

Final Report for the Reduction of Chrome (VI) to Chrome (III) in the Secondary Waste Stream of the Effluent Treatment Facility

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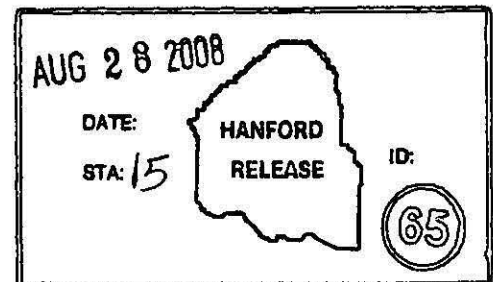
Abstract

This report documents the laboratory results of RPP-PLAN-35958, Test Plan for the Effluent Treatment Facility to Reduce Chrome (VI) to Chrome (III) in the Secondary Waste Stream. With the exception of the electrochemical corrosion scans, all work was carried out at the Center for Laboratory Science (CLS) located at the Columbia Basin College. This document summarizes the work carried out at CLS and includes the electrochemical scans and associated corrosion rates for 304 and 316L stainless steel.

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August 2008



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Office of River Protection

Contract No. DE-AC27-99RL14047

Approved for public release; distribution is unlimited

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List of Terms

Abbreviations

CLS	Center for Laboratory Sciences
ERDF	Environmental Restoration Disposal Facility
ETF	Effluent Treatment Facility
OCP	open circuit potential
TCLP	Toxicity Characteristic Leaching Procedure

1. INTRODUCTION

This report documents the laboratory results of RPP-PLAN-35958, *Test Plan for the Effluent Treatment Facility to Reduce Chrome (VI) to Chrome (III) in the Secondary Waste Stream*.

With the exception of the electrochemical corrosion scans, all work was carried out at the Center for Laboratory Science (CLS) located at the Columbia Basin College. The electrochemical corrosion scans are attached as Appendix A. The full CLS report is attached as Appendix B. This document summarizes the work carried out at CLS and includes the electrochemical scans and associated corrosion rates for 304 and 316L stainless steel.

The reduction of Cr^{+6} to Cr^{+3} will decrease the mobility of chromium in the environment; allowing the waste stream from the Effluent Treatment Facility (ETF) thin film dryer to pass the SW-846 Method 1311, "Toxicity Characteristic Leaching Procedure" (TCLP), for final disposal in the Environmental Restoration Disposal Facility (ERDF) landfill (WHC-191, *Environmental Restoration Disposal Facility Waste Acceptance Criteria*). The concentration limits for chromium at the ERDF are established using the chromium limits in Title 40, *Code of Federal Regulations*, §268.48, "Land Disposal Restrictions - Universal treatment standards" (40 CFR 268.48), of 0.6 mg/L by the TCLP method.

An earlier successful effort used sodium metabisulfite to reduce Cr (VI) to Cr (III) under an oxidation reduction potential of +280 mV and acidic (pH 2) conditions (RPP-RPT-34083, *Reduction of Chrome(VI) to Chrome(III) using Sodium Metabisulfite under Acidic Conditions*).

However, under acidic conditions it was found that the bisulfite attacks the protective chrome coating in 304 and 316L and yields the material unsuitable for processing chloride containing water [RPP-RPT-35175, *Corrosion Study for the Effluent Treatment Facility Chrome (VI) Reductant Solution Using 304 and 316 Stainless Steel*].

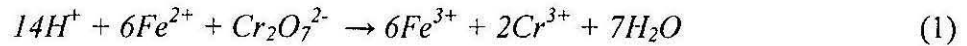
The literature has reported several candidates for the reduction of Cr (VI) "Alternative Chromium Reduction and Heavy Metal Precipitation Methods for Industrial Wastewater" (Chang 2003) reports on the use of sodium metabisulfite, ferrous sulfate, zero-valent iron, and dimethyl dithiocarbamate. From the candidates tested, the sodium metabisulfite and the ferrous sulfate were the most promising (Chang 2003)

"Overview of In Situ Remediation Case Studies," in *Chromium (VI) Handbook* (Jacobs and Rouse 2005) indicates sodium metabisulfite, hydrogen sulfide, ferrous sulfate, and calcium polysulfide as suitable reductants.

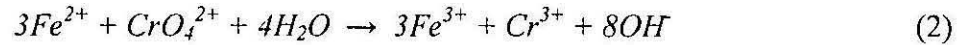
Since sodium metabisulfite has proven not to be a candidate at the ETF due to corrosion properties, it was therefore eliminated in this study. Hydrogen sulfide is considered a dangerous chemical and was not considered in this testing.

The candidate reductants considered are ferrous sulfate and calcium polysulfide. The respective reactions are the following (Jacobs and Rouse 2005):

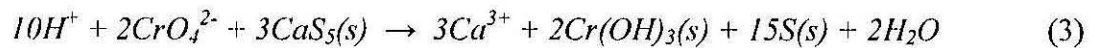
Acidic conditions



Neutral or alkaline



Calcium polysulfide (CaS₅)



According to “Calcium polysulfide remediation of hexavalent chromium contamination from chromite ore processing residue” (Graham et al 2002), reaction 3 rapidly reduces Cr (VI) over a pH range of 8 to 12.

2. MATERIALS AND METHODS

Synthetic groundwater simulant brine was formulated and served as the matrix for spiking chrome (VI) and chrome (III) during the study (Table 1)

Table 1. Synthetic Groundwater Brine Simulant.

Compound	Mass (mg)
CaCl ₂	13,513.68
CaSO ₄	65,579.51
K ₂ SO ₄	4,390.12
Mg(NO ₃) ₂	46,773.69
NaNO ₃	169,911.3
Na ₂ O·SiO ₂	25,793.77
H ₂ SO ₄ ^a	67,044.79
Sum	393,006.87
pH range^a	3.5 to 6

^a If needed, pH will be adjusted with sodium hydroxide or sulfuric acid

2.1 ELECTROCHEMICAL CORROSION SCANS

Cyclic potentiodynamic polarization scans was carried out on the following combinations, using 304 and 316L stainless steel coupons:

- The groundwater simulant at a pH of 3.5 to 6 (as formulated).
- The groundwater simulant with ferrous sulfate at the correct stoichiometry.

Table 2 is the formulation provided by CLS for electrochemical corrosion testing.

Table 2. Synthetic Groundwater Brine for Electrochemical Analysis.

Compound	Mass (g)	Mass (g)
CaCl ₂	13.5136	13 5136
CaSO ₄	65.5795	65 5795
K ₂ SO ₄	4.3901	4 3901
Mg(NO ₃) ₂	46 7736	46 7736
NaNO ₃	169 9113	169.9113
Na ₂ O-SiO ₂	25.7937	25 7937
FeSO ₄	NA	1 39 ^a
Deionized water	674 0382	672 6482

^a Based on 5 mL of a 1M solution

2.2 CHROME REDUCTANT TESTING

Based on the anticipated concentration of chrome (VI), synthetic groundwater brine simulant was prepared, chrome (VI) added, and the candidate reductant added in a timed/volumetric manner.

Final confirmation of the efficacy of the candidate reductant was confirmed by submitting samples to the inductively coupled plasma spectrometry and ion chromatography for total chrome and chrome (VI) concentrations.

2.3 TOXICITY CHARACTERISTIC LEACHING PROCEDURE

A TCLP was carried out based on the matrix in Table 3.

Table 3. Toxicity Characteristic Leaching Procedure Matrix (+ = spike; - = no spike).

Source	pH	Cr (VI) Spike	Cr (III) Spike
Simulated groundwater (~39 wt%)	10	+	+
	11	+	+
	12	+	+
Sodium sulfate (~25 wt%)	10	+	+
	11	+	+
	12	+	+
Ferrous sulfate [after reaction with Cr (VI)]	10	-	-
	11	-	-
	12	-	-
Calcium polysulfide [after reaction with Cr (VI)]	10	-	-
	11	-	-
	12	-	-

The solutions indicated in Table 3 were dried at 365 °F (185 °C) to simulate the ETF drier. After drying, a TCLP was carried out to determine what mass of chrome leached.

3. RESULTS

Laboratory results from the CLS effort are presented in detail in Appendix B. A discussion of the individual samples for TCLP preparation and the sample specifications are presented in Tables 2 and 3 of Appendix B. The summary of averaged analytical results are presented in Appendix B and reproduced here in Table 4.

Table 4. Summary of Analytical Results.

Leachate	Spike (100 ppm)	Adjusted pH ^a	Ferrous Sulfate Reduction	Average Cr(VI) (ppm)	Average Total Cr (ppm)
Na ₂ SO ₄	Cr(III)	10	NA	0.06	0.29
Na ₂ SO ₄	Cr(III)	11	NA	0.0032	0.057
Na ₂ SO ₄	Cr(III)	12	NA	0.007	0.064
Na ₂ SO ₄	Cr(VI)	10	NA	18	19
Na ₂ SO ₄	Cr(VI)	11	NA	15	14
Na ₂ SO ₄	Cr(VI)	12	NA	20	20
Groundwater simulant	Cr(III)	10	NA	1.7	17
Groundwater simulant	Cr(III)	11	NA	3.2	16
Groundwater simulant	Cr(III)	12	NA	2.7	14
Groundwater simulant	Cr(VI)	10	Yes	16	19
Groundwater simulant	Cr(VI)	11	Yes	16	19
Groundwater simulant	Cr(VI)	12	Yes	16	18
Groundwater simulant	Cr(VI)	10	NA	0.18	13
Groundwater simulant	Cr(VI)	11	NA	0.25	12
Groundwater simulant	Cr(VI)	12	NA	0.29	7.1

^a The pH was adjusted prior to evaporation.

Table 5 provides the results of the electrochemical scans.

Table 5. Results of Electrochemical Scans.

Steel	Groundwater Simulant without Ferrous Sulfate			Groundwater Simulant with Ferrous Sulfate		
	OCP ^a	Corrosion Rate (mpy) ^b	χ^2 ^c	OCP	Corrosion Rate (mpy)	χ^2
304	-161.9	1.8E-02	8.62	-148.7	2.5E-02	2.71
316L	-168.9	1.2E-02	1.67	-164.4	1.38E-02	4.29

^a OCP = Open circuit potential, the rest potential of equilibrium potential between the metal and solution

^b mpy = mils per year (1 mil = 0.001 inch)

^c χ^2 = The goodness-of-fit statistic for corrosion rate calculations, a value <100 is acceptable

4. DISCUSSION

The ferrous sulfate was successful in reducing the Cr (VI) to Cr (III). Calcium polysulfide was marginally effective and produced offensive odors during the reduction step and therefore was determined to not be a suitable process for the ETF. From the electrochemical scans, general corrosion was approximately 1E-02 mils per year.

The reduced chrome did exhibit some partial re-oxidation during the drying process, which yielded a crystalline form.

The crystalline matrix was subjected to the TCLP. This resulted in complete dissolution of the sample, allowing chrome to no longer be contained in a binding matrix, which allowed it to pass through the filter media during the filtering step. This was due to the elevated solubility of Cr(OH)₃ in the slightly acidic conditions for the TCLP leach solution.

The process to reduce Cr (VI) was successful in the groundwater brine. To pass TCLP, it is recommended that the reduced brine be grouted in a cementitious matrix.

5. REFERENCES

- 40 CFR 268.48, "Land Disposal Restrictions – Universal treatment standards," *Code of Federal Regulations*, as amended.
- Chang, L., 2003, "Alternative Chromium Reduction and Heavy Metal Precipitation Methods for Industrial Wastewater," *Environmental Progress*, Vol 22 (3), p 174-182.
- Graham, M. C., J. G. Farmer, P. Anderson, E. Paterson, S. Hillier, D. G. Lumsdon, and R. J. F. Bewley, 2002, "Calcium polysulfide remediation of hexavalent chromium contamination from chromite ore processing residue," *Science of the Total Environment*, Vol 364, p 32-44.

Jacobs, J. A., and J. M. V Rouse, 2005, "Overview of In Situ Remediation Case Studies," in *Chromium (VI) Handbook*, J Guertin, J. A. Jacobs, and C. P. Avakian, editors, CRC Press, New York, New York.

RPP-RPT-34083, 2007, *Reduction of Chrome(VI) to Chrome(III) using Sodium Metabisulfite under Acidic Conditions*, Rev 0, CH2M HILL Hanford Group, Inc., Richland, Washington

RPP-RPT-35175, 2007, *Corrosion Study for the Effluent Treatment Facility Chrome (VI) Reductant Solution Using 304 and 316L Stainless Steel*, Rev 0, CH2M HILL Hanford Group, Inc., Richland, Washington

RPP-PLAN-35958, 2008, *Test Plan for the Effluent Treatment Facility to Reduce Chrome (VI) to Chrome (III) in the Secondary Waste Stream*, Rev. 0, CH2M HILL Hanford Group, Inc., Richland, Washington.

SW-846, Method 1311, 1992, "Toxicity Characteristic Leaching Procedure," *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, U.S. Environmental Protection Agency, Washington, D.C.

WHC-191, 2008, *Environmental Restoration Disposal Facility Waste Acceptance Criteria*, Rev 0, Washington Closure Hanford, Richland, Washington.

Appendix A

Electrochemical Scans

Figure A-1. 304 Stainless Steel without Ferrous Sulfate, the Reverse Trace is a Negative Hysteresis, Returning to the Left of the Forward Trace.

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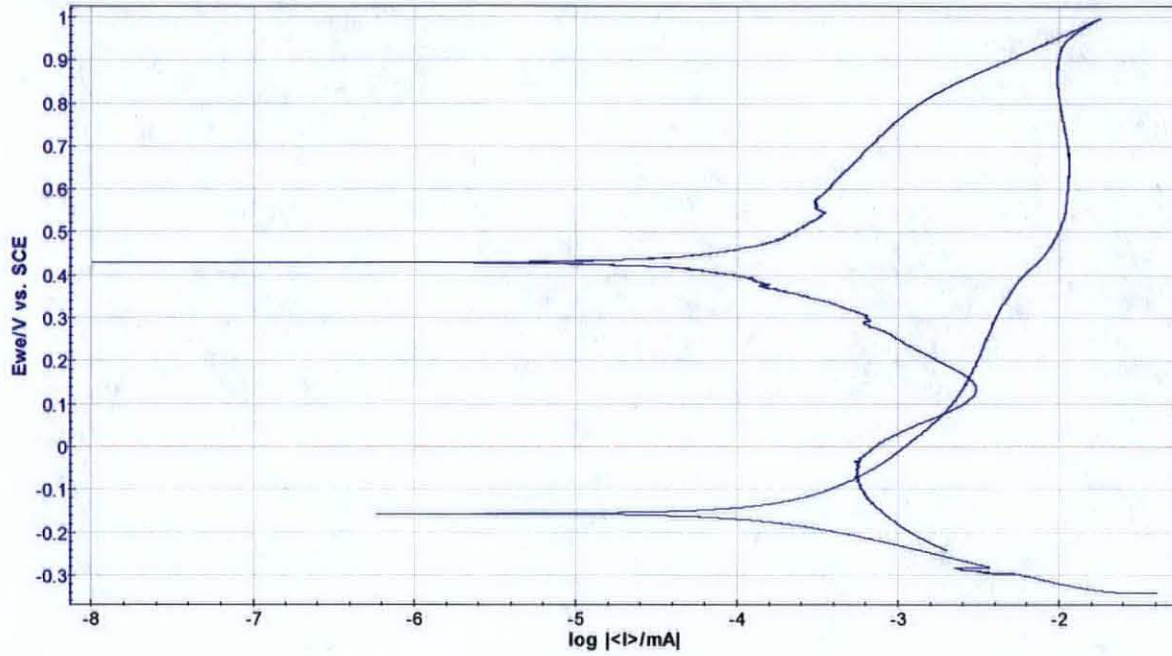


Figure A-2. 304 Stainless Steel with Ferrous Sulfate, the Reverse Trace is a Negative Hysteresis, Returning to the Left of the Forward Trace.

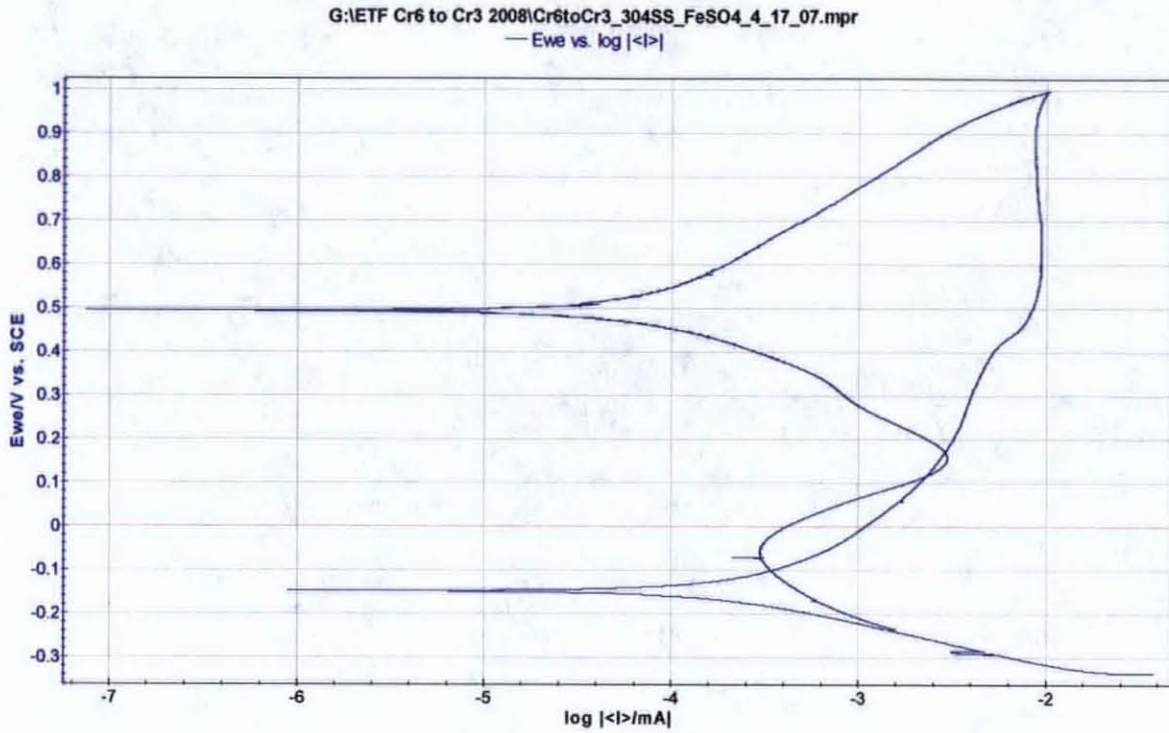


Figure A-3. 316L Stainless Steel without Ferrous Sulfate, the Reverse Trace is a Negative Hysteresis, Returning to the Left of the Forward Trace.

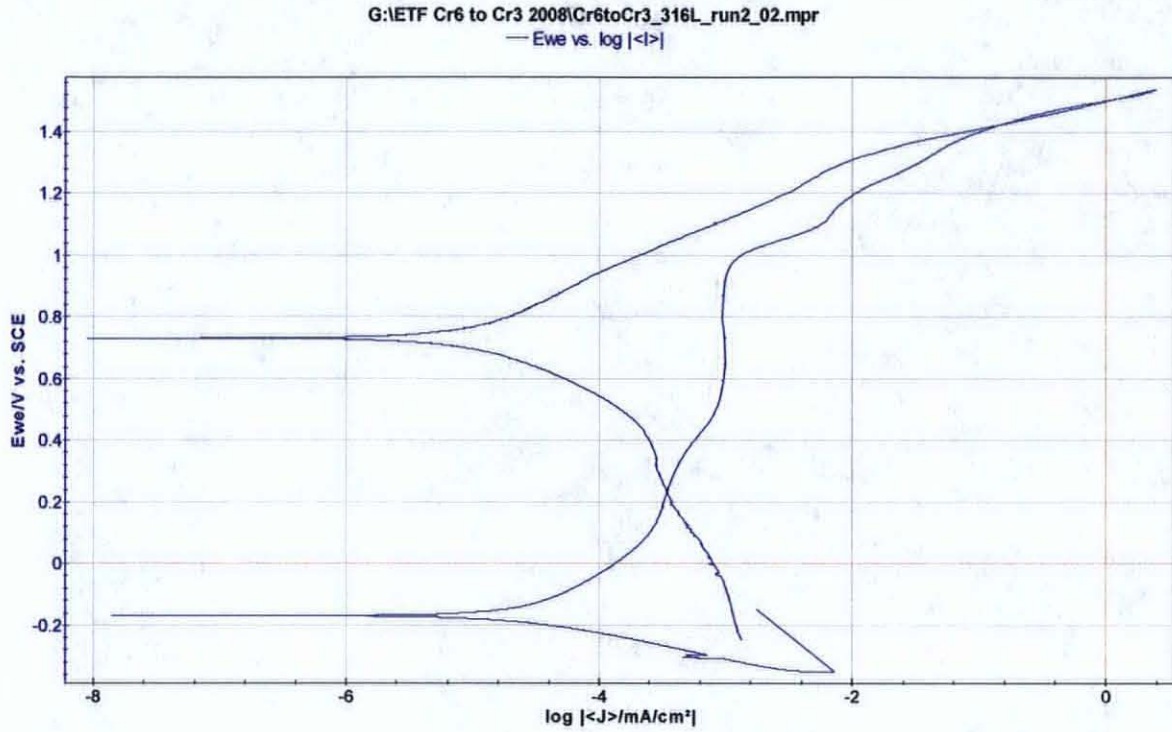
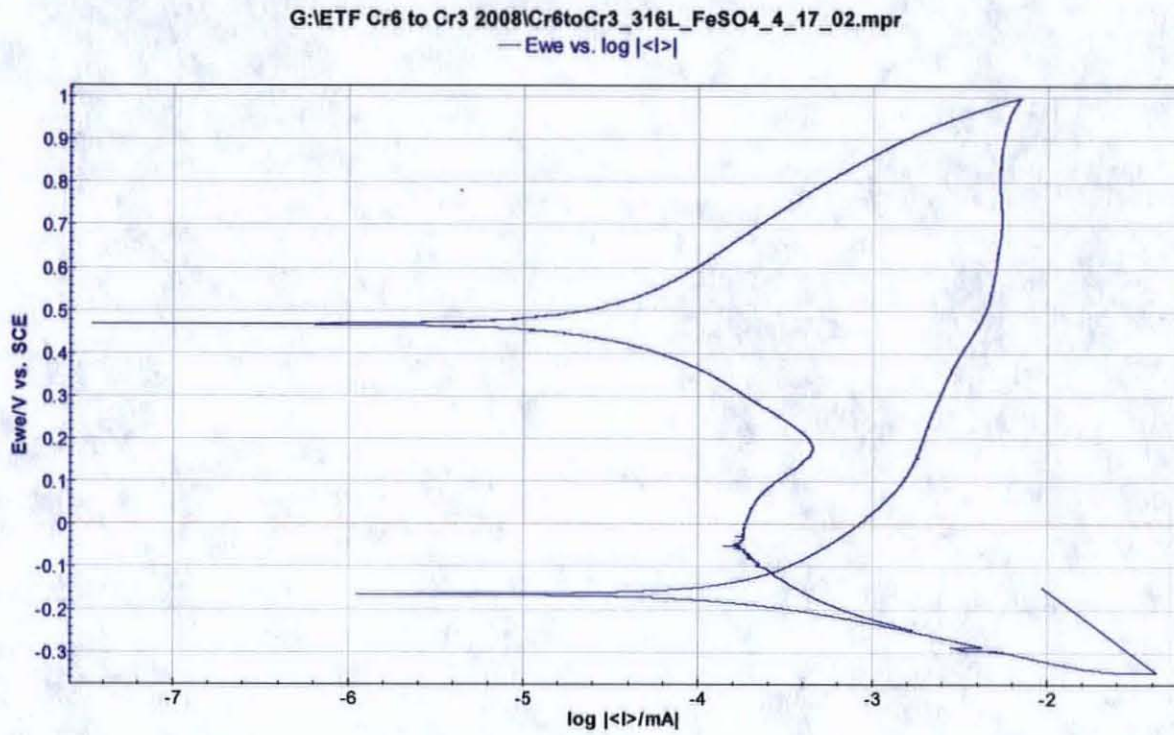
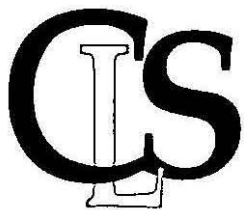


Figure A-4. 316L SS with Ferrous Sulfate, the Reverse Trace is a Negative Hysteresis, Returning to the Left of the Forward Trace.



Appendix B

Center for Laboratory Sciences Report



**Effluent Treatment Facility Secondary
Waste Stream Chrome Reduction**

Laboratory Report

Report Date: July 16, 2008

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RPP PLAN 35958

RJ LeeGroup, Inc Project Number GAL801937
RJ LeeGroup, Inc CLS Document Control Number CLSRPT-0802

RJ LeeGroup, Inc.
Center for Laboratory Sciences

Report for Activities Undertaken at the Center for Laboratory Sciences (CLS) in Support
of CH2M Hill Hanford: Report for RPP-PLAN-35958: Test Plan for the Effluent
Treatment Facility (ETF) to Reduce Chrome (VI) to Chrome (III) in the Secondary Waste
Stream.

Author.

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Report for Activities Undertaken at the Center for Laboratory Sciences (CLS) in Support
of CH2M Hill Hanford: Report for RPP-PLAN-35958: Test Plan for the Effluent
Treatment Facility (ETF) to Reduce Chrome (VI) to Chrome (III) in the Secondary Waste
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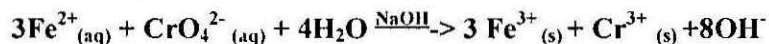
 Date: 7/16/08

The CLS staff operated under the instructions presented in the CH2M Hill Hanford project Test Plan (RPP-PLAN-35958) and under the guidance of CH2M Hill and Fluor Hanford technical staff. The premise of this test plan is that chromium present in the waste stream in the hexavalent state can be reduced to the less mobile trivalent state and then sequestered so as to be impervious to the acid digestion involved with the EPA Method 1311 Toxicity Characteristic Leaching Procedure (TCLP).

Literature review resulted in several candidate reductants, all of which were excluded within the test plan introduction except for ferrous sulfate and calcium polysulfide. Per the test plan, the following chemical equilibria are predicted to be operative during the reduction activities:

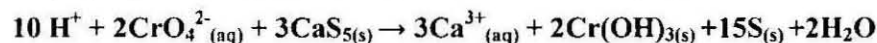
Neutral or alkaline conditions:

Employing ferrous sulfate (FeSO₄) as the reducing agent:



Acidic Conditions:

Employing calcium polysulfide (CaS₅) as the reducing agent:



According to both equilibria, the reduction of the hexavalent chromium will result in the precipitation of insoluble chromium (III) hydroxide.

Formulation for groundwater brine (~39wt %), simulated to represent the influent to the Thin Film Dryer at the Effluent Treatment Facility, was provided in the RPP-PLAN-35958. CLS was also instructed to prepare a sodium sulfate solution (~25wt %). The sodium sulfate solution is included in this project to represent a resin regeneration solution employed at ETF.

Evaporation of simulated groundwater brine and sodium sulfate samples spiked with hexavalent chromium and then reduced by the ferrous sulfate or Ca-polysulfide under 185°C will simulate the conditions at the ETF drier. Once desiccated and subjected to Toxicity Leaching Characteristic Procedure (TCLP) digestion, Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) analysis will address the suitability of the waste for disposal.

The activities undertaken at CLS fall into six phases:

- 1.0 Preparatory Work
 - 1.1 Preparation of the simulated groundwater brine, sodium sulfate and ferrous sulfate solutions
 - 1.2 Fortification of the simulated groundwater brine and sodium sulfate solution with Cr (III) and Cr (VI) standard solutions
- 2.0 Testing of the reduction of hexavalent chromium to trivalent chromium by ferrous sulfate and calcium polysulfide
- 3.0 Preparation of 100 ppm Cr (III) and Cr (VI) fortified sodium sulfate and groundwater simulant samples
- 4.0 Preparation of the chromium fortified sodium sulfate and groundwater simulant and solutions per EPA method 1311, Toxicity Leaching Characteristic Procedure (TCLP)
- 5.0 Analysis of TCLP extracts using EPA 200.8 ICP-MS analysis for metals and EPA method 218.6 Ion Chromatography (IC) analysis for hexavalent chromium
- 6.0 Preparation of simulated groundwater brine for submission to 222S for electro-chemical analysis.

1.1 Preparation of the simulated groundwater brine, sodium sulfate, and ferrous sulfate solutions

RPP-PLAN-35958 directed a simulated groundwater to be prepared from the reagents and masses as listed in Table 1. The pH of the solution was to be adjusted with concentrated H₂SO₄ to fall in the range of 3.5-6.0.

Table 1: Synthetic Groundwater Brine Simulant Preparation

	RPP-PLAN-35958 Formulation	RJLG, CLS Batch 1	RJLG, CLS Batch 2
Volume Deionized Water	1 Liter	2 Liters	2 Liters
Compound	grams	Actual grams	Actual grams
CaCl ₂	13.5136851	27.0389	27.0274
CaSO ₄	65.57951	131.1572	131.159
K ₂ SO ₄	4.39012	8.7812	8.78024
Mg(NO ₃) ₂	46.77369	93.5602	93.5474
NaNO ₃	169.9113	339.8248	339.8226
Na ₂ O-SiO ₂	25.79377	51.5891	41.5875

The groundwater simulant was prepared in two 2 liter batches as per Table 1 and combined into one 4 liter flask. Following thorough mixing, the initial pH of the simulated groundwater was 5.92. Titration with concentrated H₂SO₄ and monitored with a Thermo-Orion¹ 3-Star pH meter resulted in the final pH of 5.43.

The groundwater brine was split into five 400 ml portions and each split was fortified to 100 ppm trivalent or hexavalent chromium. Section 1.2 presents the details for the Cr fortifications.

Sodium sulfate solution preparation

A total of 4 liters of 25% sodium sulfate solution was prepared in two 2 liter batches as follows:

- 500 g Na₂SO₄ (American Chemical Society (ACS) grade 99.0% minimum) CLSID C1020508QQ was transferred to a clean 2 L class A volumetric flask, certification #0007. Approximately 1.8 liters of deionized water was added to flask.¹
- 500 g Na₂SO₄ (ACS grade 99.0% minimum) CLSID C1020508SS was transferred qualitatively to a clean 2 L class A volumetric flask, certification #0054. Approximately 1.8 liters of deionized water was added to flask.

The two volumetric flasks were subjected to agitation on shaker table and heated to induce solution of the Na₂SO₄. Once in solution, the flasks were brought to the 2L mark with deionized water.

The sodium sulfate solution was initially split into three 300 ml and three 660ml portions for preparation of chromium spiking to a final 100 ppm Cr³⁺ or Cr⁶⁺ concentration.

Ferrous sulfate solution preparation:

A total of 100 ml of ferrous sulfate solution was prepared as follows:

- 27.801 g FeSO₄ (ACS reagent grade) was transferred quantitatively to a 100 ml volumetric flask. Deionized water was added to the flask to a final solution volume of 100 ml. The ferrous iron concentration was made at 1 molar.

1.2 Fortification of the simulated groundwater brine and sodium sulfate solution with Cr (III) and Cr (VI) standard solutions

¹ Thermo-Orion is a registered trademark of the Thermo Scientific Corporation, Waltham, Massachusetts.

Trivalent chromium spike solution:

Single element trivalent chromium standard at a concentration of 10,000 µg/mL (Lot #E00369A, CLS ID #MR042407G), was employed to generate 100 ppm Cr³⁺ concentration. No preparation of the standard solution was necessary. Spike volume to bring the sodium sulfate and groundwater brine aliquots to 100 ppm Cr³⁺ were calculated based on the volume of the aliquot and transferred via pipette.

- Spike volume for 100 ppm Cr concentration using Na₂CrO₄.

$$V_{\text{initial}} = ((V_{\text{final}})(C_{\text{final}}))/C_{\text{initial}}$$

V_{initial} volume of stock reagent required to yield necessary concentration

C_{initial} concentration of stock reagent

V_{final} final volume required (sample volume)

C_{final} final concentration required

Example spike volume to prepare a 100 ppm sodium sulfate solution

$$V_{\text{initial}} = ((0.300 \text{ L})(100 \text{ mg/L}))/10^4 \text{ mg/L}$$

$$V_{\text{initial}} = 3.0 \text{ ml Cr}^{3+} \text{ standard}$$

Hexavalent Chromium spike material:

The Cr⁶⁺ standard material was composed of a Na₂CrO₄ · 4 H₂O solid reagent (Lot #534816, CLS ID #MR020708A)

- Mass of Na₂CrO₄ required for a spike of 100 ppm Cr⁶⁺ concentration:

$$(\text{FW Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O} \text{ g/mol} / \text{MW Cr g/mol}) * \text{required final concentration mg/L} * \text{sample volume Liters}$$

- Na₂CrO₄ · 4 H₂O mg to prepare a 100 ppm Cr⁶⁺ sample.

$$(234.04 \text{ g/mol} / 52 \text{ g/mol}) * 100 \text{ mg/L} * 0.660 \text{ L} = 297.05 \text{ mg Na}_2\text{CrO}_4 \cdot 4 \text{ H}_2\text{O}$$

- (for each of three 660 ml Na₂SO₄ samples)

$$(234.04 \text{ g/mol} / 52 \text{ g/mol}) * 100 \text{ mg/L} * 2.00 \text{ L} = 900.2 \text{ mg Na}_2\text{CrO}_4 \cdot 4 \text{ H}_2\text{O}$$

- (135 g Na₂CrO₄ · 4 H₂O for each of six 300 ml groundwater brine samples)

2.0 Testing the reduction of hexavalent chromium to trivalent chromium by ferrous sulfate and calcium polysulfide

Oxidation/Reduction potential testing of the two hexavalent chromium reductant candidates was conducted by the Fluor Hanford and CH2M Hill Hanford technical staff. During this testing, calcium

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polysulfide was rejected as a potential reducing agent. The CLS analytical staff was instructed to continue preparation and analysis with ferrous sulfate as the only reductant. The CLS staff was also instructed to add a 5 ml volume of ferrous sulfate to the fortified simulated groundwater samples decanted for evaporation and TCLP preparation (300 ml total volume at 100 ppm Cr⁶⁺). All HACH² Chromium VI meter testing was conducted by the Fluor Hanford and CH2M Hill Hanford technical personnel.

3.0 Preparation of 100 ppm Cr (III) and Cr (VI) sodium and groundwater simulant samples

Table 2 defines the attributes of fifteen 500 ml beakers prepared for evaporation and eventual TCLP digestion. Prior to evaporation, the solutions fortified with either Cr³⁺ or Cr⁶⁺ were titrated with NaOH to pH of 10, 11 and 12.

The beakers were then subjected to evaporation in a two step process. The initial evaporation occurred in a 95° C oven for approximately 110 hours. The temperature was then elevated to 185°C for an additional 24 hours.

During evaporation, the sodium sulfate solutions formed large well defined crystals. The evaporated groundwater samples dried to solid dense material.

Table 2 presents the aliquots of the sodium sulfate and groundwater samples that were established for evaporation for final TCLP digestion and analysis. The table also illustrates the trivalent and hexavalent chromium spiking and reduction by ferrous sulfate where appropriate.

Table 2: Sample Specifics for TCLP Preparation

Source	NaOH adjusted pH	Cr Fortification	Total Aliquot Volume (mL)	Spike Volume or mg for 100 ppm	Ferrous Sulfate Reduction	ICP/MS and IC Sample IDs
Sodium Sulfate (25 wt %)	10	Cr ³⁺	300	3.0 ml	No	WA030120080048 001-003
	11	Cr ³⁺	300	3.0 ml	No	WA030120080048 004-006
	12	Cr ³⁺	300	3.0 ml	No	WA030120080048 007-009
	10	Cr ⁶⁺	660	297.05 mg	No	WA030120080048 010-012
	11	Cr ⁶⁺	660	297.05 mg	No	WA030120080048 013-015
	12	Cr ⁶⁺	660	297.05 mg	No	WA030120080048 016-018
Simulated Groundwater Brine	10	Cr ³⁺	300	3.0 ml	No	WA030120080048 019-021
	11	Cr ³⁺	300	3.0 ml	No	WA030120080048 022-024
	12	Cr ³⁺	300	3.0 ml	No	WA030120080048 025-027
	10	Cr ⁶⁺	300	135 mg	No	WA030120080048 028-030
	11	Cr ⁶⁺	300	135 mg	No	WA030120080048 031-033
	12	Cr ⁶⁺	300	135 mg	No	WA030120080048 034-036
	10	Cr ⁶⁺	300	135 mg	6 ml	WA030120080048 037-039
	11	Cr ⁶⁺	300	135 mg	6 ml	WA030120080048 040-042
	12	Cr ⁶⁺	300	135 mg	6 ml	WA030120080048 043-045

4.0 Preparation of the evaporated groundwater simulants and Sodium Sulfate solutions using EPA method 1311 (RJLG, CLS LAP-032) Toxicity Leaching Characteristic Procedure (TCLP)

Once evaporated, the remaining solids were pulverized in their containers (500mL beakers). Approximately 5 g from each of the pulverized solids were tested for determination of extraction fluid per EPA1311.

EPA Method 1311 specifies leaching material in one of two extraction fluids based on the pH of a 5 g sample in deionized water. If the pH is <5 then extraction fluid #1 is used. Extraction fluid #2 is

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used for a pH of >5 which requires the use of 3.5 mL of 1N HCL and heating to 50 degrees C. The test sample is maintained at 50 degrees C and then allowed to come to room temperature. The method further requires a leachant to leachate ratio of 1: 20.

The evaporated sodium sulfate solutions required the use of extraction fluid #2, while the evaporated groundwater simulants required extraction fluid #1. Approximately 100g of solids were used for leaching all TCLP preparations. Table 3 provides the TCLP sample specifics.

The extraction fluid and 100 g portions were rotated at ~30 RPM for a period of 18 hours at room temperature (23 degrees C plus or minus 2 degrees) room temp, in accordance with EPA 1311. During the rotation process sample WA100320080048 (007-009) leaked approximately 40% of its solution. Rotation continued to completion once the spill was contained and cleaned.

As Figure 1 illustrates, the CrVI fortified samples exhibited strong color providing visual indication of the dominate chromium oxidation state.



Figure 1 Post TCLP Leached Groundwater Samples

At the conclusion of the rotation process it was evident that the greater portion of the 100 grams of solids exposed to the TCLP extraction solution in each TCLP extract dissolved. The undissolved solids from the simulated groundwater simulant formed a heavy white particulate. When agitated, this particulate settled rapidly to the bottom of the container. Therefore, the groundwater simulants were not filtered prior to further digestion.

Table 3: TCLP Sample Specifications

Source	ICP/MS and IC Sample IDs	Cr Fortification	grams of Sample for Extraction Fluid	grams of Sample for Digestion	Extraction Fluid	gram of Extraction Fluid
Sodium Sulfate (25 wt %)	WA030120080048 001-003	Cr ³⁺	5 0463	71 2	2	1427 5
	WA030120080048 004-006	Cr ³⁺	4 9891	68 8	2	1377 2
	WA030120080048 007-009	Cr ³⁺	5 038	68 7	2	1394 5
	WA030120080048 010-012	Cr ⁶⁺	5 0009	100 3	2	2005.8
	WA030120080048 013-015	Cr ⁶⁺	5 0497	100 3	2	2006 4
	WA030120080048 016-018	Cr ⁶⁺	5 1486	100	2	2001 7
Simulated Groundwater Brine	WA030120080048 019-021	Cr ³⁺	5 1949	48.7023	1	974 046
	WA030120080048 022-024	Cr ³⁺	5 0762	48 9396	1	978 792
	WA030120080048 025-027	Cr ³⁺	5 0022	50 5696	1	1011 392
	WA030120080048 028-030	Cr ⁶⁺	5.0585	50 2193	1	1004 386
	WA030120080048 031-033	Cr ⁶⁺	5 0606	47 3263	1	946.526
	WA030120080048 034-036	Cr ⁶⁺	5 107	46 3983	1	927 966
	WA030120080048 037-039	Cr ⁶⁺ (R)	5 0703	50.416	1	1008 32
	WA030120080048 040-042	Cr ⁶⁺ (R)	5 0745	49 2752	1	985 704
	WA030120080048 043-045	Cr ⁶⁺ (R)	5 0841	48.8004	1	976 008

(R) Indicates reduction with FeSO₄

5.0 Analysis of TCLP extracts using EPA 200.8 ICP/MS analysis for metals and EPA method 300.0 IC analysis for hexavalent chromium

The TCLP extracts produced from section III were prepared for analysis of total Cr by ICP-MS. To ensure data validations, three samples of each TCLP leachate were drawn from the parent solution for

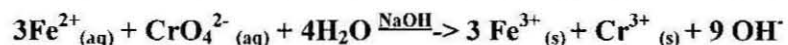
total chromium analysis The TCLP extracts were digested and prepared according to EPA method 3005A, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for analysis by FLAA or ICP Spectroscopy" Following preparation, all samples were analyzed using EPA method 200.8 (RJLG, CLS LAP-030), "Determination of Trace Elements in Waters and Wastes by ICP-MS "

An additional 3 samples were drawn from each leachate for hexavalent chromium analysis The samples were prepared and analyzed by EPA method 218.6 (RJLG, CLS LAP-007) "Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents and in PVC Filters by Ion Chromatography using EPA Method 218.6"

Discussion of Analytical Results

The complete total chromium (ICP-MS analysis) and hexavalent chromium (IC analysis) analytical reports are presented in attachments A and B respectively Table 4 presents a summary of the averaged values for each TCLP leachate.

The pH of the post rotation TCLP extracts was approximately 4-6 for all samples. According to the redox equilibria for iron sulfate and sodium chromate in the presence of sodium hydroxide, the ferrous iron reduces chromium in the hexavalent oxidation state to the trivalent oxidation state



Based on the above equation, the resulting chromium product should then be insoluble $\text{Cr}(\text{OH})_3$ $\text{Cr}(\text{OH})_3$ is relatively insoluble in a pH range of 7.0 to 8.5. Based on the total chromium ICP-MS results (>5 ppm chrome for all groundwater samples) the pH adjustment to 10, 11 and 12 did not maintain chromium in an insoluble state throughout the preparation and analytical processes

Following the TCLP extraction the pH of the solutions was in the range of 4-6. The TCLP leaching process in the acidic environment of extraction fluids resulted in the dissolution of the $\text{Cr}(\text{OH})_3$ Visual inspection of the extracts supports this conclusion as does the ICP-MS data. The average total chromium results from the ICP-MS data are relatively similar throughout the experiment regardless of the addition of the ferrous sulfate. The ICP-MS results demonstrate the inability of the pH adjustment

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and FeSO_4 reduction to sequester the chromium such that the TCLP extraction according to EPA Method 1311 will not mobilize the chrome

However, the IC results for the FeSO_4 reduced samples indicates that the reduction of the hexavalent chromium is stable following the TCLP extraction. The IC preparation and analysis of the 3 TCLP solutions fortified to 100 ppm Cr^{6+} and the 3 TCLP solutions fortified to Cr^{6+} and reduced with FeSO_4 was performed twice to verify the resulting hexavalent chromium concentrations. In both analytical runs, the hexavalent chromium concentrations were less than 1 ppm. The preparation for IC analysis requires adjustment of the pH to 9. The solutions were visually inspected following this adjustment, and no color change was evident. From a qualitative perspective, this supports the actual ion chromatography results.

The ICP-MS and IC analysis of the sodium sulfate fortified to 100 ppm trivalent chromium has been rejected for reporting purposes. The reaction of a sodium sulfate solution fortified with 100 ppm Cr^{3+} and subjected to pH adjustment with NaOH does not support the sequestering of chromium.

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Table 4: Summary of Analytical Results

Sample IDs	Leachate Description	Fortification	Adjusted pH	FeSO ₄ Reduction	Average Cr ⁶⁺ ppm	Average Total Cr ppm
001-003	Sodium Sulfate	100 ppm Cr ³⁺	10		0.060	0.29
004-006	Sodium Sulfate	100 ppm Cr ³⁺	11		0.0032	0.057
007-009	Sodium Sulfate	100 ppm Cr ³⁺	12		0.0070	0.064
010-012	Sodium Sulfate	100 ppm Cr ⁶⁺	10		18	19
013-015	Sodium Sulfate	100 ppm Cr ⁶⁺	11		15	14
016-018	Sodium Sulfate	100 ppm Cr ⁶⁺	12		20	20
019-021	Groundwater Simulant	100 ppm Cr ³⁺	10		1.7	17
022-024	Groundwater Simulant	100 ppm Cr ³⁺	11		3.2	16
025-027	Groundwater Simulant	100 ppm Cr ³⁺	12		2.7	14
028-030	Groundwater Simulant	100 ppm Cr ⁶⁺	10		16	19
031-033	Groundwater Simulant	100 ppm Cr ⁶⁺	11		16	19
034-036	Groundwater Simulant	100 ppm Cr ⁶⁺	12		16	18
037-039	Groundwater Simulant	100 ppm Cr ⁶⁺	10	Yes	0.18	13
040-042	Groundwater Simulant	100 ppm Cr ⁶⁺	11	Yes	0.25	12
043-046	Groundwater Simulant	100 ppm Cr ⁶⁺	12	Yes	0.29	7.1

6.0 Preparation of simulated groundwater brine for submission to 222S for electro-chemical analysis

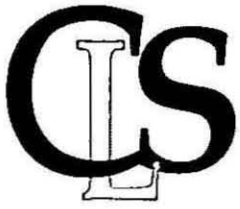
Table 5: Synthetic Groundwater Brine Simulant Preparation for Electro-Chemical Analysis at 222S

	RPP-PLAN-35958 Formulation	RJLG, CLS Batch 3	RJLG, CLS Batch 4
Volume Deionized Water	1 Liter	1 Liter	1 Liter
Compound	grams	Actual grams	Actual grams
CaCl ₂	13 5136851	13 5167	13.5130
CaSO ₄	65.57951	65 5801	65 6084
K ₂ SO ₄	4 39012	4 3897	4 4046
Mg(NO ₃) ₂	46 77369	46 7715	46 7690
NaNO ₃	169 9113	169 9092	169.9201
Na ₂ O-SiO ₂	25 79377	25 7909	25 7934

Two additional batches of the groundwater brine simulant were prepared per the above table for electro-chemical analysis. The pH of both batches was approximately 5. A 5 ml volume of 1M FeSO₄ was added to batch 4. The two 1 liter simulated groundwater brine simulants were manifested to CH2M Hill Hanford on April 4, 2008 (the chain of custody is attached as appendix D).

All work on this project was performed in conjunction with all hygiene and safety precautions as prescribed in the CLS Chemical Hygiene/Safety Plan, CLS Quality Assurance Manual and the Hanford Analytical Services Quality Assurance Requirements Document.

All samples created during this project will be archived at the RJ Lee Group CLS facility for 90 days or relinquished to CH2M Hill. Active chain of custody will be maintained until final disposal or transfer of custody. All waste including samples at the end of the archival period unless manifested to CH2M Hill Hanford will be disposed of per applicable federal and state regulations. Quality assurance operations were documented throughout the project as prescribed in the CLS Quality Assurance Plan.



APPENDIX A

Inductively Coupled Plasma-MS Analytical Reports

Prepared by:
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Operations Manager

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Richland, WA 99352
Attn Larry Lockrem
Phone (509)373-4711
Fax

RJ Lee Group Job No WA100320080048
Sample Collection 2/8/2008
Samples Received 3/10/2008
Report Date 7/15/2008
Analysis/Prep Date 3/14/2008
Preparation Method EPA 1311
Client Project Chrome VI -
GAL801937

RJLG Job No WA100320080048 Total Chromium
Analysis Method EPA 200.8, Non-Potable Water

Table with 9 columns: Sample ID, RJ Lee Group ID, Units, Result, Qualifier, % RPD, Quantitation Limit, Dilution Factor (if needed). Rows include Sodium Sulfate adjusted to pH 10, 11, 12, and Cr 3+, Cr 6+ Spike tests.

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Sample Collection 2/8/2008
Samples Received 3/10/2008
Report Date 7/15/2008
Analysis/Prep Date 3/14/2008
Preparation Method EPA 1311
Client Project Chrome VI -
GAL801937

RJLG Job No - WA100320080048 Total Chromium
Analysis Method EPA 200.8, Non-Potable Water

Table with 9 columns: Sample ID, RJ Lee Group ID, Units, Result, Qualifier, % RPD, Quantitation Limit, Dilution Factor (if needed). Rows include various groundwater samples (Cr3+, Cr6+) at different pH levels (10, 11, 12) with their respective results and qualifiers.

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RJ Lee Group Job No WA100320080048
Sample Collection 2/8/2008
Samples Received 3/10/2008
Report Date 7/15/2008
Analysis/Prep Date 3/14/2008
Preparation Method EPA 1311
Chent Project Chrome VI - GAL801937

RJLG Job No · WA100320080048 Total Chromium
Analysis Method. EPA 200.8, Non-Potable Water

Table with 9 columns: Sample ID, RJ Lee Group ID, Units, Result, Qualifier, % RPD, Quantitation Limit, Dilution Factor (if needed). Rows include Groundwater Simulant + Iron Sulfate adjusted to pH 10, pH 11, and pH 12, with individual sample results and averages.

* All solid matrices reported on a dry weight basis unless otherwise noted
* All values reported without blank correction unless otherwise noted

Analyst Comments Quantitation Limit is based on the lowest point on the calibration curve and dilution factor (where applicable)

Report Qualifiers

D=Dilution applied to sample

H = Holding times for preparation or analysis exceeded

J = Analyte detected below quantitation limit

B = Analyte detected in the associated Method Blank

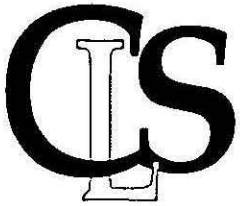
S = Spike Recovery outside accepted recovery limits

R = RPD (relative percent difference) outside accepted recovery limits

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.

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RPP-RPT-37878, Rev 0



APPENDIX B

**Ion Chromatography Analytical Report
for Hexavalent Chromium Analysis**

Prepared by:
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Sample Collection 2/8/2008
Samples Received 3/10/2008
Report Date 7/15/2008
Analysis/Prep Date 3/14/2008
Preparation Method EPA 300 0
Client Project Chrome VI - GAL801937

RJLG Job No WA100320080048 Hexavalent Chromium
Analysis Method EPA 300 0, Non-Potable Water

Table with 8 columns: Sample ID, RJ Lee Group ID, Units, Result, Qualifier, % RPD, Quantitation Limit, Dilution Factor (if needed). Rows include Sodium Sulfate adjusted to pH 10, 11, and 12 for Cr3+ and Cr6+ spikes.

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RJ Lee Group Job No WA100320080048
Sample Collection 2/8/2008
Samples Received 3/10/2008
Report Date 7/15/2008
Analysis/Prep Date 3/14/2008
Preparation Method EPA 300 0
Client Project Chrome VI - GAL801937

RJLG Job No WA100320080048 Hexavalent Chromium
Analysis Method EPA 300 0, Non-Potable Water

Table with 8 columns: Sample ID, RJ Lee Group ID, Units, Result, Qualifier, % RPD, Quantitation Limit, Dilution Factor (if needed). Rows include Groundwater Simulant adjusted to pH 10, 11, 12 for Cr3+ and Cr6+ spikes.

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RJ Lee Group Job No WA100320080048
Sample Collection 2/8/2008
Samples Received 3/10/2008
Report Date 7/15/2008
Analysis/Prep Date 3/14/2008
Preparation Method EPA 300 0
Client Project Chrome VI -
GAL801937

RJLG Job No WA100320080048 Hexavalent Chromium
Analysis Method EPA 300 0, Non-Potable Water

Table with 8 columns: Sample ID, RJ Lee Group ID, Units, Result, Qualifier, % RPD, Quantitation Limit, Dilution Factor (if needed). Rows include Groundwater Simulant, Cr** spike + Iron Sulfate adjusted to pH 10, 11, and 12 with various sample IDs and results.

* All solid matrices reported on a dry weight basis unless otherwise noted
* All values reported without blank correction unless otherwise noted

Analyst Comments Quantitation Limit is based on the lowest point on the calibration curve and dilution factor (where applicable)

Report Qualifiers

D = Dilution applied to sample

H = Holding times for preparation or analysis exceeded

J = Analyte detected below quantitation limits

B = Analyte detected in the associated Method Blank

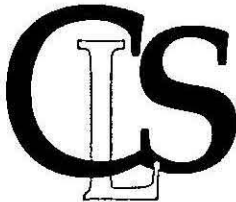
S = Spike Recovery outside accepted recovery limits

R = RPD (relative percent difference) outside accepted recovery limits

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.

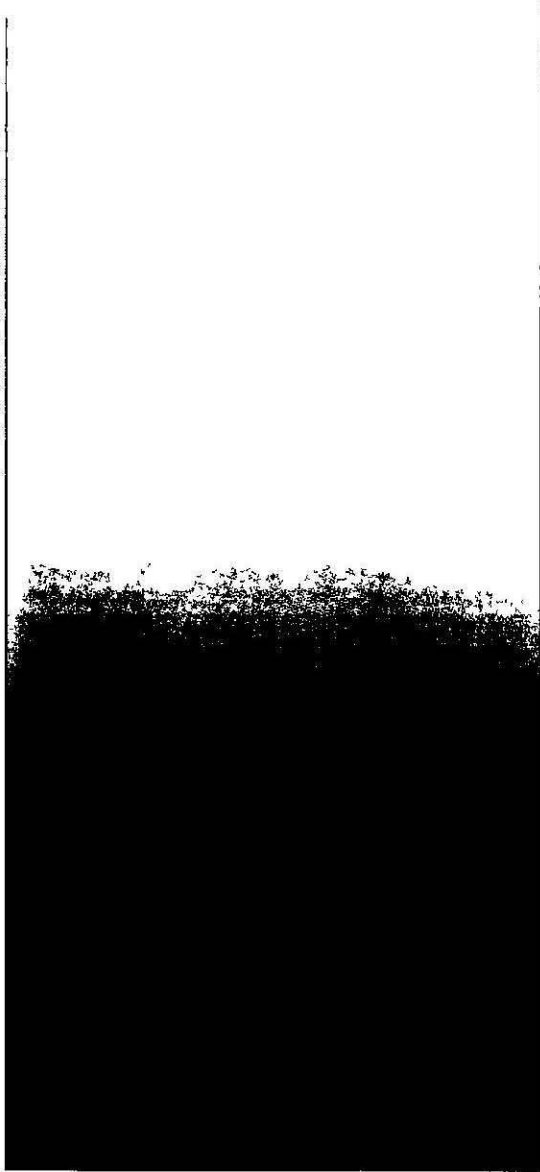
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APPENDIX C

Quality Assurance Reports



Appendix C: Quality Assurance Report

Recovery of Total Chromium for Samples 001 through 009 (ICP-MS Analysis)

The recovery of total chromium failed to meet expectations for the sodium sulfate samples (001-009) fortified to 100 ppm chromium III. Recovery of chromium following TCLP extraction of these samples was expected to be at least as high as the recovery for the chromium III fortified groundwater brine samples (019-027).

The parent sodium sulfate solutions that resulted in 001 through 009 consisted of deionized water and ACS grade Na₂SO₄ and the single element trivalent chromium standard (10,000 ppm). No other materials were added to the solution prior to evaporation. Following evaporation, the samples were treated as discussed in the report for TCLP extraction—no other materials were added to the evaporites other than the extraction fluid. The resulting leachate exhibited no color and no precipitates.

The RJ Lee Group, CLS staff has researched these anomalies and have not determined an explanation for the ICP-MS results. The following considerations were taken into account:

- Failure of the fortification delivery mechanism:

Prior to the on start of fortification, the bottle of the single element trivalent chromium standard (10,000 ppm) was unopened and contained a nominal volume of 10 ml. Following the delivery of the three 3 ml spikes the volume remaining in the standard bottle was approximately 1 ml. No other analytical work requiring fortification with a trivalent chromium standard solution occurred during the time of the preparatory work for the RPP-PLAN-35958.

- Erroneous chromium concentration reported on the label of the standard:

The remaining portion of this standard was analyzed on the ICP-MS on June 19, 2008 and the results from three sub-samples yielded an average total chromium concentration of 1.04×10^4 ppm.

- Failure of the ICP-MS sample induction mechanism to take up the appropriate volume of subsample

If the instrument's peristaltic pump fails to deliver the appropriate volume of subsample, the concentration reported on the instrument data sheets can be either greatly exaggerated or undervalued.

These samples were run again via ICP-MS with resulting similar concentrations. Three samples, one from each pH range were also checked with the ICP-AES and the recovery of total chromium was of the same magnitude as the ICP-MS data.

- Failure of the ICP-MS to recover total chromium.

Samples 001 through 009 were analyzed in the same batch run as samples 010 through 018 (fortified to 100 ppm hexavalent chromium). The total chromium recovery for samples 010 through 018 is not in question and the corresponding hexavalent chromium (IC) results compare very well with the ICP-MS data for these samples.

The batch quality control samples (calibration verification, calibration blank, detection limit, laboratory control standard, and laboratory fortified blank) analyzed with samples 001 through 009 in the ICP-MS instrument run are all within the acceptable toleration range of +/- 25% recovery.

- Fractional crystallization during evaporation resulting in a non-homogeneous evaporite.

Prior to TCLP leaching, the samples were manually crushed in order to generate a homogenous material for leaching. A portion of the crushed sample was removed to test for TCLP extraction fluid determination. The remainder of the sample (minus a portion that adhered to the beaker walls) was subjected to leaching.

In order to rule this mechanism out, the TCLP procedure and ICP-MS analysis would have to be repeated and there is insufficient material remaining to accomplish this.

Discussion of Analytical Report Qualifiers

Inductively Coupled Plasma-Mass Spectroscopy (Total Chromium Concentration)

- ICP-MS Data B Qualifiers

The reagent blanks prepared and analyzed within the instrument run for the total chromium concentration analysis were slightly above the level of the detection limit sample (0.02 ppm chromium). Therefore, the sample data is presented with a B qualifier. This data was not blank corrected. With the exception of samples 001 through 009 discussed earlier as presenting problematic data, the effect of

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blank correction at the level of 0.02 ppm would have no effect on the high chromium concentrations of the remaining 36 samples

- ICP-MS Data S Qualifiers

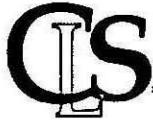
The ICP-MS quality control analyses (ICV, DL, LCS, LCSD and CCVs) were within the tolerance limits of +/- 25% with the exception of the analytical batch sample spikes

Samples -013, -022, -025, -034, -040 and -043 exhibited spikes of greater than 145% recovery. It is possible that the spike concentration introduced to the sample split was 0.5 ppm chromium as opposed to 0.05 ppm chromium.

All other sample spikes exhibit a negative recovery as compared to the sample that the spike was split from. It is likely that the spike concentration of 0.05 ppm was lost within the very high chromium concentrations of the samples (7.1 through 20 ppm). The allowable statistical variation around the sample concentration at these values is such that 0.05 ppm addition of chromium is not statistically viable. The spike concentration for this set of samples was established such that if the sequestering of chromium had been effective, a 0.05 ppm chromium spike would have been recoverable.

Ion Chromatography (Hexavalent Chromium Concentration)

There were no data qualifiers applied to the hexavalent chromium laboratory report. All of the IC quality control analyses (ICV, DL, LCS, and CCVs) were within the tolerance limits of +/- 25%. All of the spike concentration recoveries were within the limits of +/- 25%.



LABORATORY REPORT
Quality Control

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RJLG Job No. WA100320080048 Total Chromium
Analysis Method EPA 200.8, Non-Potable Water

Batch ID	Sub-Sample ID	QA Type	Units	Result	Expected/Spike Concentration	% Recovery/RPD	Qualifiers	Dilution Factor (if needed)
030608B-589		ICV	mg/L	0.04781	0.05	96		
030608B-590		LRB	mg/L	<0.02	0.02			
030608B-591		DL	mg/L	0.01899	0.02	95		
030608B-592		MB	mg/L	0.02096			B	
030608B-593		LCS	mg/L	0.07595	0.05	110		
030608B-595	WA100320080048-001-Dup	Dup	mg/L	0.32956		7.02	D	10
030608B-596	WA100320080048-001-MS	MS	mg/L	0.3653	0.05	116	B,D	10
030608B-602		CCV	mg/L	0.05004	0.05	100		
030608B-603		LRB	mg/L	<0.02	0.02			
030608B-605		CCV	mg/L	0.0455	0.05	91		
030608B-606		LRB	mg/L	<0.02				
031308A-412		ICV	mg/L	0.04702	0.05	94		
031308A-413		LRB	mg/L	<0.02				
031308A-414		DL	mg/L	0.01926	0.02	96		
031308A-417	WA100320080048-004-MS	MS	mg/L	0.533	0.5	94	B,D	10
031308A-420	WA100320080048-007-MS	MS	mg/L	0.5193	0.5	91	B,D	10
031308A-425		CCV	mg/L	0.04752	0.05	95		
031308A-426		LRB	mg/L	<0.02				
031308B-440		ICV	mg/L	0.0463	0.05	92.6		
031308B-441		LRB	mg/L	<0.02				
031308B-442		DL	mg/L	0.01926	0.02	96		
031308B-443	WA100320080048-010-MS	MS	mg/L	18.4778	0.5	-157	B,D,S	1000
031308B-448	WA100320080048-013-MS	MS	mg/L	15.3157	0.5	308	B,D,S	1000
031308B-449	WA100320080048-016-MS	MS	mg/L	19.33	0.5	-174	B,D,S	1000
031308B-452		CCV	mg/L	0.04634	0.05	93		
031308B-453		LRB	mg/L	<0.02				

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LABORATORY REPORT
Quality Control

RJ Lee Group, Inc | Center for Laboratory Sciences
2710 North 20th Avenue, Pasco WA 99301
Tel (509) 545-4989 | Fax (509) 544-6010

RJLG Job No.: WA100320080048 Total Chromium
Analysis Method: EPA 200.8, Non-Potable Water

Batch ID	Sub-Sample ID	QA Type	Units	Result	Expected/Spike Concentration	% Recovery/RPD	Qualifiers	Dilution Factor (if needed)
031308D-483		ICV	mg/L	0.46342	0.5	93		
031308D-484		LRB	mg/L	<0.02				
031308D-485		DL	mg/L	0.20158	0.2	101		
031308D-486		LCS	mg/L	0.0777	0.05	114		
031308D-487		LCSD	mg/L	0.07467	0.05	108		
031308D-488		MB	mg/L	0.02054			B	
031308D-490	WA100320080048-019-MS	MS	mg/L	16.8493	0.05	-1585	B,D,S	100
031308D-494	WA100320080048-022-Dup	Dup	mg/L	16.85771		6.09	D	100
031308D-495	WA100320080048-022-MS	MS	mg/L	16.005	0.05	287	B,D,S	100
031308D-496		CCV	mg/L	0.47048	0.5	94		
031308D-497		LRB	mg/L	<0.02				
031308D-501	WA100320080048-025-MS	MS	mg/L	13.108	0.05	258	B,D,S	100
031308D-505	WA100320080048-028-Dup	Dup	mg/L	18.71458		3.40	D	100
031308D-506	WA100320080048-028-MS	MS	mg/L	18.0838	0.05	-11	B,D,S	100
031308D-508		CCV	mg/L	0.4697	0.5	94		
031308D-509		LRB	mg/L	<0.02				
031308D-512	WA100320080048-031-MS	MS	mg/L	18.8528	0.05	-192	B,D,S	100
031308D-516	WA100320080048-034-MS	MS	mg/L	18.1052	0.05	301	B,D,S	100
031308D-520		CCV	mg/L	0.48931	0.5	98		
031308D-521		LRB	mg/L	<0.02				
031308D-505	WA100320080048-037-Dup	Dup	mg/L	12.8336		3.34	D	100
031308D-523	WA100320080048-037-MS	MS	mg/L	13.1984	0.05	-157	B,D,S	100
031308D-527	WA100320080048-040-MS	MS	mg/L	12.11802	0.05	145	B,D,S	100
031308D-532		CCV	mg/L	0.49277	0.5	99		
031308D-533		LRB	mg/L	<0.02				
031308D-578	WA100320080048-043-MS	MS	mg/L	7.38941	0.05	769	B,D,S	10

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LABORATORY REPORT
Quality Control

RJ Lee Group, Inc | Center for Laboratory Sciences
2710 North 20th Avenue, Pasco WA 99301
Tel (509) 545-4989 | Fax (509) 544-6010

RJLG Job No., WA100320080048 Total Chromium
Analysis Method. EPA 200.8, Non-Potable Water

Batch ID	Sub-Sample ID	QA Type	Units	Result	Expected/Spike Concentration	% Recovery/RPD	Qualifiers	Dilution Factor (if needed)
031308D-580		CCV	mg/L	0.4917	0.5	98		
031308D-581		LRB	mg/L	<0.02				
031308D-583		CCV	mg/L	0.4807	0.5	96		
031308D-584		LRB	mg/L	<0.02				

* All solid matrices reported on a dry weight basis unless otherwise noted
Analyst Comments

Report Qualifiers

D = Dilution applied to sample

H = Holding times for preparation or analysis exceeded

L = Sample condition at receipt out of compliance with method defined conditions

B = Analyte detected in the associated Method Blank

S = Spike Recovery outside accepted recovery limits

R = RPD (relative percent difference) outside accepted recovery limits

These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which the results are used or interpreted. Unless notified in writing to return the samples covered by this report, RJ Lee Group will store the samples for a period of thirty (30) days before discarding. A shipping and handling fee will be assessed for the return of any samples.

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LABORATORY REPORT
Quality Control

RJ Lee Group, Inc | Center for Laboratory Sciences
2710 North 20th Avenue, Pasco WA 99301
Tel (509) 545-4989 | Fax (509) 544-6010

RJLG Job No : WA100320080048 Hexavalent Chromium
Analysis Method: EPA 300.0, Non-Potable Water

Batch ID	Sub-Sample ID	QA Type	Units	Result	Expected/Spike Concentration	% Recovery	Qualifier	Dilution Factor (if needed)
X031208A-004		LRB	mg/L	<0 0025				
X031208A-005		ICV	mg/L	0.095253	0 1	95		
X031208A-007		LCS	mg/L	0 009228	0 01	92		
X031208A-010		DL	mg/L	0 001982	0 0025	79		
X031208A-020		CCV	mg/L	0 093454	0 1	93		
X031208A-021		LRB	mg/L	<0 0025				
X031208A-030	WA100320080048-001-MS	MS	mg/L	0 106054	0 05	96		
X031208A-031	WA100320080048-002-MS	MS	mg/L	0 107526	0 05	93		
X031208A-032	WA100320080048-003-MS	MS	mg/L	0 106949	0 05	94		
X031208A-033		CCV	mg/L	0.097597	0 1	98		
X031208A-034		LRB	mg/L	<0 0025				
X031208A-035	WA100320080048-013-MS	MS	mg/L	18.754091	5	85	D	100
X031208A-042		CCV	mg/L	0.094846	0 1	95		
X031208A-043		LRB	mg/L	<0 0025				
X031308A-005		LRB	mg/L	<0 0025				
X031308A-006		ICV	mg/L	0.107451	0 1	107		
X031308A-011		CCV	mg/L	0 109339	0 1	109		
X031308A-012		LRB	mg/L	<0 0025				
X051608A-001		ICV	mg/L	0.097052	0 1	97		
X051608A-002		DL	mg/L	0 002188	0 0025	88		
X051608A-003		LCS	mg/L	0 097861	0 1	98		
X051608A-004		LCSD	mg/L	0 09928	0 1	99		
X051608A-005		LRB	mg/L	<0 0025				
X051608A-019		CCV	mg/L	0.093966	0 1	94		
X051608A-020		LRB	mg/L	<0 0025				
X051608A-030		CCV	mg/L	0 097878	0 1	98		
X051608A-031		LRB	mg/L	<0 0025				

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LABORATORY REPORT
Quality Control

RJ Lee Group, Inc | Center for Laboratory Sciences
2710 North 20th Avenue, Pasco WA 99301
Tel (509) 545-4989 | Fax (509) 544-6010

RJLG Job No. WA100320080048 Hexavalent Chromium
Analysis Method EPA 300.0, Non-Potable Water

Batch ID	Sub-Sample ID	QA Type	Units	Result	Expected/Spike Concentration	% Recovery	Qualifier	Dilution Factor (if needed)
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* All solid matrices reported on a dry weight basis unless otherwise noted

Analyst Comments

Report Qualifiers

D = Dilution applied to sample

H = Holding times for preparation or analysis exceeded

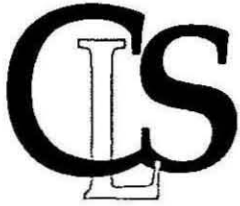
L = Sample condition at receipt out of compliance with method defined conditions

B = Analyte detected in the associated Method Blank

S = Spike Recovery outside accepted recovery limits

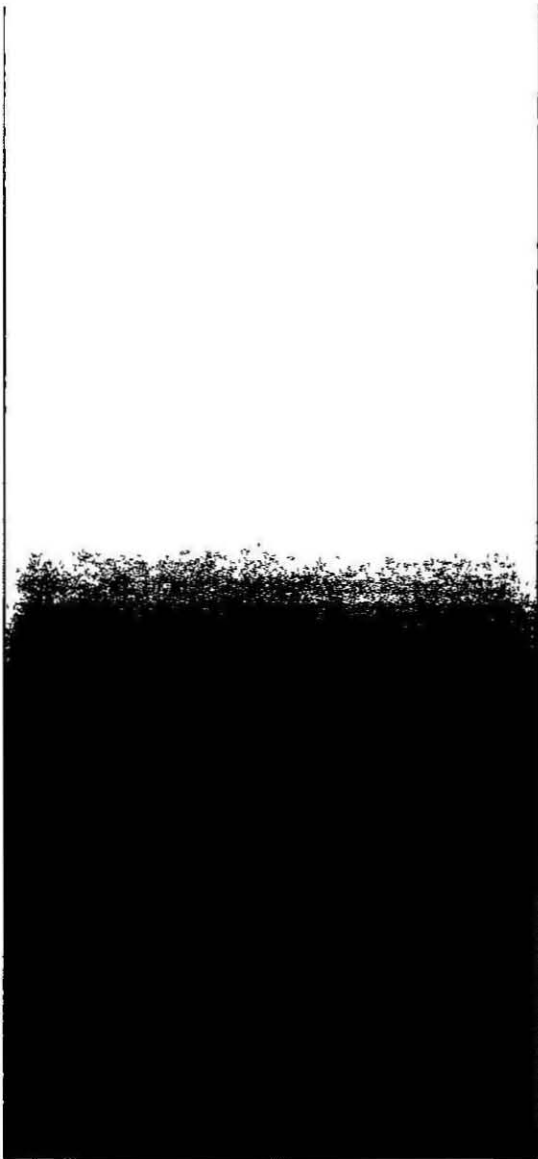
R = RPD (relative percent difference) outside accepted recovery limits

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APPENDIX D

**Chain of Custody for
Simulated Groundwater
Brine Samples Relinquished
to 222S**



Request for Laboratory Analytical Services
Chain of Custody

Purchase Order No.: _____ Client Job No.: _____
 Project No.: _____ Client No.: _____
 Date Logged In: _____ Logged In By: _____

Name: James Deacon
 Company: CLAM Hill
 Address: _____
 City, State, Zip: _____ Phone: _____ Fax: _____
 Call with Verbal Results: _____
 Email Results To: _____
 Fax Results To: _____
 Name: _____
 Company: _____
 Address: _____
 City, State, Zip: _____ Phone: _____ Fax: _____

Send Invoice To: _____
 Name: _____
 Company: _____
 Address: _____
 City, State, Zip: _____ Phone: _____ Fax: _____

Special Instructions: Simultaneous analysis @ 2005
CrVI Synthetic
Groundwater Base Simulant
pH 5 (no Fe SO4)
CrVI Synthetic
Groundwater Base Simulant
pH 5 with Fe SO4

Sample Identification: _____
 Sample Description: _____
 Date: _____
 Wipe Area / Air Volume: _____

Sample Time: _____
 Start: _____ Stop: _____

Turnaround Request: _____
 Standard: Yes? No? _____
 Sample Purpose: Information Regulatory
 System ID No.: _____
 DOH Source No.: _____
 Multiple Sources No.: _____
 Preservation: _____
 Matrix: _____
 Containers: _____
 Uropes: H2SO4, HCl, NaOH, No-SO4
 4°C, HNO3, E-Extract
 SW-Surface Water, DW-Drinking Water, O-Oil, X-Other
 Pol-Plastic, G-Glass, W-Wipe, A-Air (filter or tube)

Analysis Requested: _____
 Pres. Upon Receipt (Y/N): _____
 No. Containers: _____
 Container Type: _____
 Marks: _____
 Preservation: _____

Chain of Custody: _____
 Received By (Signature): _____ Date: _____ Time: _____
 Received By (Print Name): _____
 Company Name: _____ Method of Shipment: _____

Chain of Custody: _____
 Received By (Signature): _____ Date: _____ Time: _____
 Received By (Print Name): _____
 Company Name: _____ Method of Shipment: _____

10503 Brittonview Parkway
 Minnessis, VA 20109
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 Fax: (703) 368-7761

330 McCormick Street
 San Leandro, CA 94577
 Tel: (510) 567-0480
 Fax: (510) 567-0488

350 Hochberg Road
 Monroeville, PA 15146
 Tel: (724) 325-1776
 Fax: (724) 733-1799

Questions?
 Contact Client Services:
 (724) 367-1833