Appendix LData Validation Reports – March and June through July<br/>2013



5405 Twin Knolls Road, Suite 1 • Columbia, MD 21045 • ph: 410.740.1911 • fax: 410.740.3299 • www.cgs.us.com

May 15, 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 (Sediment Sample) **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 validated sample was a sediment soil sample collected on March 12, 2013 from a domestic supply well pressure tank at a residence near the former Green Valley Citgo. The sample was analyzed by Phase Separation Science, Inc. for metals via EPA SW 846 Method 6020A.

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 laboratory protocol/contractual deviations. These include continuing laboratory calibration that was not performed within the required recovery limits and interference check sample analyses that were not within required recovery limits. The qualified mercury sample result was due to the continuing laboratory calibration percentage of recovery being higher than the acceptable limit. However, the qualifier is only applicable if the analyte was detected above the reporting limit (mercury was not detected above the reporting limit). All of the other qualified metal sample results listed were due to the interference check sample analysis percentage of recoveries being higher than the acceptable limit. This indicates that the detected results may be biased high.

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.

Sean P. Daniel Operations Manager

Attachments:

Data Validation Results Summary Laboratory Data Consultants, Data Validation Report 29656

### Monrovia BP/Former Green Valley Citgo MDE Case No. **2005-0834FR** Data Validation Results Summary – Qualified Results Only Inorganic Metals (Method 6020A)

Sample ID	Flagged Analysis Reported Concentration (mg/kg)	Flagged Analysis	Validation Qualifiers	Reason for Qualifier
	0.10 U	Method 6020A Mercury	K / P	1
	9.6	Method 6020A Arsenic	K / P	2
	120	Method 6020A Chromium	K / P	2
11712Serene-PTSediment	13	Method 6020A Cobalt	K / P	2
	220	Method 6020A Copper	K / P	2
	41	Method 6020A Nickel	K / P	2
	5.4	Method 6020A Vanadium	K / P	2

Table Notes:

U - Analyte Not Detected Above Specified Reporting Limit

Bold - Detected analyte concentration

mg/kg - milligrams per kilogram (parts per million)

K - Indicates the reported value may be biased high for all detected concentrations.

P - Indicates the finding is related to a protocol/contractual deviation.

1 – Continuing calibration (Percents of recovery (%R) of 118%/125% are outside acceptable limit range of 90-110%)

2 – Interference check sample analysis (%R of 125% / 133% /127 % / 123% / 125% / 124% are outside the acceptable limit range of 80-120%)



### 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 May 14, 2013

SUBJECT: Green Valley Citgo Project, Data Validation

Dear Mr. Daniel,

Enclosed is the final validation report for the fraction listed below. This SDG was received on May 3, 2013. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### LDC Project # 29656:

<u>SDG #</u>	<u>Fraction</u>

13031516 Metals

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
- EPA SW 846, Third Edition, Test Methods for Evaluating Solid Waste, update 1, July 1992; update IIA, August 1993; update II, September 1994; update IIB, January 1995; update III, December 1996; update IIIA, April 1998; IIIB, November 2004; Update IV, February 2007

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

Sincerely,

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Pei Geng / Project Manager/Senior Chemist

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Page	VII	SDG#	er/So	13031516																												T/PG	Shaded cells indicate Level IV validation (all other cells are Level III validation). These sample counts do not include MS, MSD, or DUP's.
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### Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
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Collection Date: March 12, 2013

LDC Report Date: May 14, 2013

Matrix: Sediment

Parameters: Metals

Validation Level:EPA Region III, Level IM2

Laboratory: Phase Separation Science, Inc.

Sample Delivery Group (SDG): 13031516

Sample Identification

11712Serene-PTSediment

### Introduction

This data review covers one sediment sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020A for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc.

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 with the following exceptions:

Date	Lab. Reference/ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
3/22/13	CCV 16:17	Mercury	118 (90-110)	11712Serene-PTSediment	K (all detects)	Р
3/22/13	CCV 17:44	Mercury	125 (90-110)	11712Serene-PTSediment	K (all detects)	P

### IV. Blanks

Method blanks were reviewed for each matrix as applicable. No metal 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

The frequency of analysis was met.

The criteria for analysis were met with the following exceptions:

ICS ID	Date/ Time	Analyte	%R (Limits)	Associated Samples	Flag	A or P
ICSAB	3/21/13 11:42	Arsenic Chromium Cobalt Copper Nickel Vanadium	125 (80-120) 133 (80-120) 127 (80-120) 123 (80-120) 125 (80-120) 124 (80-120)	11712Serene-PTSediment	K (all detects) K (all detects) K (all detects) K (all detects) K (all detects) K (all detects)	Ρ

### VI. 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.

### VII. Duplicate Sample Analysis

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.

### 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 Metals - Data Qualification Summary - SDG 13031516

SDG	Sample	Analyte	Flag	A or P	Reason
13031516	11712Serene-PTSediment	Mercury	K (all detects)	P	Continuing calibration (%R)
13031516	11712Serene-PTSediment	Arsenic Chromium Cobalt Copper Nickel Vanadium	K (all detects) K (all detects) K (all detects) K (all detects) K (all detects) K (all detects)	Ρ	ICP interference check sample analysis (%R)

### Green Valley Citgo Project Metals - Laboratory Blank Data Qualification Summary - SDG 13031516

### No Sample Data Qualified in this SDG

### Green Valley Citgo Project Metals - Field Blank Data Qualification Summary - SDG 13031516

No Sample Data Qualified in this SDG

LDC #:	29656A4	VALIDATION COMPLETENESS WORKSHEET	Date: 5-9-13
SDG #:_	13031516	Level IV	Page: 1 of
Laborato	ory: Phase Separatic	n Science, Inc.	Reviewer: MG

2nd Reviewer:

### METHOD: Metals (EPA SW846 Method 6020A)

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: 3-12-13
11.	ICP/MS Tune	A	
.	Calibration	SW	
_IV.	Blanks	SWA	
<u>V.</u>	ICP Interference Check Sample (ICS) Analysis	SW	
VI.	Matrix Spike Analysis	N	client specified
VII.	Duplicate Sample Analysis	N	it 66
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	SWA	r
Х.	Furnace Atomic Absorption QC	N	not utilized not performed
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	2	

Note:

A = Acceptable N = Not provided/applicable ND = No compounds detected R = Rinsate FB = Field blank

SW = See worksheet

Validated Samples:

	<u> </u>				 	
1 2	11712Serene-PTSediment	11		21	31	
2		12	,,,,,,,,,	22	 32	
3		13		23	 33	
4		14		24	 34	
5		15		25	35	
6		16		26	 36	
7		17		27	37	
8		18	•=	28	38	
9		19		29	 39	
10	[	20	PBS	30	40	

D = Duplicate

TB = Trip blank EB = Equipment blank

Notes:\_\_\_\_

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

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

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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				
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	$\bigvee$			
Were %RSD of isotopes in the tuning solution ≤5%?	$\checkmark$			
III. Calibration				
Were all instruments calibrated daily, each set-up time?	1			
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 <a> 0.995?</a>	/			
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.	X	$\checkmark$		
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?	$\checkmark$			
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?	~			

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### VALIDATION FINDINGS CHECKLIST



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				
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.			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?	$\checkmark$	x		
If the %Rs were outside the criteria, was a reanalysis performed?		X		
XI. Regional Quality Assurance and Quality Control			<b>.</b>	
Were performance evaluation (PE) samples performed?		1	ļ	
Were the performance evaluation (PE) samples within the acceptance limits?			$\checkmark$	1
XII. Sample Result Verification		r <u></u>		
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	1			
XIII. Overall assessment of data				······
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates	r	F		1
Field duplicate pairs were identified in this SDG.		/	ļ,	
Target analytes were detected in the field duplicates.			1	
XV. Field blanks				· · · · · · · · · · · · · · · · · · ·
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.				1

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### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



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All circled elements are applicable to each sample.

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Sample ID	Matrix	Target Analyte List (TAL)
	S	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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>-</sup> ,
		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 <sup>*</sup> ,
		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 <sup>-</sup> ,
<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 <sup>-</sup> ,
		Analysis Method
	S	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		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 <sup>*</sup> ,
GEAA		Al Sh As Ba Be Cd Ca Cr Co Cu Ee Ph Mg Mn Hg Ni K Se Ag Na TL V Zn Mo B Si CN <sup>-</sup>

Comments: Mercury by CVAA if performed

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$\mathbf{A}$
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LDC #:

## VALIDATION FINDINGS WORKSHEET Calibration

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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".

Were all instruments calibrated daily, each set-up time, and were the proper number of standards used? Were all initial and continuing calibration verification percent recoveries (%R) within the control limits of 90-110% for all analytes except mercury (80-120%) and cyanide (85-115%)? AN N M/A

### LEVEL IV ONLY:

Was a midrange cyanide standard distilled?

K N K AN N

Were recalculated results acceptable? See Level IV Initial and Continuing Calibration Recalculation Worksheet for recalculations. Are all correlation coefficients 20.995?

	ASSOCIATED SAMPLES	Qualification of Data
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## VALIDATION FINDINGS WORKSHEET **ICP Interference Check Sample**

Reviewer. MG Page: Lof 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". <u>Y W N/A</u> Were the AB solution percent recoveries (%R) within the control limits of 80-120% ?

LEVEL IV ONLY:

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

	Date	ICS Identification	Analyte	Finding	Associated Samples	Qualifications
-	3-21-13	(en: II) SASS I	As	(001-08) 501		K dets / P
			C r			
			Co	() () ()		
			Cu	1,23 ( )		
			!N	135 ( )		
		~	>	( <b>↑</b> ) h e	•	~
-	a da a ana anta an anta ana					
	-					
ō	Comments:					
			1. v.			

ICS.4SW

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LDC # 39656 A4

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

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

# 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)						
TCV	ICP/MS (Initial calibration)	Co	41.33	40.0	103.0	103.1	Y
	CVAA (Initial calibration)						<b></b>
	ICP (Continuing calibration)						
1210 CCV	ICP/MS (Continuing calibration)	١	38.87	40.0	97.2	97.2	>
	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

Page: 1 of 1 Reviewer: AG 2nd 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:

 $RPD = \underline{|S-D|} \times 100$ (S+D)/2

S = Original sample concentration D = Duplicate sample concentration Where,

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

%D = ||-SDR| x 100

Where, 1 = 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)
LCSAB	ICP interference check	As	AS 25.06 (mg/)	(7/gm) 08	125	125	<b>`</b>
1752 LCS	Laboratory control sample	٩Ş	19.00	(palken) co.ce (palken)	95	95	1
ļ	Matrix spike		(ssr.sr)	s 	l	I	ł
	Duplicate	١	1	)			
)	ICP serial dilution	١	J	•	1	)	l

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.



### VALIDATION FINDINGS WORKSHEET **Sample Calculation Verification**

Page:_	<u> </u>
Reviewer:	MG
2nd reviewer:	R

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

Pleas	e see
$(\underline{\mathbb{N}}N)$	N/A
ΜN Ν	N/A
$\overline{C}$ N	N/A
$\underline{\mathbf{W}}$	N/A

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

Detected analyt	e results for	# 1, M.	n	were recalculated and verified using the following
equation:				
Concentration =	(RD)(FV)(Dil)		Recalculation:	

		(11. 00.)
RD	=	Raw data concentration
FV	=	Final volume (ml)
in. Vol.	=	Initial volume (ml) or weight (0
Dil	=	Dilution factor

RD = FV = in. Vol. = Dil =	(In. Vol.) Raw data concentratic Final volume (ml) Initial volume (ml) or v Dilution factor		(0.500L)( .90)	<u>10)</u> = 193	8.7 <u>mg</u> or	mg Kq
#	Sample ID	Analyte	Reported Concentration ( <sup>Mg</sup> /kg	Calculated Concentration ( <sup>M</sup> g/Kq)	Acceptable (Y/N)	
(		AI	4500	4500	Ý	
		As	9.6	9.6	1	1
		ßa	10	10		
		Ca	320	320		1
		Cr	120	120		
		Co	13	13		1
		Cu	220	220		
		Cu Fe	430000	430000		
		Ръ	46	46		
		Mq	1500	1500		
		M^	1900	1900		
		Hq	0.086	0.086		
		N;	41	41		
		К	190	190		
		Na	140	140		
		V	5.4	5.4		
		Zn	43	43		
		·				

Note:



5405 Twin Knolls Road, Suite 1 • Columbia, MD 21045 • ph: 410.740.1911 • fax: 410.740.3299 • www.cgs.us.com

August 29, 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-12-0788.07

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 sample analyses that were validated include analyses of water samples collected from domestic supply wells at residences near the former Green Valley Citgo from June 21 through July 11, 2013. The well water 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 reviewed following the IM2 guidelines for inorganics, as described in the *EPA Region III Innovative Approaches to Data Validation (June 1995)*. The attached table summarizes the qualified sample results, defines the qualifiers, and gives the reason for the qualifier. The hexavalent chromium sample results listed on the table were qualified due to a matrix spike and matrix spike duplicate sample pair with percent recoveries that were below the acceptable limit. For detected analytes, this indicates that the reported value may be biased low, and for analytes not detected, it indicates that the detection limit is probably higher than what was reported. One dissolved lead sample result was greater than the acceptable limit. This indicates that the detection limit for that sample result is an estimated value.

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.

Sean P. Daniel Operations Manager

Attachments:

Data Validation Results Summary Laboratory Data Consultants, Data Validation Reports: 30160C4/30160C6/ 30160D4/30160D6/ 30160E4/30160E6/30160F4/30160F6

### 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 (µg/L)	Flagged Analysis	Validation Qualifiers	Reason for Qualifier
3998Farm-POU	0.020 U	Method 218.7 Hexavalent Chromium	UL / A	1
3998Farm-PT1	0.024	Method 218.7 Hexavalent Chromium	L/A	1
3998Farm-PT1DB	0.025	Method 218.7 Hexavalent Chromium	L/A	1
3998Farm-PT2	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3998Farm-PT3	0.020 U	Method 218.7 Hexavalent Chromium	UL / A	1
3998Farm-PT4	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3998Farm-WP1	0.023	Method 218.7 Hexavalent Chromium	L/A	1
3998Farm-FB	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-WP3	1.0 U	Method 200.8 Dissolved Lead	UJ / P	2
3740Blueberry-POU	0.020 U	Method 218.7 Hexavalent Chromium	UL / A	1
3740Blueberry-PT1	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-PT/DB	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-PT2	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-PT3	0.020 U	Method 218.7 Hexavalent Chromium	UL / A	1
3740Blueberry-PT4	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-WP1	0.020 U	Method 218.7 Hexavalent Chromium	UL / A	1
3740Blueberry-WP2	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-WP3	0.020 U	Method 218.7 Hexavalent Chromium	UL/A	1
3740Blueberry-FB	0.020 U	Method 218.7 Hexavalent Chromium	UL / A	1

Table Notes:

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.

UJ - Indicates the compound or element was analyzed for but not detected. The sample detection limit is an estimated value.

A -Indicates the finding is based upon technical validation criteria.

**P**-Indicates the finding is related to a protocol/contractual deviation.

U - Analyte Not Detected Above Specified Reporting Limit

**Bold** - Detected analyte concentration

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

1 - Matrix spike/Matrix spike duplicate (Percent recoveries (%R) of 76.3%/74.7% are below acceptable limit of 85%)

2 – Internal standards (%R of 138.669% is greater than the acceptable limit of 125%)



Chesapeake GeoScience, Inc. 5405 Twin Knolls Rd, Suite 1 Columbia, MD 21045 ATTN: Mr. Sean P. Daniel August 13, 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 August 2, 2013. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### LDC Project # 30160:

SDG #

### **Fraction**

ECL028637/ECL028662 Metals ECL028907 ECL029474 ECL029318 ECL029507 ECL029324

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 Geng Project Manager/Senior Chemist

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	IV	L	_DC #3	016	<b>i0 (</b>	Che	esa	pea	ake	Ge	oS	cie	nce	s, l	nc	- Co	olu	mb	ia, I	MD	/ G	Gree	en \	/all	ey	Cit	go	Pro	ojeo	ct)	51. D					
LDC	SDG#	DATE REC'D	(3) DATE DUE	Cr, (20	, Pb 0.8)		ss Pb 0.8)	Ci	iss rVI 8.7)																	-										
Matrix:	Water/Soil-Prod	luct		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
A	ECL028637/ ECL028662	08/02/13	08/23/13	12	0	12	0	12	0																											
в	ECL028907	08/02/13	08/23/13	7	Ô.	7	0	7	0			3																								$\neg$
С	ECL029474	08/02/13	08/23/13	8	0			8	Ó																											$\neg$
D	ECL029318		08/23/13	2422 11982			0	10																												
E	ECL029507		08/23/13		0			10															_													$\uparrow$
F	ECL029324	08/02/13	08/23/13	10	0			10																												
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Total	A/PG			57	0	57	0	57	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0 1

### LDC Report# 30160C4

### Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	July 9, 2013
LDC Report Date:	August 9, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.

### Sample Delivery Group (SDG): ECL029474

### Sample Identification

3998 Farm-POU Total 3998 Farm-POU Dissolved 3998 Farm-PT1 Total 3998 Farm-PT1 Dissolved 3998 Farm-PT1DB Total 3998 Farm-PT1DB Dissolved 3998 Farm-PT2 Total 3998 Farm-PT2 Dissolved 3998 Farm-PT3 Total 3998 Farm-PT3 Dissolved 3998 Farm-PT4 Total 3998 Farm-PT4 Dissolved 3998 Farm-WP1 Total 3998 Farm-WP1 Dissolved 3998 Farm-FB Total 3998 Farm-FB Dissolved 3998 Farm-PT4 TotalMS 3998 Farm-PT4 TotalDUP 3998 Farm-POU TotalMS 3998 Farm-POU TotalDUP

1

### 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.

2

### 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.

Samples 3998 Farm-FB Total and 3998 Farm-FB Dissolved were 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 analysis was performed by the laboratory. The analysis criteria were met.

### 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 3998 Farm-PT1 Total and 3998 Farm-PT1DB Total and samples 3998 Farm-PT1 Dissolved and 3998 Farm-PT1DB Dissolved were identified as field duplicates. No chromium or lead contaminants were detected in any of the samples with the following exceptions:

	Concentrat	tion (ug/L)	
Analyte	3998 Farm-PT1 Total	3998 Farm-PT1DB Total	RPD
Lead	10.2	6.7	41

	Concentra	tion (ug/L)	
Analyte	3998 Farm-PT1 Dissolved	3998 Farm-PT1DB Dissolved	RPD
Lead	4.7	4.7	0

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDG ECL029474

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL029474

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL029474

No Sample Data Qualified in this SDG

5

LDC #: <u>30160C4</u>	VALIDATION COMPLETENESS WORKSHEET	Date: 8/9/13
SDG #: <u>ECL029474</u>	Level IV	Page: of 1
Laboratory: Enviro-Che	m Laboratories, Inc.	Reviewer:
		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
<u>l.</u>	Technical holding times	A	Sampling dates: 7/9/13
11.	ICP/MS Tune	A	
-111.	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	$\mathcal{N}$	Not required
VI.	Matrix Spike Analysis	A	ms
VII.	Duplicate Sample Analysis	A	Dip
VIII.	Laboratory Control Samples (LCS)	A	LES
IX.	Internal Standard (ICP-MS)	A	
Х.	Furnace Atomic Absorption QC	$\mathcal{N}$	
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	(3,5)(4,6)
XV	Field Blanks	NO	1=B=15,16

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:

Va	ilidated Samples:		water		ana
1	3998 Farm-POU Total	11	3998 Farm-PT4 Total	21	31
2	3998 Farm-POU Dissolved	12	3998 Farm-PT4 Dissolved	22	32
3	3998 Farm-PT1 Total	13	3998 Farm-WP1 Total	23	33
4	3998 Farm-PT1 Dissolved	14	3998 Farm-WP1 Dissolved	24	34
5	3998 Farm-PT1DB Total	15	3998 Farm-FB Total	25	35
6	3998 Farm-PT1DB Dissolved	16	3998 Farm-FB Dissolved	26	36
7	3998 Farm-PT2 Total	17	3998 Farm-PT4 TotalMS	27	37
8	3998 Farm-PT2 Dissolved	18	3998 Farm-PT4 TotalDUP	28	38
9	3998 Farm-PT3 Total	19	(ms)ms	29	39
10	3998 Farm-PT3 Dissolved	20	1 Dig	30	40

Notes:\_\_

LDC #: 30160C4



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.		`		
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?	$\langle \rangle$			
Were the proper number of standards used?		ŕ		
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?		ر		
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.		$\langle$		
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 Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			-	<b>,</b>
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				
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%?				<u> </u>
Was there evidence of negative interference? If yes, professional judgement will be used to gualify 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?	$\langle$			
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				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	$\sim$			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	$\langle$			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	$\square$			
Target analytes were detected in the field duplicates.	_			
XV. Field blanks				
Field blanks were identified in this SDG.	$\frown$			
Target analytes were detected in the field blanks.				

LDC #: 30160C4

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-16		Al, Sb, As, Ba, Be, 🗭 Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,
		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, Sn, Ti,
3: 17.18		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Nì, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		Analysis Method
CP		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, Sn, Ti,
CP-MS		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, Sn, Ti,
FAA		Al, Sh, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti

LDC#: 30160C4

### VALIDATION FINDINGS WORKSHEET Field Duplicates



### METHOD: Metals (EPA Method 6010B/7000)

	Concentra		
Analyte	3	5	RPD
Lead	10.2	6.7	41

	Concentra		
Analyte	4	6	RPD
Lead	4.7	4.7	0

\LDCFILESERVER\Validation\FIELD DUPLICATES\FD\_inorganic\30160C4.wpd

LDC #: 30/60CC

### VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification



### 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:

 %R = Found\_x 100
 Where,
 Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

 True
 True = concentration (in ug/L) of each analyte in the ICV or CCV source

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
ŦĊV	ICP/MS (Initial calibration)	PD	97,68	100	98	98	$\mathcal{M}$
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
COV ·	ICP/MS (Continuing calibration)	$\langle \langle \rangle$	1934	200	97	97	T
	CVAA (Continuing calibration)	Ì					
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

Comments: <u>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.</u>
# 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:

 %R = Found\_x 100
 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
 True = Concentration of each analyte in the source.

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

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

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

%D = <u>|I-SDR|</u> 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)
$\sim$	ICP interference check						
LCS	Laboratory control sample	$\sim$	45,8	50	91,6	91,5	Ý
19	Matrix spike	Ċ	(SSR-SR) 47, ()	50	94,0	93,7	
K	Duplicate	C	36,5	44,0	18.6	18,6	
$\sim$	ICP serial dilution						

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



# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	OR
2nd reviewer:	$\sim$

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

Please X N I Y N I Y N I	N/A Are results v	been reported a	and calculate ted range of	ed correctly? the instruments	s and within the line	ear range of the IC	P?
Detect equation	ed analyte results for _ on:				were recalcu	lated and verified	using the following
Concent	tration = $\frac{(RD)(FV)(Dil)}{(ln. Vol.)}$		fromra	Recalculation:	<u>-</u>		
RD FV In. Vol. Dil	<ul> <li>Raw data conce</li> <li>Final volume (m</li> <li>Initial volume (n</li> <li>Dilution factor</li> </ul>			3= 10.1 4= 4.6	80 vg/L 090 vg/L		
#	Sample ID		Analyte		Reported Concentration ( <i>Mg</i> 1(–)	Calculated Concentration	Acceptable (Y/N)
	3			Pb	10.2	10.2	
	Ч			Pb	4,7	4.7	Y
			·				
				· · · · · · · · · · · · · · · · · · ·			

Note:\_\_\_\_

## LDC Report# 30160C6

# Laboratory Data Consultants, Inc. Data Validation Report

**Dissolved Chromate as Chromium** 

Project/Site Name:	Green Valley Citgo Project

Collection Date: July 9, 2013

LDC Report Date: August 9, 2013

Matrix: Water

Parameters:

Validation Level: EPA Region III, Level IM2

Laboratory: Enviro-Chem Laboratories, Inc.

Sample Delivery Group (SDG): ECL209474

### Sample Identification

3998 Farm-POU Dissolved 3998 Farm-PT1 Dissolved 3998 Farm-PT1DB Dissolved 3998 Farm-PT2 Dissolved 3998 Farm-PT3 Dissolved 3998 Farm-PT4 Dissolved 3998 Farm-WP1 Dissolved 3998 Farm-FB Dissolved 3998 Farm-POU DissolvedMS 3998 Farm-POU DissolvedMSD

### 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.

Sample 3998 Farm-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 with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
3998 Farm-POU DissolvedMS/MSD (All samples in SDG ECL029474)	Dissolved chromate as chromium	76.3 (85-115)	74.7 (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 samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

# **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 3998 Farm-PT1 Dissolved and 3998 Farm-PT1DB Dissolved were identified as field duplicates. No dissolved chromate as chromium was detected in any of the samples with the following exceptions:

	Concentratio		
Analyte	3998 Farm-PT1 Dissolved	3998 Farm-PT1DB	RPD
Dissolved chromate as chromium	0.024	0.025	4

## Green Valley Citgo Project Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL209474

SDG	Sample	Analyte	Flag	A or P	Reason
ECL209474	3998 Farm-POU Dissolved 3998 Farm-PT1 Dissolved 3998 Farm-PT1DB Dissolved 3998 Farm-PT2 Dissolved 3998 Farm-PT3 Dissolved 3998 Farm-PT4 Dissolved 3998 Farm-WP1 Dissolved 3998 Farm-FB Dissolved	Dissolved chromate as chromium	L (all detects) UL (all non-detects)	А	Matrix spike/Matrix spike duplicate (%R)

# **Green Valley Citgo Project**

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL209474

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Dissolved Chromate as Chromium - Field Blank Data Qualification Summary - SDG ECL209474

No Sample Data Qualified in this SDG

LDC #: <u>30160C6</u> VA	ALIDATION COMPLETENESS WORKSHEET	Date: <u>5/</u> 9/
SDG #:	Level IV	Page: <u>`of</u>
Laboratory: Enviro-Chem Laborat	<u>ories, Inc.</u>	Reviewer:
		2nd Reviewer:

# 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
1.	Technical holding times	A	Sampling dates: 7/9/13
11	Initial calibration	A	
111.	Calibration verification	A	
IV	Blanks	A,	
v	Matrix Spike/Matrix Spike Duplicates	SW	msp
VI.	Duplicates	$\sim$	•
VII.	Laboratory control samples	A	LCS
VIII.	Sample result verification	K	
IX.	Overall assessment of data	Â	
<b>X</b> .	Field duplicates	SW	(2,3)
L XI	Field blanks	NO	F8=8

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:

water

\_\_\_\_\_

1	3998 Farm-POU Dissolved	11	21	31
2	3998 Farm-PT1 Dissolved	12	22	32
3	3998 Farm-PT1DB Dissolved	13	23	33
4	3998 Farm-PT2 Dissolved	14	24	34
5	3998 Farm-PT3 Dissolved	15	25	35
6	3998 Farm-PT4 Dissolved	16	26	36
7	3998 Farm-WP1 Dissolved	17	27	37
8	3998 Farm-FB Dissolved	18	28	38
9	3998 Farm-POU DissolvedMS	19	29	39
10	3998 Farm-POU DissolvedMSD	20	30	40

Notes:\_\_\_





Method:Inorganics (EPA Method See cover)					
Validation Area	Yes	No	NA	Findings/Comments	
I. Technical holding times					
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?	$\square$				
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)				f	
III. Blanks					
Was a method blank associated with every sample in this SDG?	$\mathcal{L}$				
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?	$\langle \rangle$				
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?					

C#:\_\_\_\_\_O160CG

# 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.	/					
IX. Field duplicates						
Field duplicate pairs were identified in this SDG.	<					
Target analytes were detected in the field duplicates.	$\frown$					
X. Field blanks						
Field blanks were identified in this SDG.						
Target analytes were detected in the field blanks.						

LDC #:\_\_\_\_\_\_\_\_\_\_\_\_\_

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page:of
Reviewer: <u>02</u>
2nd Reviewer:

METHOD: Inorganics, EPA Method

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

<u>V N N/A</u> Was a matrix spike analyzed for each matrix in this SDG?

Y/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.

(2) N N/A Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$ 35% for soil samples?

LEVEL IV ONLY:

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

#	MS/MSD ID	Matrix	Analyte	MS <u>%Recovery</u> 76,3(85-115)	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
	9/10	W	Chromate	76.3(85-115)	74,7(85-115)		AII	3 L/UL/A
					_			•
					-			
Ш								

Comments:\_\_\_\_\_

# LDC#<u>30160C6</u>

# VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:of	
Reviewer:	
2nd Reviewer:	

Inorganics: Method See Cover

	Concentra	tion (mg/L)	
Analyte	2	3	RPD
Chromate	0.024	0.025	4

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LDC #: \_30160C6

### Validation Findings Worksheet Initial and Continuing Calibration Calculation Verification

	1		١
Page:	`	of	·
Reviewe	r:_(	Q	
2nd Revie	ewe	er:_1	$\sim$

Method: Inorganics, Method \_\_\_\_\_ QVC.つ

The correlation coefficient (r) for the calibration of  $\underline{CrO_4}$  was recalculated. Calibration date:  $\underline{GZY/R}$ 

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

%R = Found 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

						Recalculated	Reported	Acceptable
Type of analysis	An	nalyte	Standard	Conc. (mg/l)	uv*s	r or r <sup>2</sup>	r or r <sup>2</sup>	(Y/N)
Initial calibration			s1	0.02	0.0506			
			s2	0.05	0.1208	1.0000	1.0000	
			s3	0.1	0.2176			
	Ca	<u>)</u> , ,	s4	0.25	0.544			(
		-4	s5	0.5	1.0689			
			s6	1	2.1432			
		]	s7	5	10.6889			
Calibration verification			CCV	l	1.0533	105	105	Ŷ
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.\_\_\_\_\_



# VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Inorganics, Method Secon

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

 %R = Found\_x 100
 Where,
 Found =
 concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

 True
 Found = SSR (spiked sample result) - SR (sample result).

 True = concentration of each analyte in the source.

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

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

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated	Reported %R / RPD	Acceptable (Y/N)
QLS	Laboratory control sample	Chromate	0.973	0,02	87	_	
9	Matrix spike sample		(SSR-SR) (),747	l	74,7	74.7	
9/10	Duplicate sample		0.747	0.763	Э.1	2.1	

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.

TOTCLC.6

LDC #: \_\_\_\_OLGOCU

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification



METHOD: Inorganics, Method See OVE

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>N\_N/A</u> Have results been reported and calculated correctly?

N N/A 'N N/A

Are results within the calibrated range of the instruments?

Are all detection limits below the CRQL?

Chomate \_\_\_\_reported with a positive detect were

Compound (analyte) results for recalculated and verified using the following equation:

Concentration =

Recalculation:

y= 2.1346×+0.0089

 $\frac{0.06 - 0.0089}{2.1346} = 0.0239 ug/L$ 

#	Sample ID	Analyte	Reported Concentration (US(	Calculated Concentration $(\mathcal{Mg}^{(-)})$	Acceptable (Y/N)
	6	GOy	0,024	0.024	4
			· · · · · · · · · · · · · · · · · · ·		
		· · · · · · · · · · · · · · · · · · ·			
			· · · · · · · · · · · · · · · · · · ·		
	····				
				· · · · · · · · · · · · · · · · · · · ·	

Note:

# Laboratory Data Consultants, Inc. Data Validation Report

Chromium & Lead

**Project/Site Name:** 

Green Valley Citgo Project

Collection Date: June 21, 2013

LDC Report Date: August 9, 2013

Matrix: Water

Parameters:

Validation Level: EPA Region III, Level IM2

Laboratory: Enviro-Chem Laboratories, Inc.

Sample Delivery Group (SDG): ECL029318

### Sample Identification

11713 Serene-POU Total 11713 Serene-POU Dissolved 11713 Serene-PT1 Total 11713 Serene-PT1 Dissolved 11713 Serene-PT1DB Total 11713 Serene-PT1DB Dissolved 11713 Serene-PT2 Total 11713 Serene-PT2 Dissolved 11713 Serene-PT3 Total 11713 Serene-PT3 Dissolved 11713 Serene-PT4 Total 11713 Serene-PT4 Dissolved 11713 Serene-FB Total 11713 Serene-FB Dissolved 11713 Serene-WP1 Total 11713 Serene-WP1 Dissolved 11713 Serene-WP2 Total 11713 Serene-WP2 Dissolved 11713 Serene-WP3 Total 11713 Serene-WP3 Dissolved

11713 Serene-FB TotalMS 11713 Serene-FB TotalDUP 11713 Serene-PT1 TotalMS 11713 Serene-PT1 TotalDUP

## Introduction

This data review covers 24 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.

2

# 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.

Samples 11713 Serene-FB Total and 11713 Serene-FB Dissolved were 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

Samples 11713 Serene-PT1 Total and 11713 Serene-PT1DB Total and samples 11713 Serene-PT1 Dissolved and 11713 Serene-PT1DB Dissolved were identified as field duplicates. No chromium or lead contaminants were detected in any of the samples with the following exceptions:

	Concentration (ug/L)		
Analyte	11713 Serene-PT1 Total	11713 Serene-PT1DB	RPD
Chromium	10.5	6.5	47

	Concentration (ug/L)			
Analyte	11713 Serene-PT1 Dissolved	11713 Serene-PT1DB Dissolved	RPD	
Chromium	4.4	4.6	4	

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDG ECL029318

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL029318

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL029318

No Sample Data Qualified in this SDG

LDC #:	30160D4	VALIDATION COMPLETENESS WORKSHEET	Date: 8/9/1
SDG #:	ECL029318	Level IV	Page: <u>of</u>

Laboratory: Enviro-Chem Laboratories, Inc.

Reviewer:\_ 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
1.	Technical holding times	A	Sampling dates: 6/2//13
11	ICP/MS Tune	A	
	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	$\overline{N}$	Not required
VI.	Matrix Spike Analysis	A	ms
VII.	Duplicate Sample Analysis	A	Dep
VIII.	Laboratory Control Samples (LCS)	A	LES
IX.	Internal Standard (ICP-MS)	A	
<b>X</b> .	Furnace Atomic Absorption QC	$\sim$	
<u>XI.</u>	ICP Serial Dilution	N	
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	5₩	(3,5)(4,6)
xv	Field Blanks	ND	FB=13,14

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: wall

1	11713 Serene-POU Total	11	11713 Serene-PT4 Total	21 -	11713 Serene-FB TotalMS	31	
2	11713 Serene-POU Dissolved	12	11713 Serene-PT4 Dissolved	22	11713 Serene-FB TotalDUP	32	
3	11713 Serene-PT1 Total	13	11713 Serene-FB Total	23	(#3) ms	33	
4	11713 Serene-PT1 Dissolved	14	11713 Serene-FB Dissolved	24	I DUP	34	
5	11713 Serene-PT1DB Total	15	11713 Serene-WP1 Total	25	AB	35	
6	11713 Serene-PT1DB Dissolved	16	11713 Serene-WP1 Dissolved	26		36	
7	11713 Serene-PT2 Total	17	11713 Serene-WP2 Total	27		37	
8	11713 Serene-PT2 Dissolved	18	11713 Serene-WP2 Dissolved	28		38	
9	11713 Serene-PT3 Total	19	11713 Serene-WP3 Total	29		39	
10	11713 Serene-PT3 Dissolved	20	11713 Serene-WP3 Dissolved	30		40	

Notes:\_



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.				
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?	$\langle$			
Were the proper number of standards used?	$\square$			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?				
Were all initial calibration correlation coefficients $\geq 0.995$ ?				
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?				f
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?	$\int$			
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

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)			_	^ 
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)?				
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?			$\sim$	·
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?	$\overline{}$			
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.	~			
Target analytes were detected in the field blanks.				



# VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

Sample ID	_Matrix	Target Analyte List (TAL)
1-20		
1.00		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, Sn, Ti,
Guad 01		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, Sn, Ti,
QC 21.22		Al, Sb, As, Ba, Be, Cd, Ca, Čr) Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ní, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
ICP-MS		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, Sn, Ti,
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 Sn Ti

LDC#:<u>30160D4</u>

# VALIDATION FINDINGS WORKSHEET Field Duplicates



METHOD: Metals (EPA Method 6010B/7000)

	Concentra	ation (ug/L)	
Analyte	3	5	RPD
Chromium	10.5	6.5	47

	Concentra	Concentration (ug/L)				
Analyte	4	6	RPD			
Chromium	4.4	4.6	4			

\\LDCFILESERVER\Validation\FIELD DUPLICATES\FD\_inorganic\30160D4.wpd

LDC #: 30160

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification



#### 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:

 %R = Found\_x 100
 Where,
 Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

 True
 True = concentration (in ug/L) of each analyte in the ICV or CCV solution

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						-
ŦĊV	ICP/MS (Initial calibration)	Cr	100.1	100	100	100	4
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
CCV)	ICP/MS (Continuing calibration)	PD	1993	300		100	Ĭ
	CVAA (Continuing calibration)						,
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

Comments: <u>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.</u>

LDC #: 30160

# 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:

 %R = Found\_x 100
 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
 True = Concentration of each analyte in the source.

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

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

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

 %D = <u>|I-SDR|</u> x 100
 Where, I = Initial Sample Result (mg/L)

 I
 SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

			Found / S / I	True / D / SDR (units)	Recalculated	Reported	Acceptable
Sample ID	Type of Analysis	Element	(units)		%R / RPD / %D	<u>%R / RPD / %D</u>	(Y/N)
N	ICP interference check						
LCS	Laboratory control sample	Cr	54,7	50	109.4	1091,3	$\gamma$
20	Matrix spike		(SSR-SR) 45,6	50	91,2	89,9	
24	Duplicate		9,9	10,5	5,9	5,1	J
$\sim$	ICP serial dilution						

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



# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:of	
Reviewer: <u>CR</u>	
2nd reviewer:	

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

e see qua <u>N/A</u> <u>N/A</u> <u>N/A</u>	Have results Are results w	been reported a vithin the calibra	and calculated of the	correctly?	·		
ted analy on:	/te results for _		C	X	were recalcu	lated and verified	using the following
tration =	<u>(RD)(FV)(Dil)</u> (In, Vol.)		Re from (	calculation: andate	~ ,		
2 2 2 2	Final volume (m	l)	3= 4=	10.46	, ugl L Ingl		
S	ampie ID		Analyte		Reported Concentration ( <i>M</i> &1L)	Calculated Concentration	Acceptable (Y/N)
	3		(	9	10,5	10.5	Y
	4			9	4,4	4.4	Ч
						· · · · · · · · · · · · · · · · · · ·	
			······				
	<u>N/A</u> <u>N/A</u> ted analy on: tration =	N/A       Have results         N/A       Are results w         N/A       Are results w         N/A       Are results w         N/A       Are all detect         ted analyte results for          on:          tration =       (RD)(FV)(Dil) (In. Vol.)         =       Raw data conce         =       Final volume (m         =       Initial volume (m         =       Dilution factor         Sample ID	N/A       Have results been reported in the calibration         N/A       Are results within the calibration         N/A       Are all detection limits below         ted analyte results for	N/A       Have results been reported and calculated of N/A         N/A       Are results within the calibrated range of the Are all detection limits below the CRDL?         ted analyte results for	N/A       Have results been reported and calculated correctly?         N/A       Are results within the calibrated range of the instruments         N/A       Are all detection limits below the CRDL?         ted analyte results for $(n)$ tration =       (RD)(FV)(Dil) (In. Vol.)       Recalculation: $\mbox{Com data}$ =       Raw data concentration = $\mbox{Final volume (ml)}$ $\mbox{3} = 10.46$ =       Dilution factor $\mbox{3} = 10.46$ Sample ID       Analyte	N/A       Have results been reported and calculated correctly?         N/A       Are results within the calibrated range of the instruments and within the line         N/A       Are results within the calibrated range of the instruments and within the line         N/A       Are results within the calibrated range of the instruments and within the line         N/A       Are results within the calibrated range of the instruments and within the line         N/A       Are all detection limits below the CRDL?         ted analyte results for $\end{were}$ recalculation:         on:       Recalculation:         tration =       (RD)(FV)(Dil)       Recalculation:         (In. Vol.)       Recalculation: $schward$	N/A       Are results within the calibrated range of the instruments and within the linear range of the IC         N/A       Are all detection limits below the CRDL?         Are all detection limits below the CRDL?       were recalculated and verified         ted analyte results for $\mathcal{M}$ were recalculated and verified         on:       Recalculation:       were recalculated and verified         tration =       (RD)(FV(Dil) (In. Vol.)       Recalculation:       Gran Can data         =       Raw data concentration $\mathcal{F}$ can data $\mathcal{F}$ can data         =       Initial volume (ml) $\mathcal{F}$ can data $\mathcal{F}$ can data         =       Dilution factor $\mathcal{F}$ can data $\mathcal{F}$ can data $\mathcal{F}$ can can data $\mathcal{F}$ can data $\mathcal{F}$ can data         =       Dilution factor $\mathcal{F}$ can data $\mathcal{F}$ concentration         =       Dilution factor $\mathcal{F}$ can data $\mathcal{F}$ concentration $\mathcal{F}$ concentration         Sample ID       Analyte       Reported       Calculated       Concentration $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ 3 $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ $\mathcal{F}$ 3

Note:\_\_\_\_

### **LDC Report#** 30160D6

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	June 21, 2013
LDC Report Date:	August 9, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.

Sample Delivery Group (SDG): ECL029318

# **Sample Identification**

11713 Serene-POU Dissolved 11713 Serene-PT1 Dissolved 11713 Serene-PT1DB Dissolved 11713 Serene-PT2 Dissolved 11713 Serene-PT3 Dissolved 11713 Serene-PT4 Dissolved 11713 Serene-FB Dissolved 11713 Serene-WP1 Dissolved 11713 Serene-WP2 Dissolved 11713 Serene-WP3 Dissolved 11713 Serene-WP1 DissolvedMS 11713 Serene-WP1 DissolvedMS

1

## Introduction

This data review covers 12 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.

2

# 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 11713 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 samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

#### 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 11713 Serene-PT1 Dissolved and 11713 Serene-PT1DB Dissolved were identified as field duplicates. No dissolved chromate as chromium was detected in any of the samples with the following exceptions:

	Concentrat	ion (mg/L)	
Compound	11713 Serene-PT1 Dissolved	11713 Serene-PT1DB Dissolved	RPD
Dissolved chromate as chromium	0.111	0.112	1

Green Valley Citgo Project

Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL029318

No Sample Data Qualified in this SDG

**Green Valley Citgo Project** 

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL029318

No Sample Data Qualified in this SDG

Green Valley Citgo Project Dissolved Chromate as Chromium - Field Blank Data Qualification Summary -SDG ECL029318

No Sample Data Qualified in this SDG

LDC #:_	30160D6	VALIDATION COMPLETENESS WORKSHEET	Date: 8/9/13
SDG #:_	ECL029318	Level IV	Page: \ of \
Laborato	ory: Enviro-Chem La	boratories, Inc.	Reviewer: 2
			2nd Reviewer:

#### 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
1.	Technical holding times	A	Sampling dates: 6/2/13
11	Initial calibration	A	
- 111.	Calibration verification	A	
١V	Blanks	A	
V	Matrix Spike/Matrix Spike Duplicates	Ð	msp
VI.	Duplicates	N	
VII.	Laboratory control samples	A	LCS
VIII.	Sample result verification	A	1
IX.	Overall assessment of data	A	
Х.	Field duplicates	SW	(2,3)
	Field blanks	ND	FB=7

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

watch

ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

1	11713 Serene-POU Dissolved	11	11713 Serene-WP1 DissolvedMS	21	31	
2	11713 Serene-PT1 Dissolved	12	11713 Serene-WP1 DissolvedMSD	22	32	
3	11713 Serene-PT1DB Dissolved	13		23	33	
4	11713 Serene-PT2 Dissolved	14		24	34	
5	11713 Serene-PT3 Dissolved	15		25	35	
6	11713 Serene-PT4 Dissolved	16		26	36	
7	11713 Serene-FB Dissolved	17		27	37	
8	11713 Serene-WP1 Dissolved	18		28	38	
9	11713 Serene-WP2 Dissolved	19		29	39	
10	11713 Serene-WP3 Dissolved	20		30	40	

Notes:\_\_\_\_\_





Method: Inorganics (EPA Method See cover)						
Validation Area	Yes	No	NA	Findings/Comments		
I. Technical holding times						
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?	$\square$					
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?						
Were titrant checks performed as required? (Level IV only)			<u> </u>			
Were balance checks performed as required? (Level IV only)				<u> </u>		
III. Blanks	<del></del>	r	·	······		
Was a method blank associated with every sample in this SDG?	$\leq$					
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?	$\square$					
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?		$\square$				
Were the performance evaluation (PE) samples within the acceptance limits?						



# VALIDATION FINDINGS CHECKLIST



Validation Area		es No NA Findings/Con		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.	$\langle$				
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.	1				
Target analytes were detected in the field blanks.					

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LDC#<u>30160D6</u>

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Inorganics: Method\_See Cover\_

	Concentration (mg/L)		
Analyte	2	3	RPD
Chromate	0.111	0.112	1

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LDC #: 3016006

## Validation Findings Worksheet Initial and Continuing Calibration Calculation Verification

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Page: of
Reviewer: <u>C</u>
2nd Reviewer:

Method: Inorganics, Method \_\_\_\_\_\_\_\_

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

%R = <u>Found X 100</u>

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

						Recalculated	Reported	Acceptable
Type of analysis	Ana	lyte	Standard	Conc. (mg/l)	uv*s	r or r <sup>2</sup>	r or r <sup>2</sup>	(Y/N)
Initial calibration			s1	0.02	0.0506			
			s2	0.05	0.1208	1.0000	1.0000	
			s3	0.1	0.2176			
	(Â	)4	s4	0.25	0.544			Ý
	0	·	s5	0.5	1.0689			
	1	l	s6	11	2.1432			
			s7	5	10.6889			
Calibration verification			CCV	5	4,9698	99	99	
Calibration verification			T	0,02	0.0219	110	1(()	
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.

# VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Inorganics, Method Second

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

%R = Found x 100 Where, True 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.

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

 $\begin{array}{cccc} \text{RPD} = \underline{|S-D|} & x \ 100 & \text{Where,} & S = & & \text{Original sample concentration} \\ & (S+D)/2 & & D = & & \text{Duplicate sample concentration} \end{array}$ 

Sample ID	Type of Analysis	Ele	ment	Found / S (units)	True / D (units)	Recalculated	Reported	Acceptable (Y/N)	
QCS	Laboratory control sample	Chear	nete 1	1.8226	2	91			
- 11	Matrix spike sample	Ø		(SSR-SR) 0,998 +,033	Ø9,	100	100		
WIN	Duplicate sample	, Y	Y	1.03	0.998	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.



### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification



METHOD: Inorganics, Method See. Over

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

Have results been reported and calculated correctly? Are results within the calibrated range of the instruments?

N N/A <u>/N\_N/A</u>

Are all detection limits below the CRQL?

\_\_\_\_\_reported with a positive detect were homate Compound (analyte) results for recalculated and verified using the following equation:

Concentration =

Recalculation:

y= 2.1346×+0.0089

 $\frac{0.246 - 0.0089}{2.1346} = 0.1111 \text{ Lg/L}$ 

#	Sample ID	Analyte	Reported Concentration ( <i>US</i> JL)	Calculated Concentration (ℳஜ1└──)	Acceptable (Y/N)
	2	Chromate	0.111	0.111	Υ
				· · · · · · · · · · · · · · · · · · ·	
L				 	

Note:

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	July 11, 2013
LDC Report Date:	August 9, 2013
Matrix:	Water
Parameters:	Chromium & Lead
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECL029507

#### Sample Identification

3740 Blueberry - POU total 3740 Blueberry - POU dissolved 3740 Blueberry - PT1 total 3740 Blueberry - PT1 dissolved 3740 Blueberry - PT/DB total 3740 Blueberry - PT/DB dissolved 3740 Blueberry - PT2 total 3740 Blueberry - PT2 dissolved 3740 Blueberry - PT3 total 3740 Blueberry - PT3 dissolved 3740 Blueberry - PT4 total 3740 Blueberry - PT4 dissolved 3740 Blueberry - WP1 total 3740 Blueberry - WP1 dissolved 3740 Blueberry - WP2 total 3740 Blueberry - WP2 dissolved 3740 Blueberry - WP3 total 3740 Blueberry - WP3 dissolved 3740 Blueberry - FB total 3740 Blueberry - FB dissolved

3740 Blueberry - POU totalMS 3740 Blueberry - POU totalDUP 3740 Blueberry - PT4 totalMS 3740 Blueberry - PT4 totalDUP

1

#### Introduction

This data review covers 24 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.

Samples 3740 Blueberry - FB total and 3740 Blueberry - FB dissolved were 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 with the following exceptions:

Sample	Internal Standard	%R (Limits)	Analyte	Flag	A or P
3740 Blueberry - WP3 dissolved	Bismuth-209	138.669 (60-125)	Lead	J (all detects) UJ (all non-detects)	Ρ

## 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 3740 Blueberry - PT1 total and 3740 Blueberry - PT/DB total and samples 3740 Blueberry - PT1 dissolved and 3740 Blueberry - PT/DB dissolved were identified as field duplicates. No chromium or lead contaminants were detected in any of the samples with the following exceptions:

	Concentra		
Analyte	3740 Blueberry - PT1 total	3740 Blueberry - PT/DB total	RPD
Lead	32.3	41.5	25

	Concentra		
Analyte	3740 Blueberry - PT1 dissolved	3740 Blueberry - PT/DB dissolved	RPD
Lead	16.8	16.6	1

## Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDG ECL029507

SDG	Sample	Analyte	Flag	A or P	Reason
ECL029507	3740 Blueberry - WP3 dissolved	Lead	J (all detects) UJ (all non-detects)	Ρ	Internal standards (%R)

## **Green Valley Citgo Project**

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDG ECL029507

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDG ECL029507

No Sample Data Qualified in this SDG

LDC #: <u>30160E4</u>	VALIDATION COMPLETENESS WORKSHEET	Date: <u>8/8/</u> /3
SDG #: <u>ECL029507</u>	Level IV	Page: \ of \
Laboratory: Enviro-Chem La	aboratories, Inc.	Reviewer:

Reviewer: 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: 7/11/13
11.	ICP/MS Tune	A	
- 111.	Calibration	A	
iV.	Blanks	À	
V.	ICP Interference Check Sample (ICS) Analysis	$\sim$	Not regired
VI.	Matrix Spike Analysis	A	ms
VII.	Duplicate Sample Analysis	A	Ove
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	SW	
<u>X.</u>	Furnace Atomic Absorption QC	$\mathbb{N}$	
XI.	ICP Serial Dilution	N	
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	R	
XIV.	Field Duplicates	SW	(3,5)(4,6)
XV	Field Blanks	NO	FB=19,20

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:

	Water Samples.				the American Market		
1	3740 Blueberry - POU total	11	3740 Blueberry - PT4 total	21	3740 Blueberry - POU totalMS	31	
2	3740 Blueberry - POU dissolved	12	3740 Blueberry - PT4 dissolved	22	3740 Blueberry - POU total	32	
3	3740 Blueberry - PT1 total	13	3740 Blueberry - WP1 total	23	3740 Blueberry - PT4 totalMS	33	
4	3740 Blueberry - PT1 dissolved	14	3740 Blueberry - WP1 dissolved	24	Q∪Q 3740 Blueberry - PT4 total <del>MSD</del>	34	
5	3740 Blueberry - PT/DB total	15	3740 Blueberry - WP2 total	25		35	
6	3740 Blueberry - PT/DB dissolved	16	3740 Blueberry - WP2 dissolved	26		36	
7	3740 Blueberry - PT2 total	17	3740 Blueberry - WP3 total	27		37	
8	3740 Blueberry - PT2 dissolved	18	3740 Blueberry - WP3 dissolved	28		38	
9	3740 Blueberry - PT3 total	19	3740 Blueberry - FB total	29		39	
10	3740 Blueberry - PT3 dissolved	20	3740 Blueberry - FB dissolved	30		40	
N	otes:		······································				

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.	/	- -		
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 $\leq$ 5%?				
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 $\geq$ 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.	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.	$\overline{}$			
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 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)			/	n
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		<u></u>		
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?				
XI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		$\square$	-	
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.	/			
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.	_			
Target analytes were detected in the field blanks.		/		



# VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-70		Al, Sb, As, Ba, Be, Cd, Ca, C), Co, Cu, Fe(Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,
<u>. 00</u>		
QC:21-24		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		Analysis Method
СР		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, Sn, Ti,
CP-MS		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, Sn, Ti,
BEAA		ALSE AS BA BE CO CA CT CO CU FE PE Mg Mn Hg Ni K Se Ag Na TI V Zn Mo B Sn Ti

LDC #: 201606

## VALIDATION FINDINGS WORKSHEET Internal Standards (ICP-MS)

Page:of	_
Reviewer: 0	_
2nd Reviewer:	_

#### METHOD: Metals (EPA Method 200.8)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Y N/A Were all internal standard percent recoveries within 60-125% of the intensity of the internal standard in the initial calibration standard ? Y N/A If the response to the above question is no, were the samples reanalyzed as required ?

#	Date	Internal Standard	Associated Metals	%R (Limits)	Associated Samples	Qualifications
		Bizoq	PD	138,669	18	J/J/P
		100175	<u>– e</u>			
		Y <u>89</u> _		162.032		5/05/p-cr
				100.03	18	57057p-41
<u> </u>	<u> </u>	l		l	1	
	· · · · · ·					
				l	<u> </u>	

LDC#: <u>30160E4</u>

## VALIDATION FINDINGS WORKSHEET Field Duplicates



METHOD: Metals (EPA Method 6010B/7000)

	Concentra		
Analyte	3	5	RPD
Lead	32.3	41.5	25

	Concentra	ation (ug/L)	
Analyte	4	6	RPD
Lead	16.8	16.6	1

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# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification



#### 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:

 %R = Found\_x 100
 Where,
 Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

 True
 True = concentration (in ug/L) of each analyte in the ICV or CCV solution

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
<b>T</b> CN	ICP/MS (Initial calibration)	PD	100,7	100	101	101	7
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
CCV.	ICP/MS (Continuing calibration)	$\zeta$	194.7	200	97	97	4
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

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

# 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:

 %R = Found\_x 100
 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
 True = Concentration of each analyte in the source.

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

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

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

%D = <u>|I-SDR|</u> 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)
$\sim$	ICP interference check						
LCS	Laboratory control sample	PD	51,4	50	107.8	102.8	4
33	Matrix spike	Pb	(SSR-SR) 49,2	50	98,4	98.3	
24	Duplicate	PD	25	ay, Cl	0,4	0.2	
N	ICP serial dilution						

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



# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: \of
Reviewer: OR
2nd reviewer:

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

$\left(\frac{X}{Y}\right)$	<u>N/A</u> Have results <u>N/A</u> Are results	low for all questions answered s been reported and calculated within the calibrated range of th ction limits below the CRDL?	correctly?	•		
Detec equati	ted analyte results for on:	Pb	)	were recalcu	lated and verified	using the following
Concen	tration = <u>(RD)(FV)(Dil)</u> (in. Vol.)	Row data	ecalculation:	32.260	vell	
RD FV In. Vol. Dil	<ul> <li>Raw data conc</li> <li>Final volume (r</li> <li>Initial volume (</li> <li>Dilution factor</li> </ul>	entration nl) ml) or weight (G)	Ч=	32.260 16,830 m	3/L	
#	Sample ID	Analyte		Reported Concentration (ルタ(∟)	Calculated Concentration	Acceptable (Y/N)
	3		PD	32.3	32,3	Y
	4		Ph	16,8	16.8	<u> </u>
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				· · · · · · · · · · · · · · · · · · ·		
			<u> </u>			
Note:				********		

#### LDC Report# 30160E6

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	July 11, 2013
LDC Report Date:	August 9, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
Sample Delivery Group (SDG):	ECI 020507

Sample Delivery Group (SDG): ECL029507

# **Sample Identification**

3740 Blueberry - POU dissolved
3740 Blueberry - PT1 dissolved
3740 Blueberry - PT/DB dissolved
3740 Blueberry - PT2 dissolved
3740 Blueberry - PT3 dissolved
3740 Blueberry - PT4 dissolved
3740 Blueberry - WP1 dissolved
3740 Blueberry - WP2 dissolved
3740 Blueberry - WP3 dissolved
3740 Blueberry - FB dissolved

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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.

Sample 3740 Blueberry - 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) 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
3998 Farm-POU DissolvedMS/MSD (All samples in SDG ECL029507)	Dissolved chromate as chromium	76.3 (85-115)	74.7 (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 samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

### **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 3740 Blueberry - PT1 dissolved and 3740 Blueberry - PT/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 ECL029507

SDG	Sample	Analyte	Flag	A or P	Reason
ECL029507	3740 Blueberry - POU dissolved 3740 Blueberry - PT1 dissolved 3740 Blueberry - PT/DB dissolved 3740 Blueberry - PT2 dissolved 3740 Blueberry - PT3 dissolved 3740 Blueberry - WP1 dissolved 3740 Blueberry - WP2 dissolved 3740 Blueberry - WP3 dissolved 3740 Blueberry - FB dissolved	Dissolved chromate as chromium	L (all detects) UL (all non-detects)	А	Matrix spike/Matrix spike duplicate (%R)

#### **Green Valley Citgo Project**

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL029507

No Sample Data Qualified in this SDG

**Green Valley Citgo Project** 

Dissolved Chromate as Chromium - Field Blank Data Qualification Summary - SDG ECL029507

No Sample Data Qualified in this SDG

LDC #:_	30160E6	VALIDATION COMPLETENESS WORKSHEET	Date: 8/8/13
SDG #:_	ECL029507	Level IV	Page: <u>of </u>
Laborato	ory: Enviro-Chem Lat	poratories, Inc.	Reviewer: 0
			2nd Reviewer:

#### 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
١,	Technical holding times	A	Sampling dates: 7/11/13
11	Initial calibration	A	
10.	Calibration verification	A	
IV	Blanks	A	
v	Matrix Spike/Matrix Spike Duplicates	ASh	MS/D (ECLOZA474)
VI.	Duplicates	N	
VII.	Laboratory control samples	A	LCS
VIII.	Sample result verification	A	
IX.	Overall assessment of data	A	
Х.	Field duplicates	NO	(2,3)
XI	Field blanks	ND	FBEID

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:

Valida	alidated Samples:						
1	3740 Blueberry - POU dissolved	11	21	31			
2	3740 Blueberry - PT1 dissolved	12	22	32			
3	3740 Blueberry - PT/DB dissolved	13	23	33			
3 4	3740 Blueberry - PT2 dissolved	14	24	34			
5	3740 Blueberry - PT3 dissolved	15	25	35			
5 6 7 8 9	3740 Blueberry - PT4 dissolved	16	26	36			
7	3740 Blueberry - WP1 dissolved	17	27	37			
8	3740 Blueberry - WP2 dissolved	18	28	38			
9	3740 Blueberry - WP3 dissolved	19	29	39			
10	3740 Blueberry - FB dissolved	20	30	40			

Notes:





# Method: Inorganics (EPA Method See cover)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
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?				
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)				f
III. Blanks				
Was a method blank associated with every sample in this SDG?	$\langle$			
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?		/		
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?	1						
Were detection limits < RL?	1						
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.							
Target analytes were detected in the field duplicates.		/					
X. Field blanks							
Field blanks were identified in this SDG.	/						
Target analytes were detected in the field blanks.							

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LDC #: 3016066

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page:of
Reviewer: <u>02</u>
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METHOD: Inorganics, EPA Method

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

<u>() N N/A</u> Y (N N/A Was a matrix spike analyzed for each matrix in this SDG? (85-115) 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.

 $N_N/A$  Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$ 35% for soil samples?

<u>Ŷ n nìa</u>

<u>A</u> Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	MS/MSD_ID	Matrix	Analyte	MS %Recovery	MSD %Recovery		Associated Samples	Qualifications
	39998 Farm-POU S	issolvedmsm	5D Champy		74,7		AII	3 L/UL/A
L			-					
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Comments:\_

LDC #:	2016000

#### Validation Findings Worksheet Initial and Continuing Calibration Calculation Verification

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Page:	of
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Method: Inorganics, Method \_\_\_\_\_\_\_\_

The correlation coefficient (r) for the calibration of  $\underline{\Box}^{\sigma \top}$  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 X 100</u>

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

GRYIN

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (mg/l)	uv*s	r or r <sup>2</sup>	r or r <sup>2</sup>	(Y/N)
Initial calibration		s1	0.02	0.0506			
		s2	0.05	0.1208	1.0000	1.0000	
		s3	0.1	0.2176	_		
	6+	s4	0.25	0.544			$\bigcup_{i=1}^{n}$
		s5	0.5	1.0689			1
		s6	1	2.1432			
		s7	5	10.6889			
Calibration verification		CCV		1.0533	105		
Calibration verification		L	5	5.1759	104		
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.\_\_\_\_\_



## VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

	Page:	_of_	
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2nd	Reviewer:	6	

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METHOD: Inorganics, Method Second

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

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

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

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

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated // RPD	Reported %R / RPD	Acceptable (Y/N)
Laboratory control sample		1,053	١	105	105		
3998 Farm-POL	Matrix spike sample		(SSR-SR) 0,747	1	TH.7	74,7	
	Duplicate sample	$\downarrow$	0.763	6.747	Q.\	Ζ.Ι	

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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## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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METHOD: Inorganics, Method See. Over

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?

<u>N N/A</u> Are results within the calibrated range of the instruments?

Y/N N/A Are all detection limits below the CRQL?

Compound (analyte) results for \_\_\_\_\_

\_\_\_\_\_reported with a positive detect were

recalculated and verified using the following equation:

Concentration =

'Y

Recalculation:

AILNO

#	Sample ID	Analyte	Reported Concentration ( )	Calculated Concentration ( )	Acceptable (Y/N)
	· · · · · · · · · · · · · · · · · · ·		· · · · · · · · · · · · · · · · · · ·		
		· · · · · · · · · · · · · · · · · · ·			
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# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Green Valley Citgo Project

Collection Date: June 22, 2013

LDC Report Date:

Matrix:

Water

Parameters:

Chromium & Lead

August 12, 2013

Validation Level:

Laboratory:

Enviro-Chem Laboratories, Inc.

EPA Region III, Level IM2

Sample Delivery Group (SDG): ECL029324

### Sample Identification

3991 Farm-PT1 3991 Farm-PT1 Dissolved 3991 Farm-PT1DB 3991 Farm-PT1DB Dissolved 3991 Farm-PT2 3991 Farm-PT2 Dissolved 3991 Farm-PT3 3991 Farm-PT3 Dissolved 3991 Farm-PT4 3991 Farm-PT4 Dissolved 3991 Farm-POU 3991 Farm-POU Dissolved 3991 Farm-WP1 3991 Farm-WP1 Dissolved 3991 Farm-WP2 3991 Farm-WP2 Dissolved 3991 Farm-WP3 3991 Farm-WP3 Dissolved 3991 Farm-FB 3991 Farm-FB Dissolved

3991 Farm-PT1DBMS 3991 Farm-PT1DBDUP 3991 Farm-WP1MS 3991 Farm-WP1DUP

#### Introduction

This data review covers 24 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.

Samples 3991 Farm-FB and 3991 Farm-FB Dissolved were 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**

Samples 3991 Farm-PT1 and 3991 Farm-PT1DB and samples 3991 Farm-PT1 Dissolved and 3991 Farm-PT1DB Dissolved were identified as field duplicates. No chromium or lead contaminants were detected in any of the samples with the following exceptions:

	Concent		
Analyte	3991 Farm-PT1	3991 Farm-PT1DB	RPD
Lead	6.4	6.3	2

	Concentration (ug/L)				
Analyte	3991 Farm-PT1 Dissolved 3991 Farm-PT1DB Dissolved		RPD		
Chromium	1.0U	1.8	200		
Lead	6.4	6.5	2		

Green Valley Citgo Project Chromium & Lead - Data Qualification Summary - SDGECL029324

No Sample Data Qualified in this SDG

Green Valley Citgo Project

Chromium & Lead - Laboratory Blank Data Qualification Summary - SDGECL029324

No Sample Data Qualified in this SDG

Green Valley Citgo Project Chromium & Lead - Field Blank Data Qualification Summary - SDGECL029324

No Sample Data Qualified in this SDG

LDC #:	30160F4	VALIDATION COMPLETENESS WORKSHEET	Da
SDG #: _	ECL029324	Level IV	Pad
Laborato	ory: Enviro-Chem L	aboratories, Inc.	Review

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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: 6/22/13
11.	ICP/MS Tune	A	
	Calibration	A	
IV.	Blanks	A	
<u>V.</u>	ICP Interference Check Sample (ICS) Analysis	$\square$	Non required
VI.	Matrix Spike Analysis	A	ms
VII.	Duplicate Sample Analysis	A	0.p
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
Х.	Furnace Atomic Absorption QC	N	
XI.	ICP Serial Dilution	N	
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	(1,3) $(2,4)$
xv	Field Blanks	NO	FB=19,20

Note:

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

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

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D = Duplicate TB = Trip blank EB = Equipment blank

Va	alidated Samples: WG+C	$\sim$					
1	3991 Farm-PT1	11	3991 Farm-POU	21	3991 Farm-PT1DBMS	31	
2	3991 Farm-PT1 Dissolved	12	3991 Farm-POU Dissolved	22	3991 Farm-PT1DBDUP	32	
3	3991 Farm-PT1DB	13	3991 Farm-WP1	23	3991 Farm-WP1MS	33	
4	3991 Farm-PT1DB Dissolved	14	3991 Farm-WP1 Dissolved	24	3991 Farm-WP1DUP	34	
5	3991 Farm-PT2	15	3991 Farm-WP2	25		35	
6	3991 Farm-PT2 Dissolved	16	3991 Farm-WP2 Dissolved	26		36	
7	3991 Farm-PT3	17	3991 Farm-WP3	27		37	
8	3991 Farm-PT3 Dissolved	18	3991 Farm-WP3 Dissolved	28		38	
9	3991 Farm-PT4	19	3991 Farm-FB	29		39	
10	3991 Farm-PT4 Dissolved	20	3991 Farm-FB Dissolved	30		40	

Notes:


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Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
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?				
Were the proper number of standards used?	$\square$			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?		۵		
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?	P		/	·
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 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)			$\leq$	·				
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)?			~					
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?			/	Í				
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.	1							
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.	$\frown$							
Target analytes were detected in the field blanks.		/						

LDC #: 30160F1

# VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference



All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-20		
1-00		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, Sn, Ti,
002121		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, Sn, Ti,
QC:21-24		Al, Sb, As, Ba, Be, Cd, Ca(Ĉr,)Co, Cu, Fe(Pb,)Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
		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, Sn, Ti,
ICP-MS		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, Sn, Ti,
GEAA		Al Sh. As, Ba, Re, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sn, Ti,

LDC#:<u>30160F4</u>

## VALIDATION FINDINGS WORKSHEET Field Duplicates



METHOD: Metals (EPA Method 6010B/7000)

	Concentra			
Analyte	1	3	RPD	
Lead	6.4	6.3	2	

	Concentr	Concentration (ug/L)			
Analyte	2	· 4	RPD		
Chromium	1.0U	1.8	200		
Lead	6.4	6.5	2		

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LDC #: 30160F

## VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification



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:

 %R = Found\_x 100
 Where,
 Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

 True
 True = concentration (in ug/L) of each analyte in the ICV or CCV solution

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
ICV	ICP/MS (Initial calibration)	C(	100,2	100	100	100	$\checkmark$
	CVAA (Initial calibration)						,
	ICP (Continuing calibration)						
QN	ICP/MS (Continuing calibration)	CG	205.1	206	103	103	T
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

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

## 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:

 %R = Found\_x 100
 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
 True =
 Concentration of each analyte in the source.

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

RPD = [S-D]x 100Where,S = Original sample concentration(S+D)/2D = Duplicate sample concentration

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

 %D = <u>|I-SDR|</u> x 100
 Where, I = Initial Sample Result (mg/L)

 I
 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)
	ICP interference check						
LCS	Laboratory control sample	p	48,6	50	97,2	97,2	$\gamma$
9/	Matrix spike	Ċ	(SSR-SR) 47,9	50	95,8	94,9	
99	Duplicate	Pb	63	63	$\bigcirc$	$\bigcirc$	$\checkmark$
	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.



# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of_	
Reviewer:	OR	
2nd reviewer:	-1	$\geq$

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

YN YN YN Detect equati	N/A     Have results       N/A     Are results w       N/A     Are all detect       red analyte results for _	Scom Gaudati. Recalculation: 1 = 6,36	s and within the line	ear range of the IC	
#	Sample ID	Analyte	Reported Concentration	Calculated Concentration	Acceptable (Y/N)
		PD	6.4	6.4	Y
	2	Pb	6,4	6,5	<u>۲</u>
					· · · · · · · · · · · · · · · · · · ·

Note:

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	Green Valley Citgo Project
Collection Date:	June 22, 2013
LDC Report Date:	August 9, 2013
Matrix:	Water
Parameters:	Dissolved Chromate as Chromium
Validation Level:	EPA Region III, Level IM2
Laboratory:	Enviro-Chem Laboratories, Inc.
	FCI 020204

# Sample Delivery Group (SDG): ECL029324

# **Sample Identification**

3991 Farm-PT1 Dissolved 3991 Farm-PT1DB Dissolved 3991 Farm-PT2 Dissolved 3991 Farm-PT3 Dissolved 3991 Farm-PT4 Dissolved 3991 Farm-POU Dissolved 3991 Farm-WP1 Dissolved 3991 Farm-WP2 Dissolved 3991 Farm-WP3 Dissolved 3991 Farm-FB Dissolved 3991 Farm-PT3 DissolvedMS 3991 Farm-PT3 DissolvedMS

1

#### Introduction

This data review covers 12 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 3991 Farm-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 samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

#### 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 3991 Farm-PT1 Dissolved and 3991 Farm-PT1DB Dissolved were identified as field duplicates. No dissolved chromate as chromium was detected in any of the samples with the following exceptions:

	Concentra		
Analyte	3991 Farm-PT1 Dissolved	3991 Farm-PT1DB Dissolved	RPD
Dissolved chromate as chromium	0.024	0.023	4

**Green Valley Citgo Project** 

Dissolved Chromate as Chromium - Data Qualification Summary - SDG ECL029324

No Sample Data Qualified in this SDG

**Green Valley Citgo Project** 

Dissolved Chromate as Chromium - Laboratory Blank Data Qualification Summary - SDG ECL029324

No Sample Data Qualified in this SDG

Green Valley Citgo Project Dissolved Chromate as Chromium - Field Blank Data Qualification Summary -SDG ECL029324

No Sample Data Qualified in this SDG

LDC #: <u>30160F6</u>	VALIDATION COMPLETENESS WORKSHEET	Date: <u>8/8/1</u> 3
SDG #: ECL029324	Level IV	Page: <u>of</u>
Laboratory: Enviro-Chem La	aboratories, Inc.	Reviewer:
		2nd Reviewer:

#### 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
1.	Technical holding times	A	Sampling dates: 6[22/13
	Initial calibration	A	
III.	Calibration verification	A	
IV	Blanks	A	
v	Matrix Spike/Matrix Spike Duplicates	A	mslp
VI.	Duplicates	$\sim$	
VII.	Laboratory control samples	A	LES
VIII.	Sample result verification	A	
IX	Overall assessment of data	Ð	
<b>X</b> .	Field duplicates	SW	(1, 2)
XI	Field blanks	<u>_ND</u>	FB=10

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	3991 Farm-PT1 Dissolved	11	3991 Farm-PT3 DissolvedMS	21	31	
2	3991 Farm-PT1DB Dissolved	12	3991 Farm-PT3 DissolvedMSD	22	32	
3	3991 Farm-PT2 Dissolved	13		23	33	
4	3991 Farm-PT3 Dissolved	14		24	34	
5	3991 Farm-PT4 Dissolved	15		25	35	
6	3991 Farm-POU Dissolved	16		26	36	
7	3991 Farm-WP1 Dissolved	17		27	37	
8	3991 Farm-WP2 Dissolved	18		28	38	
9	3991 Farm-WP3 Dissolved	19		29	39	
10	3991 Farm-FB Dissolved	20		30	40	

Notes:\_\_\_\_\_





# Method: Inorganics (EPA Method See cover)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
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?				
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?				
Were titrant checks performed as required? (Level IV only)			$\sim$	
Were balance checks performed as required? (Level IV only)			/	
III. Blanks				
Was a method blank associated with every sample in this SDG?	$\langle$			
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?	1			
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?	_	<u>`</u>		
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			r	
Were the performance evaluation (PE) samples within the acceptance limits?				Y

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## 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.				
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.		P		
Target analytes were detected in the field blanks.		/		

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LDC#<u>30160F6</u>

## VALIDATION FINDINGS WORKSHEET Field Duplicates



Inorganics: Method See Cover

	Concentra		
Analyte	1	2	RPD
Chromate	0.024	0.023	4

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LDC #:	20160FG

#### Validation Findings Worksheet Initial and Continuing Calibration Calculation Verification

Page: of
Reviewer:
2nd Reviewer:

Method: Inorganics, Method \_\_\_\_\_\_\_\_\_

The correlation coefficient (r) for the calibration of _	$Q^{6^+}$	was recalculated.Calibration date:_	6/24/	[]

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

%R = <u>Found X 100</u>

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

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (mg/l)	uv*s	r or r <sup>2</sup>	r or r <sup>2</sup>	(Y/N)
Initial calibration		s1	0.02	0.0506			
	· · ·	s2	0.05	0.1208	1.0000	1.0000	
		s3	0.1	0.2176			
	64	s4	0.25	0.544			$\gamma$
		s5	0.5	1.0689			1
		s6	1	2.1432			
		s7	5	10.6889			
Calibration verification	2	CCV	1	0,9894	99	9	
Calibration verification	Z	L	5	4,9698	99	99	
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.

## VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet



METHOD: Inorganics, Method Second

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

%R = Found x 100 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.

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

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

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated // RPD	Reported %R / RPD	Acceptable (Y/N)
QCS	Laboratory control sample	C( <sup>6+</sup>	1.8226	6	91		
17	Matrix spike sample		(ssr-sr) 6,95		95	95	
11/12	Duplicate sample		0,971	0,965	0,6		

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 #: 30160Fb

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	<u>of</u>
Reviewer:	$\alpha$
2nd reviewer:	
	<b>v</b>

METHOD: Inorganics, Method See OVE

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>N N/A</u> Have results been reported and calculated correctly?

Are results within the calibrated range of the instruments?

<u>'N N/A</u> Are all detection limits below the CRQL?

Q-G+ Compound (analyte) results for \_\_\_\_\_reported with a positive detect were

recalculated and verified using the following equation:

Concentration =

<u>N N/A</u>

Recalculation:

 $y = 2.1346 \times +0.0087$ 

0.06-0.0089 = 0.0239 vg/L 2.1346

#	Sample ID	Analyte	Reported Concentration (MSL)-	Calculated Concentration (1814 0.024	Acceptable (Y/N)
		Chromate	0.024	0.024	7
	·				
				······································	
					· ·
	· · · · · · · · · · · · · · · · · · ·				
				· · · · · · · · · · · · · · · · · · ·	
	· · · · · · · · · · · · · · · · · · ·				

Note: