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Gas Treatment of Cr(VI)-Contaminated Sediment Samples from the North 60's Pits of the Chemical Waste Landfill

E. C. Thornton
J. E. Amonette

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**Gas Treatment of Cr(VI)-Contaminated
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Pacific Northwest National Laboratory
Richland, Washington 99352

Summary

Twenty sediment samples were collected at depths ranging from 5 to 100 ft (1.5 to 30 m) beneath a metal-contaminated plating-waste site and extensively characterized for Cr(VI) content and environmental availability. Three samples were selected for treatment with diluted gas mixtures with the objective of converting Cr(VI) to Cr(III), which is relatively nontoxic and immobile. These tests were designed to provide information needed to evaluate the potential application of gas injection as an in situ remediation technique. Gas treatment was performed in small columns (4.9-cm ID, 6.4- to 13.9-cm long) using 100 ppm ($\mu\text{L L}^{-1}$) H_2S or ethylene mixtures in N_2 . Treatment progress during the tests involving H_2S was assessed by monitoring the breakthrough of H_2S .

Evaluation of H_2S treatment efficacy included 1) water-leaching of treated and untreated columns for ten days, 2) repetitive extraction of treated and untreated subsamples by water, 0.01 M phosphate (pH 7) or 6 M HCl solutions, and 3) Cr K-edge X-ray absorption near-edge structure (XANES) spectroscopy of treated and untreated subsamples. Results of the water-leaching studies showed that the H_2S treatment decreased Cr(VI) levels in the column effluent by 90% to nearly 100%. Repetitive extractions by water and phosphate solutions echoed these results, and the extraction by HCl released only 35-40% as much Cr in the treated as in the untreated samples. Analysis by XANES spectroscopy showed that a substantial portion of the Cr in the samples remained as Cr(VI) after treatment, even though it was not available to the water and phosphate extracting solutions. These results suggest that this residual Cr(VI) is present in low solubility phases such as PbCrO_4 or sequestered in unreacted grain interiors under impermeable coatings formed during H_2S treatment. However, this fraction is essentially immobile and thus unavailable to the environment.

Water-leaching of sediment treated with the diluted ethylene mixtures indicated that no significant reduction of Cr(VI) occurred. Control tests conducted on uncontaminated soil indicated interaction of H_2S with the soil matrix occurs and that treatment progress is likely to be slower if H_2S mixtures in air, instead of N_2 , are used. A series of H_2S treatment tests was also conducted under varying degrees of treatment progress. Water-leaching results of these tests indicate that the degree of Cr(VI) reduction is roughly proportional to the percentage of H_2S breakthrough achieved during treatment.

Treatment of Cr(VI)-contaminated sediment with diluted H_2S gas mixtures was thus demonstrated to be an effective approach for immobilizing Cr. Knowledge of the specific waste phases present at a waste site is also essential, however, for predicting treatment effectiveness and field performance characteristics. Anticipated field treatment-time requirements associated with diluted H_2S injection at the subject waste site was deduced from the laboratory treatment-test results presented in this study. It is concluded that about 0.0004 pounds of H_2S are required to treat one pound of sediment. This indicates, for example, that a treatment period of about four months would be required at the waste site if a 200-ppm mixture of H_2S in N_2 is injected at a flow rate of 200 cfm.

Acknowledgments

Grateful acknowledgment is made of the laboratory and field assistance provided by Michael A. Baechler, Julie A. Olivier, Deborah L. Huang, Rand D. Miller, Stacey D. Evelo, Ying Shi, and Sallie R. Sanders, and the important collaborations with Will F. Bleam (University of Wisconsin), Carol L. Stein (University of Washington), Khris B. Olsen (PNNL), Mark A. Beck (WHC), James M. Phelan (SNL), and Dianne C. Marozas (SNL). Reviews by Robert G. Riley, Jonathan S. Fruchter, and Dianne C. Marozas greatly improved the manuscript. Funding for the study was provided by the U.S. DOE Office of Science and Technology.

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1.0 Introduction

Contamination of soil by metals and radionuclides is an important environmental concern in private industry and at governmental facilities and often leads to groundwater contamination as a result of infiltration (Riley et al. 1992). Conventional approaches to site remediation generally have emphasized either site isolation using physical barriers or removal of contaminated media to offsite locations. Limitations of these approaches include uncertainties associated with the long-term isolation capabilities of engineered barriers, exposure of workers during site excavation or pumping, limited landfill capacities, and long-term liabilities associated with offsite disposal (Ghassemi 1988).

These limitations have generated interest in the development of a variety of in situ remediation technologies, including in situ chemical treatment or immobilization (e.g., EPA 1985, EPA 1990, Smith 1985, and Gee and Wing 1994). In situ chemical treatment should be especially effective for metals and radionuclides, whose solubility or sorption characteristics are strongly dependent on concentrations of associated ligands and the redox and pH characteristics of the aqueous environment. The benefits of in situ remediation techniques include 1) a reduction of risks associated with excavation activities and 2) an elimination of the need to relocate wastes to offsite locations. In addition, in situ treatment may be more cost effective in certain situations.

Demonstration of in situ chemical treatment approaches have been limited to small-scale applications. Solid treatment agents, for example, have been used to remediate relatively small sites consisting of metal or radionuclide-contaminated soils on or near the ground surface (Czupryna et al. 1989, Kim et al. 1990, and Browman and Spalding 1984). Injection of aqueous solutions of dithionite into an aquifer to reduce structural Fe in sediment clay minerals has also been proposed and a single-well field demonstration has been carried out (Amonette et al. 1994; Fruchter et al. 1996). For treatment of contamination in the vadose zone, the use of treatment agents in the form of gas mixtures seems to offer the best combination of ease in the introduction, control of the agent, and efficient removal of residual agent at the completion of treatment.

The thermodynamic properties of chromate [Cr(VI)] suggest that vadose zone contamination could be treated by exposure to dilute gas mixtures containing chemically reducing constituents such as H_2S , H_2 , or various hydrocarbons (e.g., ethylene, acetylene, or methane). Such a treatment would be expected to convert Cr(VI) to Cr(III), which is relatively insoluble under most naturally occurring conditions (Rai et al. 1989). Thus, preliminary investigations have demonstrated that H_2S gas treatment is potentially effective in remediating Cr(VI)-contaminated soils (Thornton and Trader 1993; Thornton et al. 1994; Thornton and Jackson 1994). In these tests, clean soil samples were spiked with Cr(VI) solutions and then treated with a 100 ppm ($\mu L L^{-1}$) mixture of H_2S in nitrogen. Subsequent leaching tests demonstrated that more than 90% of the Cr(VI) in these freshly spiked soils had been immobilized.

The success of the studies with freshly spiked soils suggests that gaseous reduction could provide a means of remediating Cr(VI)-contaminated sediments in situ. A potential application of this technology includes disposal pits that have received plating-bath waste solutions. Many sites also exist that have become contaminated due to the infiltration of cooling waters released from power plants or nuclear reactors. Chromate compounds are commonly present in these cooling waters, having been

added as a corrosion inhibitor. Subsequent infiltration of Cr(VI) through the vadose zone beneath either of the above waste-site types often results in the contamination of groundwater systems.

A potential site for application of this treatment approach has been identified within the North 60's Pits of the Chemical Waste Landfill (CWL) near Sandia National Laboratories (SNL) in Albuquerque, New Mexico. The waste site is a small disposal cell that received spent plating-bath solutions containing Cr and other metals, and is underlain by sediments contaminated with Cr(VI). In situ gas treatment at this or similar sites would involve the injection of a dilute mixture of H_2S in air into the waste site sediment and extraction by a network of wells positioned at the edge of the site.

The primary objective of this study, therefore, was to evaluate, through laboratory testing activities, whether the H_2S -gas treatment would result in significant immobilization of Cr in the sediments of the waste site. In addition, several tests were conducted with ethylene mixtures to evaluate it as a potential alternative to H_2S . The experimental approach involved packing portions of sediment samples into columns and passing reactive-gas mixtures through the columns. Gas sensors were situated at the inlet and outlet ports of these columns to monitor treatment progress in the tests involving H_2S . Following completion of soil treatment, the columns containing treated and untreated controls were leached with deionized water and the leachate samples were analyzed for hexavalent Cr. Mass balance considerations were then employed to determine the fraction of chromate reduced and immobilized during treatment. Portions of unreacted and reacted sediment samples were also used for determination of Cr oxidation state and changes in bonding characteristics associated with H_2S treatment.

The information gained during these tests provides a basis for designing a treatment approach at the CWL waste site. In particular, it is illustrated how H_2S /soil mass ratios associated with the laboratory tests can be used to estimate treatment times required to complete treatment at the site as a function of H_2S concentration and gas-flow rates.

2.0 Methods and Materials

2.1 Collection and Initial Characterization of Sediment Samples

An uncontaminated sediment sample collected in the vicinity of the CWL was characterized to obtain initial information regarding the physical, chemical, and mineralogical properties of the sediment. This sample consisted of 92% sand, 7% silt, and 1% clay size-fractions. X-ray diffraction analysis of the bulk sediment indicated the presence of 71% quartz, 16% plagioclase, 5% calcite, 3% potassium feldspar, and minor clays. The clay size-fraction consisted primarily of smectite. The sample was determined to contain 5.35% total carbon and 3.6% inorganic carbon by weight. The pH of a 1:1 sediment/water slurry was 7.7, reflecting the alkaline nature of the sediment.

Chromate-contaminated sediment samples were obtained during the sonic drilling of a borehole (CWL 180,273) at the North 60's Pit in March of 1994. A total depth of 100 ft was drilled, and samples were collected and homogenized from 5-ft intervals, except for the first 5 ft where no recovery was reported. These samples ranged from silts to medium-grained sands and gravels and consisted primarily of a mixture of granitic debris and limestone clasts.

All the samples collected from the CWL borehole were analyzed for Cr(VI), total Cr, and other metals and anions by Enseco Rocky Mountain Analytical Laboratory using SW-846 methods (EPA 1992). Total Cr was considerably higher than Cr(VI) in these samples (Table 2.1), probably as a result of 1) reduction of Cr(VI) by magnetite and other reductants before sampling, and 2) the relatively low solubility of some of the Cr(VI) phases in water, which would prevent quantitative recovery by EPA Method 7196. A general correlation exists between the amounts of Cr(VI) and the total amounts of Cr, Pb, Cu, and SO_4 in the sediments (Table 2.1), with the highest values observed from a depth of 5 to 35 ft and in the sample associated with the 50-55 ft interval. The covariance of these metals is consistent with the nature of the waste stream, which was a spent chromium plating-bath H_2SO_4 solution. Iron does not exhibit any notable trend with respect to depth or Cr content, although it is distinctly higher in the sample collected from the 20-25 ft interval.

A series of sediment batch leach tests was also conducted with water and with a diluted acetic acid solution as described in the toxicity characteristic leaching procedure (TCLP; SW-846 Method 1311 of EPA 1992). The primary objective of this activity was to develop a field screening procedure for Cr(VI). In these tests, 5 g of soil sample were leached with 100 mL of TCLP extraction fluid or deionized water. The slurries were continuously agitated during extraction, and the extractant fluid samples were filtered before analysis. The water leaching tests were undertaken to assess the optimum time for extraction of hexavalent chromium, whereas comparison of the water leach and TCLP results should indicate if similar amounts of hexavalent chromium can be extracted by both approaches. The results of these tests are presented in Table 2.2. This information suggests that a 15-min leach is sufficient, at least for screening purposes. The data also indicate that the percentage recovery of hexavalent chromium achieved by the TCLP approach is typically less than that resulting from water leaching. This is probably due to the increased susceptibility of hexavalent chromium to reduction under acidic conditions. This suggests that the TCLP approach is not suitable for assessing the mobility of hexavalent chromium in natural environments.

Table 2.1. Selected Sediment Analytical Data for CWL Borehole 180,273^(a)

Depth Interval	Cr ⁶⁺ Method 7196	Total Cr Method 6010	Fe Method 6010	Pb Method 7421	Cu Method 6010	SO ₄ Method 300.0
-- ft -----			μg g ⁻¹ -----			
5-10	2.2	2190	633	45100	17.1	9370
10-15	20.9	56.1	6510	103	4.5	1350
10-15 (duplicate)	34.6	73.4	6930	89.5	4.6	895
15-20	6.0	2400	9340	18500	458	5890
20-25	0.27	6060	11600	38600	1250	8180
25-30	291	6110	6220	818	1440	7940
30-35	112	138	6150	15.2	8.5	226
35-40	0.11	11.2	6540	7.6	6.1	177
40-45	<0.25	7.7	6900	9.1	5.4	128
40-45 (duplicate)	<0.25	4.9	5940	6.4	4.3	134
45-50	<0.25	2.6	4800	3.4	3.5	40.9
50-55	17.6	633	8480	304	125	962
55-60	<0.050	7.8	7480	6.8	11.3	78.8
60-65	<0.025	3.0	7300	5.3	5.7	34.0
70-75	<0.050	9.5	9220	27.1	5.4	134
75-80	<0.050	5.9	8630	6.7	5.3	88.3
80-85	<0.050	5.5	7310	5.2	5.4	96.3
85-90	<0.050	2.4	5310	4.5	4.1	29.0
90-95	<0.050	3.0	5470	4.1	4.4	41.1
95-100	0.066	4.0	6360	6.4	4.7	38.9

(a) Samples were analyzed by SW-846 Methods (EPA 1992).

Table 2.2. Amounts of Cr(VI)^(a) Extracted from Contaminated Sediments by Water Leaching and the TCLP Method

Depth	Water Leaching Time					TCLP Leaching Time	
	5 min	15 min	30 min	60 min	16 hr	15 min	16 hr
-- ft --	----- $\mu\text{g g}^{-1}$ -----						
10-15	31.4	37.3	36.3	36.9	40.5	30.1	21.2
15-20	71.9	214	218	201	--	165	5.1
20-25	2.6	3.0	3.2	3.1	--	2.3	1.7
25-30	85.1	86.8	97.0	104	--	14.8	5.1
30-35	35.6	34.6	39.2	35.8	--	12.2	12.2
35-40	9.0	9.8	10.3	10.8	--	--	--

(a) Determined by diphenylcarbazide per Method 7196 (EPA 1992).

-- Not determined.

Significant differences can be observed between the Cr(VI) concentrations presented in Tables 2.1 and 2.2. In particular, Cr(VI) determinations for the 15-20' interval varied greatly. It appears that this variation is related to the high Pb content present in this interval (see Table 2.1 and discussion below). Differences may also be due in part to the fact that these analyses are associated with different sections of the sediment cores collected at the CWL. The values reported in Table 2.2 correspond to portions of the samples used in the experiments conducted in this study.

2.2 Gas Treatment Procedure

Portions of the samples recovered from the 15-20, 25-30, and 30-35 ft intervals (hereafter referred to as the 20', 30', and 35' samples, respectively) were selected for gas-treatment testing activities. At least two portions of each sample were used in this study; one was treated with gas, and the other was used as an untreated control. The untreated and treated columns were leached, and the leachate chemistries were compared to determine the degree of Cr(VI) immobilization achieved by gas treatment. In addition, a third column was packed and treated for both the 20' and 30' samples. Subsamples of this material were characterized by selective-extraction and X-ray absorption spectroscopic techniques. Several control tests were also conducted in which uncontaminated sediment was treated with diluted H_2S . A test matrix that indicates major test parameters is presented in Table 2.3, and associated test data and information are presented in Appendices A, B, and C.

The test samples were packed into columns of known volume per American Society for Testing and Materials (ASTM) protocol (WHC 1990). The measured mass of sediment and known volume of the column, together with a determination of the particle density and moisture content of the sediments (Table 2.4), were used to calculate the pore volume of the column. Short columns [4.9 cm (2") ID by 7.35 cm (2.9")] and long columns [4.9 cm (2") ID by 13.85 cm (5.5")] were used in this study. The short columns were employed when limited amounts of sample were available.

The components and configuration of the gas treatment system are schematically illustrated in Figure 2.1. The system, located in a hood, permits delivery of a regulated, continuous flow of a mixture of the treatment gas in a carrier gas. The treatment gases used in these tests were prepared using a compressed gas cylinder containing 1% H_2S or ethylene in nitrogen. This was further diluted with nitrogen from a second cylinder using two electronic flow controllers and an associated electronic control-box to produce a 100 ppm ($\mu L L^{-1}$) H_2S mixture. The flow rate of the mixture was adjusted to 2.5 L/min and directed through the packed sediment column for the duration of each test. The uncertainty associated with the H_2S -gas concentration and flow-rate values was about 5%.

Amperometric electrochemical gas sensors were used to measure the H_2S concentrations at the inlet and outlet sides of the column. The sensor associated with the column inlet was used to verify that the treatment gas concentration was 100 ppm (± 5 ppm), while the outlet sensor monitored H_2S breakthrough. The accuracy of these sensors was verified using a certified H_2S calibration mixture. The concentration measurements were recorded during each test using a data logger, as illustrated in Figure 2.1. This information was downloaded to a computer at the end of each test for storage and analysis. The outlet concentration data were used to prepare breakthrough curves, which indicate the degree of treatment achieved. Treatment tests involving H_2S were generally continued until the ratio C/C_0 exceeded 0.7 (i.e., an outlet gas concentration of greater than 70 ppm was observed).

Table 2.3. Test Matrix

Experiment Number	Sediment Sample	Gas Treatment Concentration	Comments
4-1092-1	SNL 20' (SNLA014796)	untreated	long column, leached
4-1092-2	SNL 20'	100 ppm H ₂ S in N ₂	long column, leached
4-1093-1	SNL 30' (SNLA014798)	untreated	long column, leached
4-1093-2	SNL 30'	100 ppm H ₂ S in N ₂	long column, leached
4-1092-SC	SNL 20'	100 ppm H ₂ S in N ₂	short column, not leached - solids characterization
4-1093-SC	SNL 30'	100 ppm H ₂ S in N ₂	short column, not leached - solids characterization
5-035-2	SNL 35' (SNLA014799)	untreated	short column, leached
5-035-3	SNL 35'	pure N ₂	short column, leached
5-035-4	SNL 35'	100 ppm H ₂ S in N ₂	short column, leached
5-035-6	SNL 35'	1000 ppm ethylene in N ₂	short column, leached
5-035-7	SNL 35'	100 ppm ethylene in N ₂	short column, leached
5-035-8	SNL 35'	100 ppm H ₂ S in N ₂ , 10% breakthrough	short column, leached
5-035-9	SNL 35'	100 ppm H ₂ S in N ₂ , 25% breakthrough	short column, leached
5-035-10	SNL 35'	100 ppm H ₂ S in N ₂ , 50% breakthrough	short column, leached
5-035-11	SNL 35'	100 ppm H ₂ S in N ₂ , >70% breakthrough	short column, leached, duplicate of 5-035-4
5-000-12	uncontaminated soil	100 ppm H ₂ S in N ₂	short column, not leached
5-000-13	uncontaminated soil	100 ppm H ₂ S in air	short column, not leached

Table 2.4. Sediment Sample Moisture Contents and Particle Densities

Sediment Sample	Moisture Content	Particle Density
uncontaminated CWL soil	5.09%	3.11 g/cm ³
15-20' interval	2.06%	2.74 g/cm ³
25-30' interval	4.22%	2.76 g/cm ³
30-35' interval	3.63%	2.77 g/cm ³

H9409032.1

Figure 2.1. Schematic of Gas Treatment System

2.3 Column Leach Procedures and Analysis of Leachate Solutions

For each of the sediment sample intervals tested, treated and untreated columns were leached with deionized water. A peristaltic pump was adjusted to provide a constant flow rate of about one pore volume per day (approximately 100 mL/day) for the duration of the tests. Eight leachate samples were collected per test over a period of 10 days. Leachate samples were weighed, and the number of column pore volumes was determined. The first leachate sample of each test was filtered to 0.45 μm ; subsequent samples were not filtered owing to the lack of visual evidence of particulate matter. Leachate samples were analyzed for pH, Cr(VI), and total Cr, and other cations and anions.

All activities associated with leachate analysis involved the use of reagent-grade chemicals and standards. Selected samples were analyzed in duplicate to provide an indication of analytical precision. Chromate concentrations in the leachate samples were measured with a spectrophotometer using the diphenylcarbazide colorimetric method per Environmental Protection Agency (EPA) SW-846 Method 7196 (EPA 1992). A detection level of 0.05 ppm (mg L^{-1}) was reported. Total Cr and other cations were determined by inductively coupled plasma (ICP) emission spectrometry per the quality-assurance requirements of SW-846 Method 6010. Sulfate and other major anions in the leachate samples were analyzed by ion chromatography per EPA Method 300.0. Solution pH determinations were performed using standardized pH electrodes. These values are judged to be accurate to ± 0.05 units.

2.4 Determination of Cr Oxidation State and Bonding Characteristics

A series of extraction and dissolution tests was conducted on ten samples collected in the borehole interval from 10 to 60 ft. Speciation of the Cr was determined using selective repetitive and sequential wet-chemical extractions as described below.

The first set of selective extractions involved three repetitive extractions of the sediments with either deionized water, 0.01 M NaH_2PO_4 buffered at pH 7, or 6 M HCl. The H_2O and PO_4 extractions were designed to measure easily and difficultly exchangeable Cr. The HCl extraction measured the total "environmentally available" fraction of Cr; that is, the Cr that could ever conceivably be released to solution. Ten grams of sediment were treated with 20 mL of extractant and placed on an orbital shaker for an overnight incubation at room temperature. Samples were removed, filtered, and the filtrates were analyzed for total Cr by inductively-coupled plasma atomic-emission spectroscopy (ICP-AES) and for Cr(VI) by the diphenylcarbazide colorimetric method (H_2O -extracted samples only).

Chromium associated with exchange sites, carbonates, amorphous oxide and silicate phases, crystalline iron and manganese oxides, organic matter, and the HF-soluble portions of the sediments was determined by a six-stage sequential series of selective extractions performed in succession on each sediment sample, based on methods described in Tessier et al. (1979) and Yanase et al. (1991). Amounts of Cr in the extracts were determined by ICP-AES. Extractions were performed in duplicate on 1-g sediment samples in 50-mL centrifuge tubes as follows:

1. **Exchangeable Cr.** Add 8 mL of 1 M MgCl_2 buffered to pH 7 with acetic acid to the sediment sample. Shake for 1 hour. Centrifuge, and remove supernate with a pipet. Save for

analysis. Add 8 mL of deionized water to the sample. Centrifuge, and discard supernate. Save washed sample for Step 2.

2. **Cr Bound to Carbonates.** Add 8 mL of 1 M NaOAc adjusted to pH 5.0 with acetic acid to sample. Shake for 5 hr. Centrifuge, and remove supernate with a pipet. Save for analysis. Add 8 mL of deionized water to the sample. Centrifuge, and discard supernate. Save washed sample for Step 3.
3. **Cr Associated with Amorphous Phases.** Add 40 mL of Tamm's solution (10.9 g/L oxalic acid + 16.1 g/L ammonium oxalate) to sample. Shake, in the dark, at room temperature, for 4 hr. Centrifuge, and remove supernate with a pipet. Save for analysis. Add 8 mL of deionized water to the sample. Centrifuge, and discard supernate. Save washed sample for Step 4.
4. **Cr Bound to Crystalline Fe and Mn Oxides.** Add 20 mL 0.04 M $\text{NH}_2\text{OH}\cdot\text{HCl}$ in 25 % (v/v) HOAc to sample. Heat at $96\pm 3^\circ\text{C}$ for 6 hr. Centrifuge, and remove supernate with a pipet. Save for analysis. Add 8 mL of deionized water to the sample. Centrifuge, and discard supernate. Save washed sample for Step 5.
5. **Cr Bound to Organic Matter.** Add 3 mL of 0.02 M HNO_3 and 5 mL of 30% H_2O_2 adjusted to a pH value of 2 with HNO_3 to sample. Heat at $85\pm 2^\circ\text{C}$ for 2 hr. Add 3 mL of 30% H_2O_2 and continue heating for 3 hr. After cooling, add 5 mL of 3.2 M NH_4OAc in 20% (v/v) HNO_3 . Dilute to 20 mL and agitate for 30 min. Centrifuge, and remove supernate with a pipet. Save for analysis. Add 8 mL of deionized water to the sample. Centrifuge, and discard supernate. Save washed sample for Step 6.
6. **Residual Cr.** Add 5 mL of 48% HF and 5 mL of deionized water to sample. Heat at 60°C for 4 hr. Centrifuge, and filter supernate, dilute to 10 mL, and analyze.

Nondestructive estimation of Cr oxidation states of selected samples was also performed by X-ray absorption near edge structure (XANES) spectroscopy using the high-intensity radiation provided by the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. Sediment samples and three aqueous 100 ng mL^{-1} Cr standards [100% Cr(VI), 100% Cr(III), and a 50:50 mixture of Cr(VI) and Cr(III)] were analyzed by XANES. This involved scanning across the Cr-K edge and measuring the fluorescent X-rays using a Lytle detector. The spectra were background-corrected and normalized and then the height of the pre-edge peak at approximately 5993 eV was used to estimate the Cr(VI) content of the samples using the standard curve of Peterson et al. (1997).

3.0 Results and Discussion

3.1 Gas Treatment of Sediment Samples

Monitoring of column-outlet gas concentrations was performed to establish the breakthrough of H_2S during treatment of the sediment samples. The H_2S breakthrough curves obtained during the treatment tests provide a means of assessing reaction progress during treatment and serve as a basis for defining treatment completion, as discussed below. Control tests were also conducted with uncontaminated CWL soil that were treated with diluted H_2S to establish a baseline. In addition, several tests were conducted that involved treatment of the 35' sediment sample with diluted ethylene.

H_2S treatment of the 20' sediment sample packed in one of the longer columns (test 4-1092-2; Figure 3.1) was conducted in two separate time periods owing to schedule constraints. The increase in the slope of the curve at 735 minutes coincides with the break taken during treatment. Although the breakthrough curve is rather poorly defined for this test, it indicates that a value of C/C_0 of 0.5 was achieved at about 12 1/2 hours (i.e., the outlet concentration of H_2S was 50 ppm while the inlet concentration was 100 ppm). Treatment was discontinued after a total treatment period of 882 min. A value of C/C_0 of 0.72 was recorded at this time.

The H_2S breakthrough curve obtained for treatment of the 30' sample that was packed in a long column is better defined (test 4-1093-2; Figure 3.1). A value of C/C_0 of 0.5 was achieved in this test at about 880 min. The sensor failed at 990 min into the treatment, however, where C/C_0 was about 0.6. Treatment was discontinued after 25 hr; a measurement of the outlet concentration of H_2S at this time indicated that C/C_0 was approximately equal to 1.0. The greater treatment time required for the 30' sediment relative to the 20' sediment is consistent with the observation that the 30' sediment contained a higher concentration of Cr(VI) than the 20' sediment (see Table 2.1 and leach analysis results presented below).

The H_2S breakthrough curves associated with treatment tests conducted in short columns are presented in Figure 3.2. These tests included treatment of the 20' (4-1092-SC), 30' (4-1093-SC), and 35' (5-035-4) sediment samples. Increasing treatment times required for the 35', 20', and 30' sediment treatment tests, respectively, were again observed to be roughly proportional to the concentration of Cr(VI) in the untreated samples as determined by the column leach results (see below).

A series of tests was also conducted with the 35' sample in short columns where different degrees of H_2S treatment were undertaken, as indicated by increasing values of C/C_0 (10%, 25%, 50%, and >70% in tests 5-035-8, 5-035-9, 5-035-10, and 5-035-11, respectively). The breakthrough curves associated with these four tests are presented in Figure 3.3.

Figure 3.4 presents the H_2S breakthrough curves for two tests conducted with an uncontaminated sediment sample from the vicinity of the Chemical Waste Landfill. Test 5-000-12 involved treatment with 100 ppm H_2S in N_2 , while test 5-000-13 involved treatment with 100 ppm H_2S in air. Note that air appears to interfere with treatment, as indicated by a significant increase in time required to achieve an equivalent degree of H_2S breakthrough.

Figure 3.1. H₂S Breakthrough Curves for Tests 4-1092-2 and 4-1093-2

Figure 3.2. H₂S Breakthrough Curves for Tests 5-035-4, 4-1092-SC, and 4-1093-SC

Figure 3.3. H₂S Breakthrough Curves for Tests 5-035-8, 5-035-9, 5-035-10, and 5-035-11

Figure 3.4. H₂S Breakthrough Curves for Tests 5-000-12 and 5-000-13

At the end of each treatment involving H_2S , the columns were purged with nitrogen. Excess H_2S was evacuated from the columns in less than 15 min (see Figures 3.2-3.4). This observation suggests that excess H_2S can be easily purged from a waste site after gas treatment, thus preventing subsequent emission of residual H_2S .

Portions of the 35' sediment were treated with 1000 ppm ethylene and 100 ppm ethylene in N_2 in tests 5-035-6 and 5-035-7, respectively. A total gas flow rate of 500 ml/min was maintained in test 5-035-6 for 360 min and 2500 mL/min was maintained in test 5-035-7 for 182 min. Monitoring of effluent gas concentrations by gas chromatographic analysis indicated essentially immediate breakthrough of ethylene in both tests, suggesting limited reaction of ethylene with the sediment matrix (data not shown).

3.2 Column Leach Test Results

The pH values of all leachate samples were in the range of 7.1 to 8.4 and were not significantly different for the untreated and treated samples (Appendixes A and B). Concentrations of all metals, except for Cr, were not present above trace levels for leachates from either untreated or treated sediment samples, and similar major cation and anion concentrations were observed in the leachate samples associated with untreated and treated samples. Similar values for concentrations of Cr(VI) and total Cr were reported for all leachate samples, indicating that essentially all of the Cr that was mobile was Cr(VI). This observation is consistent with the known chemistry of Cr (Rai et al. 1989).

The concentration of Cr(VI) in the leachate samples associated with the H_2S treatment tests and corresponding untreated tests is presented in Figures 3.5-3.8 as a function of the corresponding number of column pore volumes. Comparison of the total Cr(VI) recovered in the treated versus the untreated tests indicated that 90% or more of the Cr(VI) in the sediments was immobilized by H_2S treatment when breakthrough was continued to near completion (see discussion below and Appendix C). Figure 3.9 further illustrates that the degree of Cr(VI) immobilization is roughly proportional to breakthrough, as measured by C/C_0 .

The corresponding data for the ethylene treatment tests are presented in Figure 3.10. It is apparent that treatment of the sediment samples with dilute ethylene had essentially no effect on the amount of leachable Cr(VI).

Measurable Cr(VI) was present in the leachates of the untreated samples even after a significant number of column pore volumes had passed through the sediments. This suggests that the phases containing Cr(VI) in the 20' and 30' sediments are only moderately soluble. A soluble Cr(VI) concentration of $186 \mu g g^{-1}$ was calculated from the column leach test results for the untreated sample from the 20' interval (test 4-1092-1; Appendix C), whereas the sediment analysis indicated a Cr(VI) concentration of only $6.0 \mu g g^{-1}$ (Table 2.1). Similarly, the column leach test performed on the untreated sample from the 30' sample (test 4-1093-1) indicated a minimum of $530 \mu g Cr(VI) g^{-1}$, whereas the sediment analysis indicated a Cr(VI) concentration of only $291 \mu g g^{-1}$. This discrepancy appears to indicate incomplete Cr(VI) recovery associated with the sediment analyses performed for this constituent, as might be anticipated if the Cr(VI) phase were sparingly soluble in water. Better

Figure 3.5. Cr(VI) Concentrations of Column Leachate Samples for Tests 4-1092-1 and 4-1092-2

Figure 3.6. Cr(VI) Concentrations of Column Leachate Samples for Tests 4-1093-1 and 4-1093-2

Figure 3.7. Cr(VI) Concentrations of Column Leachate Samples for Tests 5-035-2, 5-035-3, and 5-035-4

Figure 3.8. Cr(VI) Concentrations of Column Leachate Samples for Tests 5-035-8, 5-035-9, 5-035-10, and 5-035-11

Figure 3.9. Cr(VI) Immobilization as a Function of Breakthrough for H₂S Treatment of 35' Sediment

Figure 3.10. Cr(VI) Concentrations of Column Leachate Samples for Tests 5-035-2, 5-035-6, and 5-035-7

agreement was observed for the results associated with the 35' sample where the sediment analysis indicated a Cr(VI) concentration of $112 \mu\text{g g}^{-1}$ and the untreated column leach (test 5-035-2) resulted in a value of $97.8 \mu\text{g g}^{-1}$.

Detailed scanning-electron microscopy (SEM) and electron-microprobe studies were conducted by Stein et al. (1994) on Cr-contaminated sediments from two other disposal cells in the CWL. Grain coatings containing grimaldite, CrOOH , were identified, and anhydrous CaCrO_4 and Cr(VI)-substituted gypsum were also observed. Stein et al. (1994) concluded that these alteration phases resulted from the movement of an acidic Cr- and SO_4 -rich solution through a calcareous sediment and consequent alteration of CaCO_3 and K-spar. Additional characterization of Cr(VI)-contaminated samples from several of the CWL pits was performed by Peterson et al. (1997). In these studies, XANES spectroscopy at the Cr-K edge was used to quantify the amount of Cr(VI) present from the height of the pre-edge feature. Peterson et al. (1997) noted that native magnetite present in the CWL pits was responsible for reducing much of the Cr(VI), but that in the North 60's Pit no magnetite could be identified and the Cr(VI) fractions were significantly higher than in the other pits. Analysis of one sample (3.5 m depth) from this pit by bulk-elemental, electron-microprobe, and extended X-ray absorption fine structure (XAFS) spectroscopy, suggested that most of the Cr(VI) was present as PbCrO_4 , with minor amounts present as CaCrO_4 .

The characterization studies of Stein et al. (1994) and Peterson et al. (1997) suggest that a significant portion of the Cr(VI) in the CWL sediments is present in phases that dissolve relatively slowly. In particular, the presence of PbCrO_4 , a relatively insoluble compound, may be responsible for the sluggish release behavior of Cr(VI) in the column leach tests performed on the untreated 20' and 30' samples. These mechanisms for fixation of Cr(VI) appear to be further substantiated by the faster release of Cr(VI) observed in leaching the untreated 35' sediment (Figures 3.5-3.7), which has lower Pb and SO_4 contents (Table 2.1), and better agreement between the results of the sediment analysis and the column leach test performed on the untreated sediment sample.

Low aqueous concentrations of Pb ($<0.9 \text{ mg/L}$) were measured in the samples collected during leaching of either the untreated or treated samples in tests 4-1092-1, 4-1092-2, 4-1093-1, and 4-1093-2 (Appendix B). This observation provides additional evidence that Pb occurs as a relatively insoluble phase, such as PbCrO_4 .

3.3 Chromium Oxidation State of Untreated and Treated Sediments

The results of the repetitive extractions performed on untreated sediment samples (Table 3.1) indicate that the bulk of the Cr is not exchangeable, but could potentially be released to the environment (i.e., it was dissolved by the HCl treatment). The highest concentrations of Cr were seen in the 20' and 30' samples. In general, the H_2O -extractable Cr levels determined by the diphenylcarbazide colorimetric method and by ICP-AES agreed and the levels of PO_4 -extractable Cr were the same or slightly higher than for H_2O -extractable Cr. Judging from the high amounts of Cr extracted by HCl during the third treatment, it is concluded that significant quantities of Cr remained in the sediment after leaching with water and with the 0.01 M PO_4 solution.

Table 3.1. Amounts of Cr Extracted from Contaminated Sediments by Repetitive Extractions^(a)

Depth	H ₂ O-Cr(VI) ^(b)				H ₂ O-ICP Cr				PO ₄ -ICP Cr				HCl-ICP Cr			
	#1	#2	#3	Sum	#1	#2	#3	Sum	#1	#2	#3	Sum	#1	#2	#3	Sum
-- ft --	----- µg g ⁻¹ -----															
10-15	19	11	1	31	20	2	7	29	28	1	2	31	35	9	25	69
15-20	86	10	11	107	80	35	---	115	12	63	36	111	2500	500	500	3500
20-25	1	<1	<1	1	---	<1	---	<1	21	3	2	26	3970	900	1400	6270
25-30	179	2	1	182	150	125	80	355	270	145	85	500	5100	1300	1900	8300
30-35	69	18	1	88	50	20	---	70	8	8	10	26	360	78	155	593
35-40	1	7	1	8	---	9	9	18	165	6	4	175	560	110	230	900
40-45	2	<1	<1	3	---	---	<1	<1	---	<1	---	<1	14	2	4	20
45-50	1	---	<1	1	---	---	---	---	---	---	---	---	3	2	1	6
50-55	7	4	1	12	---	---	6	6	7	<1	2	9	300	200	120	620
55-60	1	---	---	---	---	---	---	---	---	---	---	---	6	33	4	43

(a) Extractions were performed in three steps with either H₂O, 0.01 M PO₄, or 6 M HCl solutions. Data are means of two replicate samples.

(b) Determined by diphenylcarbazide.

(c) None detected.

Table 3.2. Amounts of Cr Extracted from Contaminated Sediments by Sequential Extractions^(a)

Depth	Exchangeable		Carbonates	Amorphous Oxides	Crystalline Oxides	Organic Matter	Residual	Total ^(e)
	Cr(VI) ^(b)	ICP						
-- ft --					μg g ⁻¹			
10-15	43	30	6	20	12	60	7	135
15-20	12	71	124	1710	2020	180	--- ^(d)	4110
20-25	1	---	31	1090	4380	685	70	6260
25-30	600	196	207	1970	5880	145	12	8400
30-35	96	73	26	345	155	15	8	625
35-40	17	8	27	490	395	71	11	1000
40-45	<1	---	---	---	---	13	11	24
45-50	---	---	65	---	---	---	---	65
50-55	10	6	---	355	240	---	---	600
55-60	---	---	---	250	---	---	---	250

(a) Data are means of two replicate samples.

(b) Determined by diphenylcarbazide.

(c) Using ICP data for exchangeable Cr.

(d) None detected.

The results of the sequential extractions performed on untreated sediment samples (Table 3.2) show that, with the exception of the shallowest depth, between 80 and 100% of the Cr is associated with phases that dissolve under the same conditions as amorphous and crystalline oxides. A surprisingly high fraction of the Cr was associated with the crystalline oxide phases suggesting substantial resistance to dissolution. These data agree with the repetitive-extraction data (Table 3.1), which also exhibited significant resistance of the Cr-bearing phases to dissolution. The total amounts of Cr extracted by sequential extraction agree well with the total amounts extracted by the repetitive extractions with 6 M HCl.

The amorphous-oxide extraction relies on the strong complexing ability of the oxalate ion to solubilize oxide phases, whereas the crystalline-oxide extractions rely on a combination of reduction and complexation. That such a large fraction of Cr is not removed until reduction is employed suggests that it may still be in the hexavalent state [albeit in an insoluble phase such as $\text{PbCrO}_4(\text{s})$], or in the trivalent state as part of a mixed-valence mineral such as magnetite (i.e., a spinel phase) that contains Fe(III) susceptible to reduction. These hypotheses are supported by the very high Pb and Fe concentrations in the 20' and 30' sediments (Table 2.1) and by work suggesting that initial precipitation of the Cr in the sediment occurred by reaction with magnetite (Peterson et al. 1997).

Repetitive extraction was conducted on portions of the 20' and 30' H_2S -treated samples with H_2O , 0.01 M PO_4 , or 6 M HCl to assess the amount of Cr(VI) reduced by gas treatment. The results of these analyses (Table 3.3) indicate that the $\text{H}_2\text{S}(\text{g})$ treatment lowered the amounts of H_2O - and PO_4 -soluble Cr to about 3% of their original values. Likewise, the HCl-soluble Cr levels also dropped to about 40% of their initial values. This latter observation suggests that the $\text{H}_2\text{S}(\text{g})$ treatment caused the formation of protective coatings, probably insoluble Fe, Pb, and Cu sulfides, on the surfaces of the sediment particles. There also was a slight tendency for the amounts of H_2O - and PO_4 -soluble Cr to increase with repeated extraction, suggesting that with reoxidation of these coatings, slightly more Cr was released. These trends are minor, however, and there is little doubt that the H_2S treatment is effective in decreasing the immediate amounts of Cr(VI) released.

XANES spectra obtained for untreated and treated 20' and 30' sediment samples, overlain by spectra for the aqueous Cr(VI) and Cr(III) standards, clearly demonstrate the presence of Cr(VI) in the samples and also verify that a significant reduction of Cr occurred during treatment with the dilute H_2S gas mixtures (Figure 3.11). The sediment pre-edge peak consists of two components, the primary one centered at 5993 eV and another minor component at about 5990 eV. The portion of the peak at 5990 eV is probably due to Cr(III) phases also present in the sample. A similar peak at 5990 eV can be seen in the XANES spectra of a model Cr(III) compound presented by Peterson et al. (1997) and was attributed to 1s to $3\text{d}(\text{t}_{2\text{g}})$ electronic transitions.

Accurate determination of the Cr(VI) fraction relative to total Cr is difficult to achieve without known solid standards for use in analyzing the spectra. Peterson et al. (1996, 1997) recommended use of the intensity of the pre-edge peak, to avoid complications associated with use of the absorption-edge energy (i.e., the edge energy at a normalized intensity of 0.5) in systems that may contain a number of Cr(III) and Cr(VI) phases. Indeed, the results (not shown) of calculations using the pre-edge intensity, edge-energy, and height of edge were quite different for our samples. Using the method and standard curve of Peterson et al. (1997) and the values for total Cr in Table 2.1, we obtained initial values for the Cr(VI) content of 240 and 740 $\mu\text{g g}^{-1}$ for the 20' and 30' samples, respectively. These results are 30-40% higher than those obtained by column leaching studies, as would be expected if the Cr(VI)

Table 3.3. Amounts of Cr Extracted from Contaminated Sediments by Repetitive Extractions Before and After H₂S Treatment^(a)

	H ₂ O-Cr(VI) ^(b)				H ₂ O-ICP Cr				PO ₄ -ICP Cr				HCl-ICP Cr			
Depth	#1	#2	#3	Sum	#1	#2	#3	Sum	#1	#2	#3	Sum	#1	#2	#3	Sum
--ft --	----- µg g ⁻¹ -----															
Before H ₂ S Treatment																
15-20	86	10	11	107	80	35	-- ^(c)	115	12	63	36	111	2500	500	500	3500
25-30	179	2	1	182	150	125	80	355	270	145	85	500	5100	1300	1900	8300
After H ₂ S Treatment																
15-20	<1	1	2	3	<1	1	2	3	1	1	1	3	550	360	470	1380
25-30	<1	7	6	13	3	5	3	11	<1	2	2	4	960	1300	800	3060

(a) Extractions were performed in three steps with either H₂O, 0.01 M PO₄, or 6 M HCl solutions. Data are means of two replicate samples.

(b) Determined by diphenylcarbazide.

(c) None detected.

Figure 3.11. XANES Spectra for Untreated and H₂S Treated Sediments Illustrating Use of Pre-Edge Peak (top) and Edge-Energy (bottom) to Estimate Cr(VI) Levels

was present in phases of low solubility such as PbCrO₄. After H₂S treatment, the Cr(VI) contents estimated by XANES decreased to 70 and 340 µg g⁻¹, respectively. Thus, the majority of the Cr(VI) is reduced to Cr(III) by the H₂S treatment, but a significant fraction of Cr(VI) remains in the samples after treatment. However, this fraction is not necessarily available to the environment.

3.4 Chromium Immobilization Factors Associated with Gas Treatment

Significant immobilization of Cr(VI) was achieved by gas treatment of the sediment sample obtained from the 20' interval at the test site (Table 3.4). The total amount of Cr(VI) recovered in the treated sample during column leaching was about 0.5% of that obtained from the untreated sample. Thus, approximately 99.5% of the Cr(VI) in the sediment was immobilized as a result of treatment. Similar values for concentrations of Cr(VI) and total Cr in the leachate samples indicate that essentially all of the Cr that was mobile was Cr(VI) (Appendix A). This observation is consistent with the known chemistry of Cr. The results obtained by repetitive extraction of Cr with water from the untreated and treated 20' interval indicated 97% immobilization.

A comparison of the Cr(VI) concentrations of the leachate samples obtained from the treated and untreated tests with the sediment collected in the 30' interval of the test site is also presented in Table 3.4. In this case, the total amount of Cr(VI) in the treated sample was 1.5% of that obtained from the untreated sample as determined by column leaching. This corresponds to immobilization of approximately 98.5% of the Cr(VI) originally present in the sample. Similar values for Cr(VI) and total Cr were also obtained for all leachate samples in these two tests. The repetitive-extraction procedure results indicated 93% immobilization.

Significant immobilization of Cr(VI) also was achieved by gas treatment of the 35' (SC) sediment test (Table 3.4). Immobilization of about 90% was observed based on column leaching results. The somewhat lower degree of immobilization achieved for this sample appears to be related to the shorter treatment time (and hence lower H₂S/mass ratio) employed in treating this sample relative to the treatment of the 20' and 30' samples. The shorter breakthrough time associated with the 35' sediment is attributed to lower Cr(VI), Cu, Pb, and Fe concentrations present in the 30-35' interval (Tables 2.1 and 3.4).

3.5 Reaction Mechanisms and Implications for In Situ Sediment Remediation

The primary chemical reaction of interest associated with these tests involves the reduction of Cr(VI) to Cr(III). The contaminant will subsequently precipitate as an hydroxide solid phase. The reduction and immobilization of Cr(VI) may be represented by the following generalized reaction:

Note that H₂S is converted to SO₄²⁻ in this reaction and that 3 moles of H₂S are required to reduce 8 moles of Cr(VI) to Cr(III). The products of this reaction include a minor amount of SO₄²⁻, which is not generally regarded as a contaminant of concern, and Cr(OH)₃, an insoluble and essentially nontoxic solid.

The relatively narrow width of the breakthrough curves observed in these tests imply that H₂S reacts quickly with constituents of the sediment samples. The time required to achieve breakthrough, however, exceeded that anticipated if the sole reactive agent were Cr(VI). That is, a molar ratio of S:Cr(VI) significantly greater than 3:8 was required to overcome the reactive capacity of the sediment

Table 3.4. Summary of Sediment Treatment Results

Test ID	Total Cr(VI) leached	C/C ₀	Cr(VI) Immobilized	H ₂ S/sediment ^(a)
	----- $\mu\text{g g}^{-1}$ -----		----- % -----	
20', column leach				
Untreated	186			
H ₂ S-treated	0.89	0.72	99.5	5.76×10^{-4}
20', repetitive extraction with water				
Untreated	107			
H ₂ S-treated	3	0.89	97.2	2.97×10^{-4}
30', column leach				
Untreated	530			
H ₂ S-treated	8.16	1.0	98.5	8.98×10^{-4}
30', repetitive extraction with water				
Untreated	182			
H ₂ S-treated	13	0.85	92.9	5.57×10^{-4}
35' (SC), column leach				
Untreated	97.8			
H ₂ S-treated	10.0	0.90	89.8	1.61×10^{-4}

(a) Mass ratio of H₂S to sediment used in column treatment tests.

samples towards H_2S . Other metals present in the sediment, notably Cu, will also react with H_2S and thus contribute to a delay in breakthrough time (Hesterberg et al. 1997). The time of breakthrough observed in these tests is an important parameter since it provides a practical basis for estimating the amount of gas that will be required to treat the waste site. Thus, tests such as those conducted in this study can be used to support technical planning activities associated with implementation of the H_2S -gas treatment approach to sediment remediation.

The time required to achieve complete treatment at the waste site can be estimated on the basis of the mass of contaminated sediment present, the mass of H_2S required per unit mass of sediment, and the rate of application of H_2S to the site. If the 15-35' interval is targeted for treatment and a treatment cell 30 ft in diameter is specified, the volume of sediment involved is 14,137 ft³. Assuming a bulk density of 110 lb/ft³, a total of 1.555×10^6 pounds of sediment is present in the treatment cell. The H_2S /sediment mass ratios associated with the treatment tests (Table 3.4) indicates that about 0.0004 lbs of H_2S is required to treat 1 lb of sediment. Thus, 622 lbs of H_2S is required to treat the site. If this is injected into the site as a 100 ppm H_2S mixture at a flow rate of 100 ft³/min (cfm), the time required to achieve breakthrough is about 489 days. However, since the treatment time required is inversely proportional to gas concentration and flow rate, the time associated with treatment can be reduced by increasing the H_2S concentration of the injection gas stream or by injecting at a higher flow rate (Figure 3.12). For example, treatment could be accomplished in about 6 months by injecting 300 ppm H_2S at a flow rate of 100 cfm or by injecting 100 ppm H_2S at a flow rate of 300 cfm. Approximately 4 months would be needed if 200 ppm H_2S were injected at 200 cfm. If a relatively short treatment period is desirable, injection of 400 ppm H_2S at a flow rate of 400 cfm would result in completion of treatment in about 1 month.

Figure 3.12. Predicted Treatment Time as a Function of H_2S Concentration and Gas Flow Rate for CWL Waste Site

4.0 Conclusions and Future Work

The results of laboratory testing activities indicate that treatment of Cr(VI)-contaminated sediments at the CWL site through the injection of a 100 ppm H₂S mixture in N₂ should prove effective in immobilizing Cr(VI). Observed treatment efficiencies ranged from 89.8% to 99.5% for C/C₀ values near unity. Test results also indicate that the degree of immobilization is directly proportional to C/C₀. Tests conducted with diluted mixtures of ethylene in nitrogen failed to produce measurable immobilization of Cr(VI).

Future testing activities should include laboratory treatment tests employing 100 ppm H₂S mixtures in air. This is important since application of H₂S/air mixtures would be more economically favorable than the use of H₂S/N₂ mixtures. Laboratory tests are needed, however, to better assess the degree to which O₂ in air may interfere with Cr(VI) reduction. Additional future studies should also be conducted to determine if alternatives to H₂S can be used in the treatment of Cr(VI)-contaminated sediments. Possible alternatives include acetylene, methane, hydrogen, and sulfur dioxide.

Batch extraction tests and XAS studies performed on the treated sediments suggests that protective rinds of reduced chromium oxides and, possibly, metallic sulfide phases served to prevent release of Cr(VI) observed during the short-term column water leaching tests. It was also noted that TCLP protocol may not be appropriate for assessing the Cr(VI) content of sediments owing to the potential for reduction of chromium under acidic pH conditions. It is proposed that performance of alkali digestion (SW-846 EPA Method 3060A) be undertaken in future studies to provide an estimate of total Cr(VI) contents of treated and untreated sediments. This is especially important for sediments such as those at the CWL site that contain relatively insoluble Cr(VI) solid phases.

Ongoing studies are being performed to determine the reactivity of Fe, Pb, and Cu in CWL sediments with H₂S (Hesterberg et al. 1997). XAS results obtained to date indicate that Cu is readily transformed to a sulfide phase in the CWL sediments, while Fe and Pb appear to be relatively unreactive. More of this type of work is needed in light of the implications for bioavailability and toxicity of heavy metals in sediments (Di Toro et al. 1992).

Results presented in this study and previous studies indicate the feasibility of utilizing diluted H₂S mixtures to reduce and immobilize Cr(VI) in waste site soils. Laboratory test results also serve to establish the breakthrough characteristics associated with soil treatment and thus provide a means of predicting the mass of hydrogen sulfide required to achieve site remediation. Monitoring of hydrogen sulfide breakthrough during site treatment activities is proposed as an approach for assessing treatment progress and could be used as a basis for defining completion criteria associated with gas treatment operations. A field demonstration of this approach has been proposed at a Cr(VI) waste site located at the Department of Defense (DOD) White Sands Missile Range in New Mexico (Thornton and Miller 1996). Completion of this demonstration will provide a basis for technical evaluation of in situ remediation of Cr(VI)-contaminated sediments through the injection of diluted H₂S mixtures. If a successful demonstration is completed at White Sands, it is anticipated that the approach may be applied in the remediation at the CWL and other government and commercial facilities having Cr(VI)-contaminated sediment waste sites.

5.0 References

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Appendix A

Chromium and pH Data for Column Leach Tests

Table A.1. Chromium and pH Analytical Data for Test 4-1092-1

Sample #	Cr(VI), mg/L	Cr(total), mg/L	pH
4-1092-1-1	317.42	295	7.70
4-1092-1-1 (dup)	314.76	--	--
4-1092-1-2	94.08	108	7.63
4-1092-1-3	63.78	67.8	7.81
4-1092-1-4	51.15	48	7.82
4-1092-1-5	37.86	38.6	7.87
4-1092-1-5 (dup)	--	35.5	--
4-1092-1-6	34.93	37.5	7.90
4-1092-1-7	30.81	33.2	7.83
4-1092-1-8	27.09	25.9	7.78

Table A.2. Chromium and pH Analytical Data for Test 4-1092-2

Sample #	Cr(VI), mg/L	Cr(total), mg/L	pH
4-1092-2-1	<0.05	0.07	7.66
4-1092-2-2	<0.05	0.31	7.76
4-1092-2-3	0.12	0.35	7.86
4-1092-2-4	0.41	0.66	7.83
4-1092-2-5	0.28	0.39	7.88
4-1092-2-5 (dup)	0.29	0.46	--
4-1092-2-6	0.23	0.48	7.96
4-1092-2-6 (dup)	0.24	--	--
4-1092-2-7	0.23	0.36	7.97
4-1092-2-7 (dup)	0.23	--	--
4-1092-2-8	0.20	0.34	8.00

Table A.3. Chromium and pH Analytical Data for Test 4-1093-1

Sample #	Cr(VI), mg/L	Cr(total), mg/L	pH
4-1093-1-1	728.20	713	7.61
4-1093-1-1 (dup)	766.76	--	--
4-1093-1-2	249.62	241	7.62
4-1093-1-3	173.82	171	7.80
4-1093-1-4	140.32	135	7.77
4-1093-1-5	97.56	94.6	7.78
4-1093-1-5 (dup)	--	105	--
4-1093-1-6	78.54	68.3	7.89
4-1093-1-7	70.69	76.1	7.95
4-1093-1-8	64.58	73.3	7.75

Table A.4. Chromium and pH Analytical Data for Test 4-1093-2

Sample #	Cr(VI), mg/L	Cr(total), mg/L	pH
4-1093-2-1	<0.05	0.86	8.01
4-1093-2-2	3.96	3.81	7.69
4-1093-2-3	3.24	3.12	7.83
4-1093-2-4	4.46	4.51	7.86
4-1093-2-5	3.20	3.22	7.92
4-1093-2-5 (dup)	--	2.13	--
4-1093-2-6	2.48	2.68	7.91
4-1093-2-7	2.10	2.23	7.87
4-1093-2-8	1.66	1.79	7.90

Table A.5. Chromium and pH Analytical Data for Test 5-035-2

Sample #	Cr(VI), mg/L	pH
5-035-2-1	278.05	7.78
5-035-2-2	21.74	8.10
5-035-2-3	8.83	8.20
5-035-2-3 (dup)	8.44	8.24
5-035-2-4	9.79	8.29
5-035-2-5	7.33	8.35
5-035-2-6	5.21	8.39
5-035-2-6 (dup)	5.17	8.31
5-035-2-7	4.60	8.37
5-035-2-8	4.26	8.28

Table A.6. Chromium and pH Analytical Data for Test 5-035-3

Sample #	Cr(VI), mg/L	pH
5-035-3-1	299.13	7.83
5-035-3-2	20.69	8.14
5-035-3-3	6.85	8.27
5-035-3-4	8.82	8.36
5-035-3-5	6.13	8.32
5-035-3-5 (dup)	6.11	8.32
5-035-3-6	4.19	8.32
5-035-3-7	4.07	8.40
5-035-3-7 (dup)	3.64	8.42
5-035-3-8	3.53	8.34

Table A.7. Chromium and pH Analytical Data for Test 5-035-4

Sample #	Cr(VI), mg/L	pH
5-035-4-1	2.20	8.19
5-035-4-1 (dup)	2.20	--
5-035-4-2	3.07	8.40
5-035-4-3	2.50	8.34
5-035-4-4	2.90	8.40
5-035-4-4 (dup)	2.90	8.41
5-035-4-5	2.78	8.33
5-035-4-6	2.15	8.34
5-035-4-7	3.64	8.39
5-035-4-8	2.10	8.35
5-035-4-8 (dup)	2.04	8.37

Table A.8. Chromium and pH Analytical Data for Test 5-035-6

Sample #	Cr(VI), mg/L	pH
5-035-6-1	488.34	7.87
5-035-6-2	25.14	8.04
5-035-6-3	12.34	8.10
5-035-6-4	11.19	8.15
5-035-6-5	4.70	8.06
5-035-6-6	3.95	8.36
5-035-6-6 (dup)	3.88	--
5-035-6-7	4.58	8.40
5-035-6-8	4.41	8.41

Table A.9. Chromium and pH Analytical Data for Test 5-035-7

Sample #	Cr(VI), mg/L	pH
5-035-7-1	537.58	7.88
5-035-7-2	27.23	8.26
5-035-7-3	12.46	8.14
5-035-7-4	10.26	8.02
5-035-7-5	4.76	8.16
5-035-7-6	3.80	8.32
5-035-7-7	4.58	8.43
5-035-7-8	4.12	8.41
5-035-7-8 (dup)	4.00	--

Table A.10. Chromium and pH Analytical Data for Test 5-035-8

Sample #	Cr(VI), mg/L	pH
5-035-8-1	229.14	7.55
5-035-8-1 (dup)	223.95	7.55
5-035-8-2	15.91	7.96
5-035-8-3	9.81	8.01
5-035-8-4	7.11	7.98
5-035-8-5	4.95	7.94
5-035-8-5 (dup)	4.78	7.95
5-035-8-6	3.86	7.96
5-035-8-7	3.60	7.98
5-035-8-8	3.49	8.03
5-035-8-9	3.36	8.05
5-035-8-9 (dup)	3.30	--
5-035-8-10	2.28	8.06

Table A.11. Chromium and pH Analytical Data for Test 5-035-9

Sample #	Cr(VI), mg/L	pH
5-035-9-1	139.59	7.45
5-035-9-2	13.18	8.02
5-035-9-2 (dup)	13.18	8.03
5-035-9-3	7.73	8.06
5-035-9-4	6.33	8.02
5-035-9-5	4.52	8.01
5-035-9-6	3.47	7.93
5-035-9-6 (dup)	3.47	7.94
5-035-9-7	3.23	7.97
5-035-9-8	3.17	8.02
5-035-9-9	2.99	7.98
5-035-9-10	2.23	8.01
5-035-9-10 (dup)	2.20	--

Table A.12. Chromium and pH Analytical Data for Test 5-035-10

Sample #	Cr(VI), mg/L	pH
5-035-10-1	86.77	7.27
5-035-10-2	10.97	8.12
5-035-10-3	6.82	8.03
5-035-10-3 (dup)	6.95	8.04
5-035-10-4	6.08	7.99
5-035-10-5	4.41	8.03
5-035-10-6	3.52	7.93
5-035-10-7	3.11	7.87
5-035-10-7 (dup)	3.23	7.90
5-035-10-8	3.13	8.05
5-035-10-9	3.02	7.95
5-035-10-10	2.19	8.04

Table A.13. Chromium and pH Analytical Data for Test 5-035-11

Sample #	Cr(VI), mg/L	pH
5-035-11-1	5.30	7.08
5-035-11-2	2.84	7.91
5-035-11-3	3.13	7.93
5-035-11-4	3.15	7.93
5-035-11-4 (dup)	3.08	7.94
5-035-11-5	2.98	7.92
5-035-11-6	2.51	7.91
5-035-11-7	2.40	7.94
5-035-11-8	2.59	7.99
5-035-11-8 (dup)	2.56	--
5-035-11-9	2.64	8.05
5-035-11-10	2.07	8.02

Appendix B

Column Leachate ICP and IC Analytical Data

Constituent	1092-1-1	1092-1-2	1092-1-3	1092-1-4	1092-1-5	1092-1-5 (Duplicate)	1092-1-6	1092-1-7	1092-1-8
Ag, uG/ML	< 1.20000E-02	2.90000E-02	2.80000E-02	1.16000E-01	1.44000E-01	< 1.20000E-02	3.40000E-02	3.60000E-02	4.60000E-02
Ba, uG/ML	< 7.90000E-03	2.50000E-02	2.10000E-02	6.40000E-02	6.80000E-02	< 7.90000E-03	1.90000E-02	2.60000E-02	2.40000E-02
Be, uG/ML	4.00000E-03	5.00000E-03	5.00000E-03	7.00000E-03	8.00000E-03	3.00000E-03	5.00000E-03	5.00000E-03	5.00000E-03
Cr, uG/ML	2.95000E+02	1.08000E+02	6.78000E+01	4.80000E+01	3.86000E+01	3.55000E+01	3.75000E+01	3.32000E+01	2.59000E+01
Ca, uG/ML	6.00000E+02	6.28000E+02	6.34000E+02	6.21000E+02	9.51000E+02	5.95000E+02	6.75000E+02	5.98000E+02	1.87000E+02
Cd, uG/ML	< 3.60000E-03	5.00000E-03	5.00000E-03	2.60000E-02	2.90000E-02	< 3.60000E-03	4.00000E-03	6.00000E-03	< 3.60000E-03
Cl, PPM	5.89930E+02	< 4.54000E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00
Cu, uG/ML	4.28000E-01	2.41000E-01	1.97000E-01	2.43000E-01	2.56000E-01	1.12000E-01	1.66000E-01	1.75000E-01	1.68000E-01
Pb, uG/ML	< 3.75000E-02	4.10000E-02	< 3.75000E-02	2.46000E-01	3.95000E-01	< 3.75000E-02	6.10000E-02	8.29999E-02	6.90000E-02
F, PPM	< 3.39000E+00	2.13040E+01	1.87430E+01	1.77900E+01	1.61870E+01	1.55900E+01	1.49310E+01	1.42280E+01	1.51660E+01
Fe, uG/ML	< 2.05000E-02	2.30000E-02	< 2.05000E-02	5.80000E-02	6.20000E-02	< 2.05000E-02	< 2.05000E-02	2.40000E-02	< 2.05000E-02
Al, uG/ML	< 1.00100E-01	2.06000E-01	1.79000E-01	6.79000E-01	8.10000E-01	< 1.00100E-01	1.60000E-01	1.90000E-01	2.14000E-01
K, uG/ML	1.83000E+02	8.61000E+01	4.85000E+01	3.29000E+01	2.04000E+01	1.87000E+01	1.03000E+01	9.59000E+00	9.96000E+00
Mg, uG/ML	1.72000E+02	8.90000E+01	5.43000E+01	3.60000E+01	2.32000E+01	2.22000E+01	1.74000E+01	1.21000E+01	8.23000E+00
Mn, uG/ML	5.50000E-02	4.80000E-02	4.30000E-02	4.80000E-02	5.60000E-02	3.80000E-02	4.90000E-02	5.00000E-02	4.60000E-02
Na, uG/ML	9.87000E+02	7.43000E+01	4.07000E+01	2.87000E+01	1.89000E+01	1.82000E+01	1.47000E+01	1.32000E+01	1.17000E+01
Ni, uG/ML	< 4.80000E-02	< 4.80000E-02	< 4.80000E-02	1.09000E-01	1.35000E-01	< 4.80000E-02	5.40000E-02	7.70000E-02	6.10000E-02
NO2, PPM	< 2.98000E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E	< 2.97700E+01	< 2.97700E+01
NO3, PPM	< 3.79300E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01
PO4, PPM	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	1.56589E+02	< 3.27800E+01	< 3.27800E+01	1.27931E+03	< 3.27800E+01
Sb, uG/ML	5.63000E+00	1.98000E+00	1.27000E+00	1.66000E+00	1.79000E+00	< 8.48900E-01	1.04000E+00	< 8.48900E-01	< 8.48900E-01
SO4, PPM	3.10893E+03	1.87698E+03	1.60303E+03	1.68454E+03	1.32104E+03	1.54258E+03	1.52364E+03	1.48096E+03	1.44048E+03
Zn, uG/ML	1.30000E-02	1.60000E-02	1.20000E-02	2.90000E-02	3.50000E-02	8.00000E-03	1.80000E-02	2.40000E-02	1.50000E-02

Constituent	1092-2-1	1092-2-2	1092-2-3	1092-2-4	1092-2-5	1092-2-5(Duplicate)	1092-2-6	1092-2-7	1092-2-8
Ag, uG/ML	< 1.20000E-02	1.04000E-01	3.20000E-02	1.98000E-01	2.54000E-01	2.47000E-01	3.34000E-01	1.43000E-01	1.15000E-01
Ba, uG/ML	7.50000E-02	9.29999E-02	4.90000E-02	1.04000E-01	1.13000E-01	1.36000E-01	1.50000E-01	8.70000E-02	9.20000E-02
Be, uG/ML	5.00000E-03	9.00000E-03	6.00000E-03	9.00000E-03	8.00000E-03	6.70000E-02	1.10000E-02	8.00000E-03	6.60000E-02
Cr, uG/ML	6.60000E-02	3.07000E-01	3.51000E-01	6.64000E-01	3.91000E-01	4.59000E-01	4.75000E-01	3.55000E-01	3.44000E-01
Ca, uG/ML	6.38000E+02	7.48000E+02	6.22000E+02	7.06000E+02	6.44000E+02	6.21000E+02	7.09000E+02	6.70000E+02	6.40000E+02
Cd, uG/ML	4.00000E-03	2.00000E-02	8.00000E-03	3.70000E-02	4.80000E-02	5.80000E-02	6.50000E-02	3.10000E-02	2.80000E-02
Cl, PPM	6.16348E+02	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00
Cu, uG/ML	2.32000E+01	8.37000E-01	5.25000E-01	4.24000E-01	3.24000E-01	3.02000E-01	3.71000E-01	2.29000E-01	2.06000E-01
Pb, uG/ML	< 3.75000E-02	2.46000E-01	7.39999E-02	5.04000E-01	6.57000E-01	6.18000E-01	8.49000E-01	4.05000E-01	2.20000E-01
F, PPM	2.54450E+01	1.29950E+01	1.36390E+01	1.22490E+01	1.14000E+01	1.19280E+01	1.10230E+01	1.11060E+01	1.13410E+01
Fe, uG/ML	< 2.05000E-02	6.40000E-02	2.30000E-02	9.69999E-02	1.20000E-01	1.33000E-01	1.54000E-01	7.80000E-02	8.10000E-02
Al, uG/ML	< 1.00100E-01	6.35000E-01	2.17000E-01	1.15600E+00	1.47800E+00	1.40700E+00	1.97000E+00	9.90000E-01	6.96000E-01
K, uG/ML	1.84000E+02	8.22000E+01	5.31000E+01	2.60000E+01	1.72000E+01	1.43000E+01	1.20000E+01	7.74000E+00	1.06000E+01
Mg, uG/ML	1.75000E+02	7.53000E+01	4.63000E+01	2.45000E+01	1.72000E+01	1.51000E+01	9.87000E+00	8.12000E+00	9.09600E+00
Mn, uG/ML	3.05800E+00	1.94900E+00	1.58400E+00	1.13100E+00	7.16000E-01	8.79000E-01	8.97000E-01	8.75000E-01	8.48000E-01
Na, uG/ML	9.67000E+02	6.67000E+01	4.16000E+01	2.22000E+01	1.76000E+01	1.56000E+01	1.33000E+01	1.21000E+01	1.12000E+01
Ni, uG/ML	1.44000E-01	1.65000E-01	7.00000E-02	2.13000E-01	2.71000E-01	3.31000E-01	3.79000E-01	1.39000E-01	1.14000E-01
NO2, PPM	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01
NO3, PPM	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01
PO4, PPM	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01
Sb, uG/ML	< 8.48900E-01	1.40700E+00	< 8.48900E-01	2.06100E+00	2.51500E+00	1.93100E+00	2.97300E+00	9.51000E-01	< 8.48900E-01
SO4, PPM	3.64614E+03	1.84878E+03	1.68352E+03	1.57315E+03	1.53265E+03	1.49739E+03	1.48222E+03	1.44625E+03	1.34298E+03
Zn, uG/ML	8.00000E-02	3.10000E-02	1.90000E-02	3.80000E-02	4.60000E-02	6.10000E-02	6.00000E-02	2.60000E-02	3.30000E-02

Constituent	1093-1-1	1093-1-2	1093-1-3	1093-1-4	1093-1-5	1093-1-5 (Duplicate)	1093-1-6	1093-1-7	1093-1-8
Ag, uG/ML	3.10000E-02	< 1.20000E-02	6.70000E-02	7.89999E-02	< 1.20000E-02	6.10000E-02	< 1.20000E-02	6.00000E-02	1.52000E-01
Ba, uG/ML	< 7.90000E-03	< 7.90000E-03	2.30000E-02	3.80000E-02	< 7.90000E-03	4.10000E-02	< 7.90000E-03	3.90000E-02	7.20000E-02
Be, uG/ML	5.00000E-03	5.00000E-03	6.00000E-03	6.00000E-03	4.00000E-03	7.00000E-03	4.00000E-03	7.00000E-03	1.00000E-02
Cr, uG/ML	7.13000E+02	2.41000E+02	1.71000E+02	1.35000E+02	9.46000E+01	1.05000E+02	6.83000E+01	7.61000E+01	7.33000E+01
Ca, uG/ML	8.35000E+02	6.74000E+02	6.62000E+02	6.67000E+02	6.66000E+02	6.42000E+02	7.56000E+02	7.68000E+02	7.70000E+02
Cd, uG/ML	< 3.60000E-03	< 3.60000E-03	7.00000E-03	1.60000E-02	< 3.60000E-03	1.20000E-02	< 3.60000E-03	1.10000E-02	3.10000E-02
Cl, PPM	6.10527E+02	< 4.52000E+00	< 4.54200E+00	< 4.54000E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00
Cu, uG/ML	5.91000E-01	2.82000E-01	2.82000E-01	2.62000E-01	1.40000E-01	2.16000E-01	1.25000E-01	2.08000E-01	2.68000E-01
Pb, uG/ML	< 3.75000E-02	< 3.75000E-02	< 3.75000E-02	8.00000E-02	< 3.75000E-02	1.24000E-01	< 3.75000E-02	9.69999E-02	3.16000E-01
F, PPM	3.65200E+01	2.01770E+01	1.74940E+01	1.66790E+01	1.39930E+01	1.46710E+01	1.33030E+01	1.33310E+01	1.22720E+01
Fe, uG/ML	< 2.05000E-02	< 2.05000E-02	3.20000E-02	4.80000E-02	< 2.05000E-02	3.40000E-02	< 2.05000E-02	< 2.05000E-02	5.70000E-02
Al, uG/ML	< 1.00100E-01	< 1.00100E-01	3.08000E-01	3.81000E-01	< 1.00100E-01	3.83000E-01	< 1.00100E-01	2.57000E-01	8.10000E-01
K, uG/ML	2.89000E+02	1.43000E+02	7.05000E+01	3.98000E+01	2.23000E+01	2.40000E+01	1.39000E+01	1.29000E+01	1.25000E+01
Mg, uG/ML	2.38000E+02	1.13000E+02	7.13000E+01	4.48000E+01	2.62000E+01	2.70000E+01	1.53000E+01	1.46000E+01	1.35000E+01
Mn, uG/ML	1.22000E-01	9.89999E-02	9.29999E-02	9.39999E-02	9.39999E-02	1.05000E-01	9.29999E-02	1.34000E-01	1.58000E-01
Na, uG/ML	1.31000E+03	6.05000E+01	3.66000E+01	2.67000E+01	1.90000E+01	1.77000E+01	1.33000E+01	1.42000E+01	1.38000E+01
Ni, uG/ML	5.60000E-02	< 4.80000E-02	9.10000E-02	1.03000E-01	< 4.80000E-02	6.20000E-02	< 4.80000E-02	9.00000E-02	1.48000E-01
NO2, PPM	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01
NO3, PPM	< 3.79200E+01	< 3.79200E+01	< 3.79300E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01
PO4, PPM	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01
Sb, uG/ML	1.29000E+01	4.17000E+00	3.63200E+00	2.75200E+00	1.32100E+00	1.80300E+00	8.81000E-01	1.20200E+00	2.27400E+00
SO4, PPM	3.98334E+03	3.87705E+03	1.69100E+03	1.72491E+03	1.52212E+03	1.49247E+03	1.42480E+03	1.48471E+03	1.41882E+03
Zn, uG/ML	2.70000E-02	1.50000E-02	2.20000E-02	2.20000E-02	1.80000E-02	2.90000E-02	< 4.00000E-03	1.80000E-02	3.70000E-02

Constituent	1093-2-1	1093-2-2	1093-2-3	1093-2-4	1093-2-5	1093-2-5 (Duplicate)	1093-2-6	1093-2-7	1093-2-8
Ag, uG/ML	< 1.20000E-02	2.30000E-02	4.30000E-02	5.90000E-02	< 1.20000E-02	2.31000E-01	3.50000E-02	3.22000E-01	2.70000E-01
Ba, uG/ML	< 7.90000E-03	4.30000E-02	4.70000E-02	4.90000E-02	9.00000E-03	1.17000E-01	4.10000E-02	1.43000E-01	1.23000E-01
Be, uG/ML	< 2.00000E-04	5.00000E-03	5.00000E-03	5.00000E-03	3.00000E-03	7.00000E-03	6.00000E-03	1.00000E-02	1.00000E-02
Cr, uG/ML	8.63000E-01	3.80800E+00	3.11500E+00	4.51000E+00	3.21900E+00	2.13400E+00	2.68100E+00	2.22800E+00	1.78700E+00
Ca, uG/ML	6.15000E+02	6.41000E+02	8.73000E+02	8.13000E+02	6.17000E+02	6.51000E+02	6.28000E+02	7.32000E+02	8.06000E+02
Cd, uG/ML	< 3.60000E-03	< 3.60000E-03	9.00000E-03	1.00000E-02	< 3.60000E-03	5.50000E-02	7.00000E-03	7.30000E-02	6.00000E-02
Cl, PPM	6.48898E+02	8.26700E+00	< 4.55420E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00	< 4.54200E+00
Cu, uG/ML	3.14000E+01	9.79999E-02	1.03000E-01	1.13000E-01	4.10000E-02	2.11000E-01	9.10000E-02	3.00000E-01	2.56000E-01
Pb, uG/ML	< 3.75000E-02	< 3.75000E-02	1.59000E-01	1.67000E-01	< 3.75000E-02	7.16000E-01	1.07000E-01	8.97000E-01	7.71000E-01
F, PPM	2.72080E+01	1.46410E+01	1.31750E+01	1.27650E+01	1.13600E+01	1.14450E+01	1.07500E+01	1.18690E+01	1.04340E+01
Fe, uG/ML	< 2.05000E-02	< 2.05000E-02	3.10000E-02	3.50000E-02	< 2.05000E-02	1.04000E-01	1.50000E-02	1.28000E-01	1.14000E-01
Al, uG/ML	< 1.00100E-01	1.78000E-01	3.47000E-01	3.67000E-01	< 1.00100E-01	1.29500E+00	2.11000E-01	1.81700E+00	1.48600E+00
K, uG/ML	2.53000E+02	1.10000E+02	7.22000E+01	4.92000E+01	2.97000E+01	2.59000E+01	2.02000E+01	1.40000E+01	6.20000E+00
Mg, uG/ML	1.74000E+02	8.77000E+01	6.47000E+01	5.15000E+01	3.23000E+01	2.50000E+01	2.01000E+01	1.34000E+01	1.46000E+01
Mn, uG/ML	9.27000E-01	7.46000E-01	6.19000E-01	5.92000E-01	4.61000E-01	3.12000E-01	4.62000E-01	4.26000E-01	4.14000E-01
Na, uG/ML	1.86000E+03	8.26000E+01	4.55000E+01	3.22000E+01	2.09000E+01	1.74000E+01	1.57000E+01	1.24000E+01	1.11000E+01
Ni, uG/ML	< 4.80000E-02	< 4.80000E-02	5.50000E-02	7.70000E-02	< 4.80000E-02	2.28000E-01	6.50000E-02	3.35000E-01	2.81000E-01
NO2, PPM	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01	< 2.97700E+01
NO3, PPM	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01	< 3.79200E+01
PO4, PPM	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01	< 3.27800E+01
Sb, uG/ML	< 8.48900E-01	4.40000E-01	< 8.48900E-01	< 8.48900E-01	< 8.48900E-01	2.11200E+00	< 8.48900E-01	2.75900E+00	2.11200E+00
SO4, PPM	3.83450E+03	1.96611E+03	1.79899E+03	1.72551E+03	1.60971E+03	1.58636E+03	1.55572E+03	1.50878E+03	1.53406E+03
Zn, uG/ML	< 4.00000E-03	6.00000E-03	1.60000E-02	1.50000E-02	< 4.00000E-03	3.80000E-02	1.50000E-02	5.40000E-02	4.40000E-02

B.5

Constituent	1122-1-1 (deionized water blank)
Ag, uG/ML	< 1.20000E-02
Ba, uG/ML	< 7.90000E-03
Be, uG/ML	< 2.00000E-04
Cr, uG/ML	< 9.70000E-03
Ca, uG/ML	< 4.81000E-02
Cd, uG/ML	< 3.60000E-03
Cl, PPM	< 4.54200E+00
Cu, uG/ML	< 1.20000E-02
Pb, uG/ML	< 3.75000E-02
F, PPM	< 3.39400E+00
Fe, uG/ML	< 2.05000E-02
Al, uG/ML	< 1.00100E-01
K, uG/ML	< 6.03000E-01
Mg, uG/ML	5.00000E-03
Mn, uG/ML	< 1.80000E-03
Na, uG/ML	< 5.03000E-02
Ni, uG/ML	< 4.80000E-02
NO2, PPM	< 2.97700E+01
NO3, PPM	< 3.79200E+01
PO4, PPM	< 3.27800E+01
Sb, uG/ML	< 8.48900E-01
SO4, PPM	< 3.86300E+01
Zn, uG/ML	1.10000E-02

Appendix C

Column Treatment Test Descriptions and Assessment of Cr(VI) Recovery

Test 4-1092-1

In this test, a sample of untreated, contaminated soil collected from a depth of 20 ft at the test site was leached using deionized water. The mass of leachable chromium recovered from the soil sample indicates that the soil contains at least 186 ppm Cr(VI).

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
4-1092-1-1	117.54	0.00 - 1.17	316.09 ^(a)	37.18
4-1092-1-2	122.93	1.17 - 2.39	94.08	11.57
4-1092-1-3	101.48	2.39 - 3.40	63.78	6.47
4-1092-1-4	132.05	3.40 - 4.71	51.15	6.75
4-1092-1-5	305.30	4.71 - 7.75	37.86	11.56
4-1092-1-6	112.09	7.75 - 8.87	34.93	3.92
4-1092-1-7	106.31	8.87 - 9.92	30.81	3.28
4-1092-1-8	110.22	9.92 - 11.02	27.09	2.99

Total Cr(VI) = 83.68 mg

(a) Average of duplicate analyses.

Sample wt = 450.89 g

Column pore volume = 100.53 mL

Concentration of total leachable hexavalent chromium in soil =

$$\frac{\text{Total Cr(VI)}}{\text{sample wt}} = \frac{83.68 \text{ mg Cr(VI)}}{450.89 \text{ g, soil}} = 186 \frac{\text{mg}}{\text{kg}} \text{ or } 186 \text{ ppm Cr(VI)}$$

Test 4-1092-2

In this test, a sample collected at a depth of 20 ft at the test site was treated with 100 ppm H₂S diluted in N₂ and leached with deionized water. A total of 0.40 mg of Cr(VI) was leached from 451.25 g of soil. This corresponds to 0.48% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
4-1092-2-1	119.91	0.00 - 1.34	<0.05	0.01
4-1092-2-2	127.50	1.34 - 2.76	<0.05	0.01
4-1092-2-3	108.58	2.76 - 3.98	0.12	0.01
4-1092-2-4	139.95	3.98 - 5.54	0.41	0.06
4-1092-2-5	824.14	5.54 - 9.21	0.285 ^(a)	0.23
4-1092-2-6	120.31	9.21 - 10.55	0.235 ^(a)	0.03
4-1092-2-7	114.61	10.55 - 11.83	0.23 ^(a)	0.03
4-1092-2-8	119.15	11.83 - 13.17	0.20	0.02

Total Cr(VI) = 0.40 mg

(a) Average of duplicate analyses.

Sample wt = 451.25 g

Column pore volume = 89.50 mL

Total Cr(VI) originally present in soil =

451.25 g, soil x $\frac{0.186 \text{ mg Cr(VI)}}{\text{g, soil}}$ = 83.93 mg, Cr(VI)

% Cr(VI) leached from soil following treatment =

$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{0.40 \text{ mg}}{83.93 \text{ mg}} \times 100 = 0.48\%$

Test 4-1093-1

In this test, a sample of untreated, contaminated soil collected from a depth of 30 ft at the test site was leached using deionized water. The mass of leachable chromium recovered from the soil sample indicates that the soil contains at least 530 ppm Cr(VI).

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
4-1093-1-1	128.41	0.00 - 1.18	747.48 ^(a)	95.98
4-1093-1-2	130.32	1.18 - 2.38	249.62	32.53
4-1093-1-3	111.16	2.38 - 3.41	173.82	19.32
4-1093-1-4	144.84	3.41 - 4.74	140.32	20.32
4-1093-1-5	340.00	4.74 - 7.87	97.56	33.17
4-1093-1-6	125.78	7.87 - 9.03	78.54	9.88
4-1093-1-7	119.89	9.03 - 10.14	70.69	8.48
4-1093-1-8	125.45	10.14 - 11.29	64.58	8.10

Total Cr(VI) = 227.79 mg

(a) Average of duplicate analyses.

Sample wt = 430.01 g

Column pore volume = 108.56 mL

Concentration of total leachable hexavalent chromium in soil =

$$\frac{\text{Total Cr(VI)}}{\text{sample wt}} = \frac{227.79 \text{ mg Cr(VI)}}{430.01 \text{ g, soil}} = 530 \frac{\text{mg}}{\text{kg}} \text{ or } 530 \text{ ppm Cr(VI)}$$

Test 4-1093-2

In this test, a sample collected at a depth of 30 ft at the test site was treated with 100 ppm H₂S in N₂ and leached with deionized water. A total of 3.46 mg of Cr(VI) was leached from 424.02 g of soil. This corresponds to 1.54% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
4-1093-2-1	119.48	0.00 - 1.04	<0.05	0.01
4-1093-2-2	131.97	1.04 - 2.19	3.96	0.52
4-1093-2-3	112.90	2.19 - 3.18	3.24	0.37
4-1093-2-4	146.28	3.18 - 4.46	4.46	0.65
4-1093-2-5	353.90	4.46 - 7.54	3.20	1.13
4-1093-2-6	127.39	7.54 - 8.66	2.48	0.32
4-1093-2-7	121.09	8.66 - 9.71	2.10	0.25
4-1093-2-8	125.46	9.71 - 10.81	1.66	0.21

Total Cr(VI) = 3.46 mg

Sample wt = 424.02 g

Column pore volume = 114.58 mL

Total Cr(VI) originally present in soil =

424.02 g, soil x $\frac{0.530 \text{ mg Cr(VI)}}{\text{g, soil}}$ = 224.73 mg, Cr(VI)

% Cr(VI) leached from soil following treatment =

$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{3.46 \text{ mg}}{224.73 \text{ mg}} \times 100 = 1.54\%$

Test 4-1092-SC

In this test, 262.60 g of soil collected at a depth of 20 ft at the test site was treated with 100 ppm H_2S in N_2 . The treated sample was utilized for solids characterization studies.

Test 4-1093-SC

In this test, 248.68 g of soil collected at a depth of 30 ft at the test site was treated with 100 ppm H_2S in N_2 . The treated soil was utilized for solids characterization studies.

Test 5-035-2

In this test, a sample of untreated, contaminated soil collected from a depth of 35 ft at the test site was leached using deionized water. The mass of leachable chromium recovered from the soil sample indicates that the soil contains at least 98 ppm Cr(VI).

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-2-1	62.66	0.00 - 1.18	278.05	17.42
5-035-2-2	65.64	1.18 - 2.41	21.74	1.43
5-035-2-3	86.25	2.41 - 4.03	8.64 ^(a)	0.75
5-035-2-4	92.28	4.03 - 5.77	9.79	0.90
5-035-2-5	269.97	5.77 - 10.85	7.33	1.98
5-035-2-6	90.49	10.85 - 12.55	5.19 ^(a)	0.47
5-035-2-7	87.77	12.55 - 14.20	4.60	0.40
5-035-2-8	78.42	14.20 - 15.67	4.26	0.33

Total Cr(VI) = 23.68 mg

(a) Average of duplicate analyses.

Sample wt = 242.18 g

Column pore volume = 53.18 mL

Concentration of total leachable hexavalent chromium in soil =

$$\frac{\text{Total Cr(VI)}}{\text{sample wt}} = \frac{23.68 \text{ mg Cr(VI)}}{242.18 \text{ g, soil}} = 97.8 \frac{\text{mg}}{\text{kg}} \text{ or } 97.8 \text{ ppm Cr(VI)}$$

Test 5-035-3

In this test, a sample of untreated, contaminated soil collected from a depth of 35 ft at the test site was treated with nitrogen gas for 4 hr and leached using deionized water. A total of 23.82 mg of Cr(VI) was leached from 243.47 g of soil. This corresponds to 100.0% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-3-1	60.73	0.00 - 1.14	299.13	18.17
5-035-3-2	68.13	1.14 - 2.42	20.69	1.41
5-035-3-3	89.42	2.42 - 4.09	6.85	0.61
5-035-3-4	96.28	4.09 - 5.90	8.82	0.85
5-035-3-5	282.66	5.90 - 11.20	6.12 ^(a)	1.73
5-035-3-6	95.61	11.20 - 12.99	4.19	0.40
5-035-3-7	92.62	12.99 - 14.73	3.86 ^(a)	0.36
5-035-3-8	82.47	14.73 - 16.28	3.53	0.29

Total Cr(VI) = 23.82 mg

(a) Average of duplicate analyses.

Sample wt = 243.47 g

Column pore volume = 53.32 mL

Total Cr(VI) originally present in soil =

$$243.47 \text{ g, soil} \times \frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}} = 23.81 \text{ mg, Cr(VI)}$$

% Cr(VI) leached from soil following treatment =

$$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{23.82 \text{ mg}}{23.81 \text{ mg}} \times 100 = 100.0\%$$

Test 5-035-4

In this test, a sample collected at a depth of 35 ft at the test site was treated with 100 ppm H₂S in nitrogen and leached with deionized water. A total of 2.37 mg of Cr(VI) was leached from 236.94 g of soil. This corresponds to 10.2% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-4-1	58.21	0.00 - 1.04	2.20 ^(a)	0.13
5-035-4-2	68.72	1.04 - 2.26	3.07	0.21
5-035-4-3	90.42	2.26 - 3.88	2.50	0.23
5-035-4-4	97.57	3.88 - 5.62	2.90 ^(a)	0.28
5-035-4-5	286.00	5.62 - 10.72	2.78	0.80
5-035-4-6	96.93	10.72 - 12.45	2.15	0.21
5-035-4-7	93.70	12.45 - 14.12	3.64	0.34
5-035-4-8	83.61	14.12 - 15.62	2.07 ^(a)	0.17

Total Cr(VI) = 2.37 mg

(a) Average of duplicate analyses.

Sample wt = 236.94 g

Column pore volume = 56.04 mL

Total Cr(VI) originally present in soil =

236.94 g, soil x $\frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}}$ = 23.17 mg, Cr(VI)

% Cr(VI) leached from soil following treatment =

$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{2.37 \text{ mg}}{23.17 \text{ mg}} \times 100 = 10.2\%$

Test 5-035-6

In this test, a sample collected at a depth of 35 ft at the test site was treated with 1000 ppm ethylene in nitrogen at a flowrate of 500 mL/min for 360 min and leached with deionized water. A total of 28.65 mg of Cr(VI) was leached from 219.93 g of soil. This corresponds to 133% of the chromate originally present in the soil (based on chromate leached during test 5-035-2).

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-6-1	44.30	0.00 - 0.72	488.34	21.63
5-035-6-2	90.08	0.72 - 2.17	25.14	2.26
5-035-6-3	98.56	2.17 - 3.77	12.34	1.22
5-035-6-4	90.58	3.77 - 5.23	11.19	1.01
5-035-6-5	292.45	5.23 - 9.96	4.70	1.37
5-035-6-6	91.43	9.96 - 11.44	3.84 ^(a)	0.35
5-035-6-7	91.01	11.44 - 12.91	4.58	0.42
5-035-6-8	88.77	12.91 - 14.35	4.41	0.39

Total Cr(VI) = 28.65 mg

(a) Average of duplicate analyses.

Sample wt = 219.93 g

Column pore volume = 61.83 mL

Total Cr(VI) originally present in soil =

$$219.93 \text{ g, soil} \times \frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}} = 21.51 \text{ mg, Cr(VI)}$$

% Cr(VI) leached from soil following treatment =

$$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{28.65 \text{ mg}}{21.51 \text{ mg}} \times 100 = 133\%$$

Test 5-035-7

In this test, a sample collected at a depth of 35 ft at the test site was treated with 100 ppm ethylene in nitrogen at a flowrate of 2500 mL/min for 182 min and leached with deionized water. A total of 29.10 mg of Cr(VI) was leached from 219.51 g of soil. This corresponds to 135.5% of the chromate originally present in the soil (based on chromate leached during test 5-035-2).

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-7-1	40.97	0.00 - 0.67	537.58	22.02
5-035-7-2	88.85	0.67 - 2.11	27.23	2.42
5-035-7-3	97.92	2.11 - 3.69	12.46	1.22
5-035-7-4	89.37	3.69 - 5.14	10.26	0.92
5-035-7-5	293.03	5.14 - 9.90	4.76	1.39
5-035-7-6	90.97	9.90 - 11.37	3.80	0.35
5-035-7-7	90.66	11.37 - 12.85	4.58	0.42
5-035-7-8	88.56	12.85 - 14.28	4.06 ^(a)	0.36

Total Cr(VI) = 29.10 mg

(a) Average of duplicate analyses.

Sample wt = 219.51 g

Column pore volume = 61.58 mL

Total Cr(VI) originally present in soil =

219.51 g, soil x $\frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}}$ = 21.47 mg, Cr(VI)

% Cr(VI) leached from soil following treatment =

$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{29.10 \text{ mg}}{21.47 \text{ mg}} \times 100 = 135.5\%$

Test 5-035-8

In this test, a sample collected at a depth of 35 ft at the test site was treated with 100 ppm H₂S in nitrogen until 10% breakthrough was achieved. The treated soil column was then leached with deionized water. A total of 20.92 mg of Cr(VI) was leached from 243.84 g of soil. This corresponds to 87.7% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-8-1	65.00	0.00 - 1.23	226.55 ^(a)	14.73
5-035-8-2	88.72	1.23 - 2.92	15.91	1.41
5-035-8-3	74.54	2.92 - 4.33	9.81	0.73
5-035-8-4	101.88	4.33 - 6.27	7.11	0.72
5-035-8-5	254.85	6.27 - 11.10	4.87 ^(a)	1.24
5-035-8-6	92.56	11.10 - 12.86	3.86	0.36
5-035-8-7	87.95	12.86 - 14.53	3.60	0.32
5-035-8-8	90.50	14.53 - 16.25	3.49	0.32
5-035-8-9	91.00	16.25 - 17.98	3.33 ^(a)	0.30
5-035-8-10	346.86	17.98 - 24.90	2.28	0.79

Total Cr(VI) = 20.92 mg

(a) Average of duplicate analyses.

Sample wt = 243.84 g

Column pore volume = 52.68 mL

Total Cr(VI) originally present in soil =

243.84 g, soil x $\frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}}$ = 23.85 mg, Cr(VI)

% Cr(VI) leached from soil following treatment =

$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{20.92 \text{ mg}}{23.85 \text{ mg}} \times 100 = 87.7\%$

Test 5-035-9

In this test, a sample collected at a depth of 35 ft at the test site was treated with 100 ppm H₂S in nitrogen until 25% breakthrough was achieved. The treated soil column was then leached with deionized water. A total of 14.98 mg of Cr(VI) was leached from 242.52 g of soil. This corresponds to 63.2% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-9-1	66.30	0.00 - 1.24	139.59	9.25
5-035-9-2	91.39	1.24 - 2.96	13.18 ^(a)	1.20
5-035-9-3	77.24	2.96 - 4.41	7.73	0.60
5-035-9-4	105.61	4.41 - 6.39	6.33	0.67
5-035-9-5	265.16	6.39 - 11.36	4.52	1.20
5-035-9-6	96.43	11.36 - 13.17	3.47 ^(a)	0.33
5-035-9-7	91.63	13.17 - 14.89	3.23	0.30
5-035-9-8	94.34	14.89 - 16.66	3.17	0.30
5-035-9-9	94.89	16.66 - 18.44	2.99	0.28
5-035-9-10	378.79	18.44 - 25.54	2.22 ^(a)	0.84

Total Cr(VI) = 14.98 mg

(a) Average of duplicate analyses.

Sample wt = 242.52 g

Column pore volume = 53.32 mL

Total Cr(VI) originally present in soil =

242.52 g, soil x $\frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}}$ = 23.72 mg, Cr(VI)

% Cr(VI) leached from soil following treatment =

$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{14.98 \text{ mg}}{23.72 \text{ mg}} \times 100 = 63.2\%$

Test 5-035-10

In this test, a sample collected at a depth of 35 ft at the test site was treated with 100 ppm H₂S in nitrogen until 50% breakthrough was achieved. The treated soil column was then leached with deionized water. A total of 9.95 mg of Cr(VI) was leached from 236.19 g of soil. This corresponds to 43.1% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-10-1	58.26	0.00 - 1.03	86.77	5.06
5-035-10-2	85.35	1.03 - 2.54	10.97	0.94
5-035-10-3	71.18	2.54 - 3.80	6.89 ^(a)	0.49
5-035-10-4	96.43	3.80 - 5.50	6.08	0.59
5-035-10-5	240.50	5.50 - 9.76	4.41	1.06
5-035-10-6	86.80	9.76 - 11.29	3.52	0.31
5-035-10-7	82.41	11.29 - 12.75	3.17 ^(a)	0.26
5-035-10-8	84.38	12.75 - 14.24	3.13	0.26
5-035-10-9	84.33	14.24 - 15.73	3.02	0.25
5-035-10-10	337.24	15.73 - 21.70	2.19	0.74

Total Cr(VI) = 9.95 mg

(a) Average of duplicate analyses.

Sample wt = 236.19 g

Column pore volume = 56.53 mL

Total Cr(VI) originally present in soil =

$$236.19 \text{ g, soil} \times \frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}} = 23.10 \text{ mg, Cr(VI)}$$

% Cr(VI) leached from soil following treatment =

$$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{9.95 \text{ mg}}{23.10 \text{ mg}} \times 100 = 43.1\%$$

Test 5-035-11

In this test, a sample collected at a depth of 35 ft at the test site was treated with 100 ppm H₂S in nitrogen until 74% breakthrough was achieved. The treated soil column was then leached with deionized water. A total of 3.86 mg of Cr(VI) was leached from 241.69 g of soil. This corresponds to 16.3% of the chromate originally present in the soil.

Note: Leachate samples were weighed and converted to volume units based on the assumption that 1 mL = 1 g. Cr(VI) refers to hexavalent chromium concentration of leachate samples as determined by spectrophotometric analysis.

Sample #	Leachate Volume, mL	# Column Pore Volumes	Cr(VI), mg/L	Cr(VI), mg
5-035-11-1	66.96	0.00 - 1.28	5.30	0.35
5-035-11-2	93.67	1.28 - 3.07	2.84	0.27
5-035-11-3	79.52	3.07 - 4.58	3.13	0.25
5-035-11-4	109.13	4.58 - 6.67	3.12 ^(a)	0.34
5-035-11-5	275.94	6.67 - 11.93	2.98	0.82
5-035-11-6	100.53	11.93 - 13.85	2.51	0.25
5-035-11-7	95.77	13.85 - 15.68	2.40	0.23
5-035-11-8	98.69	15.68 - 17.56	2.58 ^(a)	0.25
5-035-11-9	99.58	17.56 - 19.46	2.64	0.26
5-035-11-10	401.20	19.46 - 27.12	2.07	0.83

Total Cr(VI) = 3.86 mg

(a) Average of duplicate analyses.

Sample wt = 241.69 g

Column pore volume = 52.39 mL

Total Cr(VI) originally present in soil =

$$241.69 \text{ g, soil} \times \frac{0.0978 \text{ mg Cr(VI)}}{\text{g, soil}} = 23.64 \text{ mg, Cr(VI)}$$

% Cr(VI) leached from soil following treatment =

$$\frac{\text{Total Cr(VI) leached from soil}}{\text{Total Cr(VI) originally present in soil}} \times 100 = \frac{3.86 \text{ mg}}{23.64 \text{ mg}} \times 100 = 16.3\%$$

Test 5-000-12

In this test, 207.82 g of uncontaminated soil collected near the test site was treated with 100 ppm H_2S in N_2 . The treated sample was utilized for solids characterization studies.

Test 5-000-13

In this test, 213.27 g of uncontaminated soil collected near the test site was treated with 100 ppm H_2S in air. The treated soil was utilized for solids characterization studies.

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