712 Serene-FB TotalMIS 25 1712 Serene-FB Total 16 11712 Serene-FB TotalDUP 26 1712 Serene-PT1 Dissolved 17 27 1712 Serene-PT1 Dissolved 18 28 1712 Serene-PT2 Dissolved 19 29 1712 Serene-PT3 Dissolved 20 30 PB v/	1712 Serene-PT2 Total 13 11712 Serene-PT1 TotalMS 23 33 1712 Serene-PT3 Total 14 11712 Serene-PT1 TotalDUP 24 34 1712 Serene-PT4 Total 15 11712 Serene-FB TotalMS 25 35 1712 Serene-FB Total 16 11712 Serene-FB TotalDUP 26 36 1712 Serene-PT1 Dissolved 17 27 37 1712 Serene-PT1 Dissolved 18 28 38 1712 Serene-PT2 Dissolved 19 29 39 1712 Serene-PT3 Dissolved 20 30 PBW 40

Notes: Samples appended with "F" were analyzed as dissolved

LDC #: 29503F4

VALIDATION FINDINGS CHECKLIST

Page: Lof 2 Reviewer: MG 2nd Reviewer: _____

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Method: Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments	
I. Technical holding times					
All technical holding times were met.	/				
Cooler temperature criteria was met.	\checkmark				
II. ICP/MS Tune					
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	\checkmark				
Were %RSD of isotopes in the tuning solution ≤5%?	1				
III. Calibration					
Were all instruments calibrated daily, each set-up time?	\checkmark				
Were the proper number of standards used?	\checkmark				
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?	/				
Were all initial calibration correlation coefficients > 0.995?	\checkmark				
IV. Blanks					
Was a method blank associated with every sample in this SDG?	\checkmark				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/			
V. ICP Interference Check Sample					
Were ICP interference check samples performed daily?		/			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?					
VI. Matrix spike/Matrix spike duplicates					
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/				
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were \leq 5X the RL, including when only one of the duplicate sample values were \leq 5X the RL.	/				
VII. Laboratory control samples				e	
Was an LCS anaylzed for this SDG?					
Was an LCS analyzed per extraction batch?	\checkmark				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/				

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LDC #: 29503 F4

VALIDATION FINDINGS CHECKLIST

Page: 2 of	2
Reviewer: M	G
2nd Reviewer:	\leq

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Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			\checkmark	
Do all applicable analysies have duplicate injections? (Level IV only)			~	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?			\checkmark	
IX. ICP Serial Dilution				······································
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		/		
Were all percent differences (%Ds) < 10%?				
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			 ✓ 	
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)				······································
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/			
If the %Rs were outside the criteria, was a reanalysis performed?			\bigvee	
XI. Regional Quality Assurance and Quality Control			·	
Were performance evaluation (PE) samples performed?	ļ	/		· · · · · · · · · · · · · · · · · · ·
Were the performance evaluation (PE) samples within the acceptance limits?				
XII. Sample Result Verification				· · · · · · · · · · · · · · · · · · ·
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	1		ľ	
XIV. Field duplicates	· · · · · · · · · · · · · · · · · · ·			······································
Field duplicate pairs were identified in this SDG.				
Target analytes were detected in the field duplicates.				
XV. Field blanks				
Field blanks were identified in this SDG.	/	ļ	L	
Target analytes were detected in the field blanks.		 / 		

LDC #: 29503 F4

VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

Sample ID	Matrix	
1->12	W	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
6613→16		Al, Sb, As, Ba, Be, Cd, Ca(Cr, Co, Cu, Fe(Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		AI, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, TI, V, Zn, Mo, B, Si, CN ⁻ ,
		AI, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ^{-,}
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
		Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
ICP-MS	W	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ⁻ ,
GEAA		Al, Sh. As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Ph, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN ¹
Comments:	Mercun	y by CVAA if performed

LDC#: 29503F4 VALIDATION FINDINGS WORKSHEET Field Duplicates

Page: <u>l</u> of <u>l</u> Reviewer: <u>MG</u> 2nd Reviewer: <u>____</u>

METHOD: Metals (EPA Method 6010B/7000)



Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentra	tion (ug/L)		
Analyte	1	2	RPD	
Lead	567	180	104	

V:\FIELD DUPLICATES\FD_inorganic\29503F4.WPD

	Concentra	tion (ug/L)		
Analyte	7	8	RPD	
Lead	82.1	85.5	4	

V:\FIELD DUPLICATES\FD_inorganic\29503F4.WPD

LDC# 29503 F4

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

5 5) ď 2nd Reviewer: Reviewer: Page:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration (in ug/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source %R = <u>Found</u> x 100 True

					Recalculated	Renorted	
Ctondard ID	Time of Analysis	Flamant	Found (und))	True (uoli)	%R	Я%	Acceptable (Y/N)
			1-122	1-10-1 pp - 1			
ICP (II	nitial calibration)						
1758 ICPM	1S (Initial calibration)	Pه	96.600	100	96.60	96.60	>
CVAA	(Initial calibration)						
ICP (C	Continuing calibration)						
1334 ICPIN	AS (Continuing calibration)	r C	198.500	Col	99.25	99.25	
CVAA	V (Continuing calibration)						
GFAA	A (Initial calibration)						
GFAA	A (Continuing calibation)						

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

CALCLC.4SW

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VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

2nd Reviewer: Page: Reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

 Where,
 Found =
 Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

 Found =
 SSR (spiked sample result) - SR (sample result).
 True =
 Concentration of each analyte in the source.
 %R = Found_x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

Where, S = Original sample concentration D = Duplicate sample concentration $RPD = \underline{|S-D|} \times 100$ (S+D)/2

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = [i-SDR] × 100

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

	Acceptable (Y/N)		Y			>	l
Reported	%R / RPD / %D		95.9		18.5	3.2	
Recalculated	%R / RPD / %D	١	95.9	(48.4	3.2	
	(units)		(7/ bm)		5/2/1	(7/ bm)	
	True / D / SDR		50	(50	549.80	-
			(ng/L)	("0")	(~2(~)	(1/bm)	>
	Found / S (units))	47.950	(SSR-SR)	49.470	567.50	
	Element	1	٩d		3	٩d	1
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike		Duplicate	ICP serial dilution
	Sample ID	1	1309 LCS 4793	1317	13	1312/1315 14)

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 29503 F4		VALIDATION FINDINGS	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification		
METH	IOD: Trace Metals (EP)	A SW 846 Method 6010/6020/7000)			
	e see qualifications belo <u>N/A</u> Have results <u>N/A</u> Are results w <u>N/A</u> Are all detect	bw for all questions answered "N". Not app been reported and calculated correctly? within the calibrated range of the instrument tion limits below the CRDL?	blicable questions an Its and within the line	e identified as "N/. ear range of the IC	A". CP?
Detec equat	ted analyte results for _ ion:	<u>#1, P6</u>	were recalcu	lated and verified	using the following
Concer	ntration = <u>(RD)(FV)(Dil)</u> (in. Vol.)	Recalculation:	Varia	١	
RD FV In. Vol. Dil	= Raw data conce = Final volume (m = Initial volume (m = Dilution factor	entration (567.500 mg/L a) h) or weight (G) 0.05	.)(0.0501 	·) = 56 ⁻	7.500 mg/L
#	Sample ID	Analyte	Reported Concentration (Mg/L)	Calculated Concentration (Mg/L)	Acceptable (Y/N)
	i	Рь	567	568	Ý
					
9	<u> </u>	<u></u> Ръ	180	180	↓
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Note:					

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LDC Report# 29503F6

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	March 12, 2013
LDC Report Date:	April 25, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL028223

Sample Identification

11712 Serene-PT1 Dissolved 11712 Serene-PT1 DB Dissolved 11712 Serene-PT2 Dissolved 11712 Serene-PT3 Dissolved 11712 Serene-PT4 Dissolved 11712 Serene-FB Dissolved 11712 Serene-PT1 DissolvedMS 11712 Serene-PT1 DissolvedMSD

Introduction

This data review covers 8 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 218.7 for Dissolved Chromate as Chromium.

This review follows the EPA Region III Innovative Approaches for Data Validation (EPA June 1995).

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

The following are definitions of the data qualifiers:

- U Indicates the compound or element was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- K Indicates the reported value may be biased high.
- L Indicates the reported value may be biased low.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.
- UL Indicates the compound or element was analyzed for but not detected. The sample detection limit is probably higher.
- B Indicates the compound or element was analyzed for but not detected substantially above the level reported in laboratory or field blanks.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. Initial Calibration

All criteria for the initial calibration were met.

III. Calibration verification

Calibration verification frequency and analysis criteria were met.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No dissolved chromate as chromium was found in the initial, continuing and preparation blanks.

Sample 11712 Serene-FB Dissolved was identified as a field blank. No dissolved chromate as chromium was found.

V. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits.

VI. Duplicates

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VII. Laboratory Control Samples

Laboratory control sample (LCS) analysis was not required by the method.

VIII. Sample Result Verification

All sample result verifications were acceptable.

IX. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

X. Field Duplicates

Samples 11712 Serene-PT1 Dissolved and11712 Serene-PT1 DB Dissolved were identified as field duplicates. No dissolved chromate as chromium was detected in any of the samples.

Green Valley Citgo Project Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL028223

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL028223

No Sample Data Qualified in this SDG

Green Valley Citgo Project Dissolved Chromate as Chromium - Field Blank Data Qualification Summary -SDG ECL028223

No Sample Data Qualified in this SDG

LDC #: 29503F6	VALIDATION COMPLETENESS WORKSHEET	Date: 4-22-13
SDG #: ECL028223	Level IV	Page: <u></u> of <u></u>
Laboratory: Enviro-Chem	Laboratories, Inc.	Reviewer: MG

_aboratory: Enviro-Chem Laboratories, Inc.

Dissolved Chromate as Cr METHOD: <u>Chromate</u> (EPA Method 218.7)

9mA

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 3-12-13
11	Initial calibration	A	
111.	Calibration verification	A	
IV	Blanks	A	
v	Matrix Spike/Matrix Spike Duplicates	A	MS/MSD
VI.	Duplicates	N	
VII.	Laboratory control samples	N	not required
VIII.	Sample result verification	A	
IX.	Overall assessment of data	A	
Х.	Field duplicates	ND	D= 1+2
XI	Field blanks	ND	FB=6

Note: A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank 2nd Reviewer:

Validated Samples:

_		MIT WAICH				
	1	11712 Serene-PT1 Dissolved	11		21	31
_	2	11712 Serene-PF1 DB Dissolved	12		22	32
ĺ	3	11712 Serene-PT2 Dissolved	13		23	33
	4	11712 Serene-PT3 Dissolved	14		24	34
ľ	5	11712 Serene-PT4 Dissolved	15		25	35
	6	11712 Serene-FB Dissolved	16		26	36
ľ	- <u>-</u>	11712 Serene-PT1 DissolvedMS	17		27	37
	8	11712 Serene-PT1 DissolvedMSD	18		28	38
	<u> </u>	11712 Scrone PT1 Dissolved DUP	19		29	39
	10		20	PBW	30	40

Notes:____
LDC #: 29503F6

VALIDATION FINDINGS CHECKLIST

Page: _ L of 2 Reviewer: _ MG 2nd Reviewer: _ ____

Method: Inorganics (EPA Method 218.7)									
Validation Area	Yes	No	NA	Findings/Comments					
I. Technical holding times									
All technical holding times were met.	1								
Cooler temperature criteria was met.	~								
II. Calibration									
Were all instruments calibrated daily, each set-up time?	1								
Were the proper number of standards used?	/								
Were all initial calibration correlation coefficients > 0.995?									
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/								
Were titrant checks performed as required? (Level IV only)			1						
Were balance checks performed as required? (Level IV only)			1						
III. Blanks									
Was a method blank associated with every sample in this SDG?	\checkmark			LRBs					
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/							
IV. Matrix spike/Matrix spike duplicates and Duplicates	,								
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	~								
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/								
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.	/								
V. Laboratory control samples									
Was an LCS anayized for this SDG?		\checkmark							
Was an LCS analyzed per extraction batch?		\checkmark							
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?			/						
VI. Regional Quality Assurance and Quality Control									
Were performance evaluation (PE) samples performed?		\checkmark							
Were the performance evaluation (PE) samples within the acceptance limits?			\checkmark						

LDC #: 29503F6

VALIDATION FINDINGS CHECKLIST



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1.1.1.1

Validation Area	Yes	No	NA	Findings/Comments					
VII. Sample Result Verification									
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/								
Were detection limits < RL?	\checkmark								
VIII. Overall assessment of data									
Overall assessment of data was found to be acceptable.	\checkmark								
IX. Field duplicates									
Field duplicate pairs were identified in this SDG.	/								
Target analytes were detected in the field duplicates.		/							
X. Field blanks									
Field blanks were identified in this SDG.	\checkmark								
Target analytes were detected in the field blanks.									

LDC # 39503F6

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Reviewer: AG 2nd Reviewer: 1

Page: Lof

METHOD: Inorganics, Method 218.7

1-14-13 The correlation coefficient (r) for the calibration of $\frac{C v O q - C v}{V O q - C v}$ was recalculated. Calibration date: An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration of each analyte measured in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source %R = <u>Found</u> × 100 True

			(A.c.a.c.	Recaiculated	Reported	
Type of Analysis	Analyte	Standard ID	Found (units)	True (units)	r or %R	r or %R	Acceptable (Y/N)
Initial calibration		Blank	•				
		Standard 1	2.02 (mg/r	0.0425			
		Standard 2	0.05 (]	0.1146			
	C.0.1	Standard 3	0.10	0.2167			
		Standard 4	0.25 (0.5487	r 3= 1,0000	0000	>-
	ŝ	Standard 5	0.50 ()	1.1172			
	5	Standard 6	() 0.1	2.2337			
		Standard 7	5.0 (4)	7947			
Calibration verification	roy-cr	Mon -222	1/bn) 6260.0	(1/gn) 20.0	134.5	134.4	>
Calibration verification)		1			l
Calibration verification	J	١		l		1	

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recalculated results.

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the

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LDC#: 29503F6

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: Lot J Reviewer: MG 2nd Reviewer: L

METHOD: Inorganics, Method 218.7

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). True = concentration of each analyte in the source. Found = Where, %R = <u>Found</u> x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

 $RPD = \frac{|S-D|}{|S+D|/2} \times 100 \quad Where, S = Original sample concentration (S+D)/2 D = Duplicate sample concentration$

Acceptable (Y/N) 9.401 %R / RPD Reported 2.3 104.6 2.3 **Recalculated** %R / RPD (T) (m² V) (~) bn) Lieoi (~) bn 22 (mg/) 1:0012 (mg /) True / D (units) l 1.0455 (mg/) Found / S (units) (SSR-SR) Croy - Cr Element I Laboratory control sample Type of Analysis Matrix spike sample Duplicate sample 13/ (225 Sample ID 3/1 1913 ٢

Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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LDC #: 295	03F6	VALIDATION F Sample Ca	INDINGS WORK	(SHEET tion	Page:of Reviewer: MC 2nd reviewer:
METHOD: Inor	ganics, Method	218.7			
Please see qua	lifications below fo Have results beer Are results within Are all detection li	r all questions answer reported and calcula the calibrated range c imits below the CRQL	ed "N". Not applicabl ted correctly? of the instruments? ?	e questions are iden	itified as "N/A".
Compound (ana recalculated and	alyte) results for d verified using the	all samples	= N.D.	roported \	with a positive detect were

......

Concentration =

······

Recalculation:

#	Sample ID	Analyte	Reported Concentration	Calculated Concentration (#9 / L)	Acceptable (Y/N)
				0	
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