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7. Abstract

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This test plan contains requirements, instructions, and procedures for testing activities being conducted to evaluate and develop an in situ chemical treatment approach for chromate and U(VI) contaminated soil and groundwater.

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1.0 PURPOSE

This test plan provides specific guidance for conducting activities during FY93 for the U.S. Department of Energy (DOE) In Situ Remediation Integrated Program as described in Technical Task Plan (TTP) RL431001. The objective of these activities is to assess the potential for the *in situ* immobilization of metal and radionuclide contaminants in soils through a treatment approach whereby reactive gases are vented through the region of contaminated soil.

A laboratory testing approach is described in this plan that will provide the basis for evaluating this remediation approach. In these tests, chromate (chromium (VI) or Cr(VI)) will be reduced to less soluble and less toxic Cr(III) solids in an unsaturated soil matrix through the introduction of hydrogen sulfide (H₂S) gas diluted in nitrogen. The effectiveness of the hydrogen sulfide treatment in chemically reducing and immobilizing uranyl (uranium (VI) or U(VI)) to U(IV) in soil also will be tested. In addition, the effects of different soil types, variations in moisture content, and incidental nitrate (NO₃⁻) concentrations on the immobilization of chromium and uranium will be evaluated. Several concentrations of hydrogen sulfide gas mixtures will also be employed in these tests to verify that low concentrations of hydrogen sulfide can effectively immobilize chromium and uranium. The effectiveness of immobilization of chromium and uranium resulting from the reduction step will be assessed by leaching the treated soil. This information will provide data required for determining whether this approach can achieve specified remediation standards. This data is also needed for designing larger scale tests and field demonstration systems.

2.0 PROJECT SCOPE AND OBJECTIVES OF TESTING ACTIVITIES

This test plan identifies all tests to be performed and the associated testing parameters, schedules, and data collection requirements. Also identified are the organizations responsible for the several tasks involved in this work and the locations where these tasks shall be conducted.

A procedure is included for preparing the chromate-, uranyl-, and nitrate-contaminated soils used in testing activities. Included in this plan is the design of the gas treatment system and a procedure for operating this system. All of the types of data to be recorded during the gas treatment tests are identified with specific instructions for performing the associated calculations. Following gas treatment, soil column leach tests will be performed to determine the extent to which chromium and uranium have been immobilized by the treatment process. This plan defines testing parameters associated with the leach tests. Column leach tests will be performed in accordance with procedure GEL-22, "Leaching Saturated Soil and Sediment Samples Using the Column Method" (WHC 1990a). GEL-22 includes the data collection requirements and data sheets used in the column tests and describes aspects of packing the column, in addition to performing the leach test.

Attention to safety requirements is important during execution of these activities since hydrogen sulfide gas, and chromate and uranium solutions and solids are health hazards. Specific precautions are included in this plan that address requirements associated with using these chemical agents.

The chemical reactions associated with these tests involve the reduction of the contaminant (chromium or uranium) to a lower oxidation state. The contaminant will subsequently precipitate as a hydroxide solid phase. This may be illustrated for chromate by the following reaction:



Note that hydrogen sulfide is converted to sulfate in this reaction and that 3 moles of hydrogen sulfide are required to reduce 8 moles of Cr(VI) to Cr(III). Since sulfate is not generally regarded as a contaminant of concern and chromium hydroxide is an insoluble and relatively nontoxic solid, it can be concluded that it is unlikely that a significant quantity of hazardous byproducts will be generated by the gas treatment approach. Any excess hydrogen sulfide may be removed from the soil by purging with nitrogen or air. The reaction indicates, however, that the pH of soil water will be affected by the treatment process. This could result in the mobilization of certain soil constituents. It is also possible, though unlikely, that nitrate present in the soil treatment tests may be converted to nitrite. The leachate samples obtained in the treatment tests will be analyzed to evaluate changes in pH and to identify any undesirable reaction products generated during the treatment process.

Three uncontaminated soil samples will be used in this study. They are designated herein as the Hanford soil, the Sandia soil, and the Fernald soil. The Hanford soil will be collected from an uncontaminated location near the 100-D Area of the Hanford Site. The Sandia soil sample has been provided by staff associated with the Mixed Waste Landfill Integrated Demonstration (MWLID) at Sandia National Laboratory. The Fernald soil has been provided by FERMCO/DOE staff at the Fernald Site. Chromium, uranium, and nitrate are designated as the contaminants of concern for tests involving the Hanford soil and will be added to the soil in a mixture. The Fernald soil will be treated in the same manner as the Hanford soil, to provide a basis of comparison. Only chromium will be added to the Sandia soil, as chromium is the major contaminant of concern at the MWLID waste site.

Soil sampling data recently obtained from the 100-D Area of the Hanford Site indicates that chromium is present at levels of 100 ppm and above at several waste sites. Furthermore, Sandia staff have indicated that a chromium-contaminated site associated with the MWLID has concentration levels as high as 500 ppm. Thus, a value of 200 ppm hexavalent chromium is representative of contaminated soils at these sites and will be added to the soils to be treated. Section 4.2 provides further justification for contaminant concentration levels chosen for these tests.

Several concentrations of nitrate are designated in tests for the Hanford soils to provide information regarding the degree to which nitrate might interact with hydrogen sulfide and thus potentially restrict the effectiveness of treatment.

The contaminated soils will be treated with hydrogen sulfide gas diluted in nitrogen. Two concentrations of hydrogen sulfide have been specified for testing, 2,000 ppm and 100 ppm. Sufficient gas will be flushed through the contaminated soil samples during the treatment step to attain a molar ratio of $H_2S:Cr(VI)$ of 10. Testing results reported by Thornton et al. (1991) indicate that a 2,700 ppm mixture of hydrogen sulfide in nitrogen applied at a molar ratio of 20 results in about 95% immobilization of chromium for a typical Hanford Site soil containing 5 ppm hexavalent chromium. Thus the results of the tests described here will indicate if 2,000 ppm and 100 ppm hydrogen sulfide mixtures applied at a molar ratio of only 10 can produce similar levels of treatment effectiveness in soils containing 200 ppm hexavalent chromium and 200 ppm uranium. Successful treatment with the 100 ppm gas mixture is especially important in that it will demonstrate that a mixture containing a relatively low concentration of hydrogen sulfide may be employed. This will enhance the acceptability of the gas treatment approach since it will reduce safety and environmental concerns associated with utilizing hydrogen sulfide *in situ* remediation activities (Sax and Lewis 1989).

The scope of work defined in this test plan is restricted to bench scale testing activities and is consistent with FY93 project activities identified in TTP RL431001. The primary objective of this work is to document the effectiveness of the treatment process on Cr(VI) and U(VI) contaminated soils using hydrogen sulfide gas mixtures of several concentrations, and to determine the effect of variations in soil type, moisture content, and nitrate concentrations on treatment effectiveness. This information is needed to support scale-up of the technology and to provide an initial basis for estimating the cost of employing this technology.

As indicated in TTP RL431001, larger scale (pilot) tests will be undertaken in FY94 with contaminated soils from specified waste sites. The results of the pilot tests will provide a better basis for scaling up to a field demonstration and for estimating operating costs associated with the technology. In particular, reaction rates will be determined in the pilot tests by monitoring gas composition and changes in soil temperature, for example. This information can be utilized to assess reaction progress and thus predict more accurately the amount of gas and treatment time required to achieve adequate treatment. System design aspects such as modeling of gas flow through unsaturated soils, and control and removal of treatment gases will also be addressed in the pilot tests. Detailed description of the pilot tests will be provided in future revisions to the technical task plan and by a test plan that will define FY94 activities.

3.0 REQUIREMENTS

3.1 SAFETY

Laboratory personnel will comply with the Westinghouse Hanford Company (WHC) Chemical Hygiene Plan (WHC 1992) and other internal WHC industrial safety requirements. Special safety requirements identified in this test plan include: (1) hydrogen sulfide gas use, (2) chromate and uranium solutions handling, (3) and chromate- and uranium-contaminated soil handling. The hydrogen sulfide gas cylinder will be placed in a fume hood and the outlet to

the soil column will be secured directly beneath the vent from the hood during the treatment process to minimize the risk of inhalation or contact with the hydrogen sulfide/nitrogen gas mixture. Operations involving the primary chromate and uranium standards will be conducted in a hood with the operator wearing protective rubber gloves. The operator shall also pack the columns with chromate- and/or uranium-contaminated soil under a hood to eliminate the possibility of breathing contaminated airborne particles that may be released during this activity.

Material Safety Data Sheets (MSDS) and Sax and Lewis (1989) shall be utilized as references for working with the chemical materials used in these tests. The MSDS numbers for the reagents identified for the testing activities are listed in Table 1. Hazard Communications training will be conducted prior to commencing soil treatment activities involving hydrogen sulfide.

Table 1. MSDS Numbers for Reagents.

Na_2CrO_4	1486
$\text{UO}_2(\text{NO}_3)_2 \cdot 6(\text{H}_2\text{O})$	2783
H_2S	1259
NaNO_3	1506

3.2 QUALITY ASSURANCE/QUALITY CONTROL AND DATA RECORDING

Data and sample control activities shall be performed in accordance with quality assurance requirements specified in the Environmental Investigations and Site Characterization Manual (WHC 1989). Records associated with this work shall be available for review by representatives of the Environmental Engineering Support Group.

All data collected during the course of these testing activities shall be recorded on approved data sheets or in controlled laboratory notebooks issued through Document Control per procedure 12.1, Controlled Distribution of Unclassified Documents (WHC 1990b). Data sheets shall be affixed into the laboratory notebooks. These records shall be reviewed and approved by supervisory personnel within the performing organizations in a timely manner and shall be organized and stored under controlled conditions maintained by the performing organizations. These records shall include identification of all key measuring devices and associated calibration records.

Analyses of the chromium-, uranium-, and nitrate-containing solutions shall be obtained through the 222-S Analytical Laboratory to verify the concentrations of chromium, uranium, and nitrate used and to check the stability of the solutions. A sample of the Hanford Site groundwater utilized for column leaching shall also be analyzed for cations (by inductively coupled

plasma spectrometry, ICP), anions (by ion chromatography, IC), total organic carbon (TOC), total inorganic carbon (TIC), uranium, Cr(VI), and pH. Leach columns and associated equipment shall be thoroughly cleaned before each test and at least one column shall be reserved for blank tests involving uncontaminated soil samples (Section 5.0, tests 1, 8, and 13).

The reference numbers for the procedures used for chemical analyses are presented below. The revision number for the particular procedure used will correspond to the latest version.

<u>Analysis</u>	<u>Procedure number</u>
ICP	LA-505-151
IC	LA-533-105
Uranium	LA-925-106
TOC	LA-344-105
TIC	LA-622-102
Cr(VI)	LA-265-101
pH	LA-212-102

Measurements and information associated with gas treatment of soil are to be recorded on Gas Treatment Data Sheets (Figure 1). Tests shall be conducted by personnel from Process Chemistry laboratories and/or Geotechnical Engineering to verify the flow rate measurements of the flowmeters for the gases being used. Data collection requirements associated with column leaching activities are identified in procedure GEL-22, "Leaching Saturated Soil and Sediment Samples Using the Column Method" (WHC 1990a), and associated approved procedures utilized by Geotechnical Engineering. Controlled notebooks shall be utilized to record observations or data that are not routinely entered into data sheets.

3.3 REPORTING

All records generated during testing activities shall be assembled as a data package by each performing organization (Process Chemistry laboratories and Geotechnical Engineering). These data packages shall be transmitted to the Environmental Engineering Group and shall be accompanied by letter reports describing the work performed. This information shall be kept in a project file and maintained by the Environmental Engineering Support Group and shall be dispositioned by the Environmental Engineering Support Group after completion of the project per requirements of WHC (1990b). The results and conclusions obtained from the activities described in this test plan shall be compiled and documented in a project report co-authored by representatives of the following organizations: (1) Environmental Engineering Support Group, (2) Process Chemistry Laboratories, and (3) Geotechnical Engineering. This report shall be completed by September 30, 1993, and transmitted to representatives of the DOE In Situ Remediation Integrated Program.

Sample/specimen number _____ Page _____ of _____

Column #: _____ Test #: _____

Performed by _____ Date _____

Carrier gas type _____ Treatment gas type _____

Flow rate A (carrier gas) _____ mL/min
 Flowmeter A ID: _____ Flowmeter scale reading: _____

Carrier gas delivery pressure, P _____ psig + 14.7 psi = _____ psia

Flow rate B (treatment gas) _____ mL/min
 Flowmeter B ID: _____ Flowmeter scale reading: _____

Total flow rate = flow rate A + flow rate B = _____ mL/min

Percent treatment gas in mixture = $100(\text{flow rate B} / \text{total flow rate}) = \text{_____} \%$

Total mass of soil _____ g
 Concentration of Cr(VI) = _____ $\mu\text{g/g}$
 Concentration of U(VI) = _____ $\mu\text{g/g}$
 Concentration of NO_3^- = _____ $\mu\text{g/g}$

Total Cr(VI) = total mass of soil x [Cr(VI)] = _____ μg

Total U(VI) = total mass of soil x [U(VI)] = _____ μg

Total NO_3^- = total mass of soil x [NO_3^-] = _____ μg

$\mu\text{moles of Cr(VI)} = \text{total Cr(VI)} / 51.996 = \text{_____} \mu\text{mol}$

$\mu\text{moles of U(VI)} = \text{total U(VI)} / 238.03 = \text{_____} \mu\text{mol}$

$\mu\text{moles of NO}_3^- = \text{total NO}_3^- / 62.005 = \text{_____} \mu\text{mol}$

Treatment initiated (HH:MM) _____ Treatment ended (HH:MM) _____

Duration of sample treatment, T, _____ min

Flow rate B corrected to 1 atm = $(\text{flow rate B})(P/14.7\text{psi}) = \text{_____} \text{ mL/min}$

Total volume of treatment gas, V = $(\text{flow rate B})_{\text{corr}} \times T = \text{_____} \text{ mL}$

Micromoles of treatment gas = $(1000 \times V) / (22.4 \mu\text{L}/\mu\text{mol}) = \text{_____} \mu\text{mol}$

Mole ratio gas:contaminant

$\frac{\mu\text{mol gas}}{\mu\text{mol Cr(VI)}} = \text{_____}$ $\frac{\mu\text{mol gas}}{\mu\text{mol U(VI)}} = \text{_____}$ $\frac{\mu\text{mol gas}}{\mu\text{mol NO}_3^-} = \text{_____}$

All data are accurately and completely recorded. The test operator was trained and used calibrated instruments.

Checked by _____ Date _____

Figure 1. Gas Treatment Data Sheet.

4.0 PROCEDURE

4.1 GENERAL

The goal of the tests described in this test plan is to determine if chromate and hexavalent uranium (U(VI)) can be immobilized to less-soluble reduced species by reaction with hydrogen sulfide in a variety of soil types with varying moisture content and in the presence of varying concentrations of nitrate as a co-contaminant. Steps involved in this activity include:

1. Preparing chromate- and U(VI)-contaminated soil.
2. Packing contaminated soil into leach columns.
3. Treating the soil with hydrogen sulfide gas mixtures.
4. Performing leach tests.
5. Analyzing leachate solutions.
6. Integrating and interpreting results.

The Environmental Engineering Support Group shall be responsible for project management activities and shall act as the lead in Step 6. Geotechnical Engineering and Process Chemistry laboratories jointly shall be responsible for steps 1, 2, 3, and 4 and shall also be responsible for submitting selected leachate samples to the 222-S Analytical Laboratory (Step 5).

4.2 PREPARATION OF CHROMATE- AND U(VI)-CONTAMINATED SOIL AND PACKING OF THE LEACH COLUMNS

The objective of this activity is to generate artificially contaminated soils containing 200 ppm Cr(VI) and 200 ppm U(VI). These concentrations are about twice that of cleanup levels identified in guidance documents (e.g., WAC 173-340 and Table K-1 of WHC 1988) and can be considered as representative of moderately contaminated soils. Approximately 100 ppm nitrate will also be present in the soils containing uranium, since a nitrate salt of uranium will be used in preparing the soil. Additional nitrate-contaminated soils shall also be prepared which contain 5,000 ppm nitrate as the sodium salt for use in selected tests. The objective of the latter tests is to provide a basis for assessing the extent to which nitrate may interact with hydrogen sulfide.

Note that protective rubber or vinyl gloves shall be worn while working with chromate- or uranium-bearing solutions or soils. The contaminated soil must be located in a hood while mixing or column packing activities are undertaken to eliminate the potential of breathing contaminated particles. The activities described in this section will be conducted by Geotechnical Engineering and Process Chemistry Laboratory personnel in the 377 Laboratory.

The steps in preparing the chromate- and uranium-contaminated soils and in packing the soils in the columns are:

1. Weigh 1 kg of dried soil into a beaker or shallow pan.
2. Transfer the weighed soil sample to a hood.

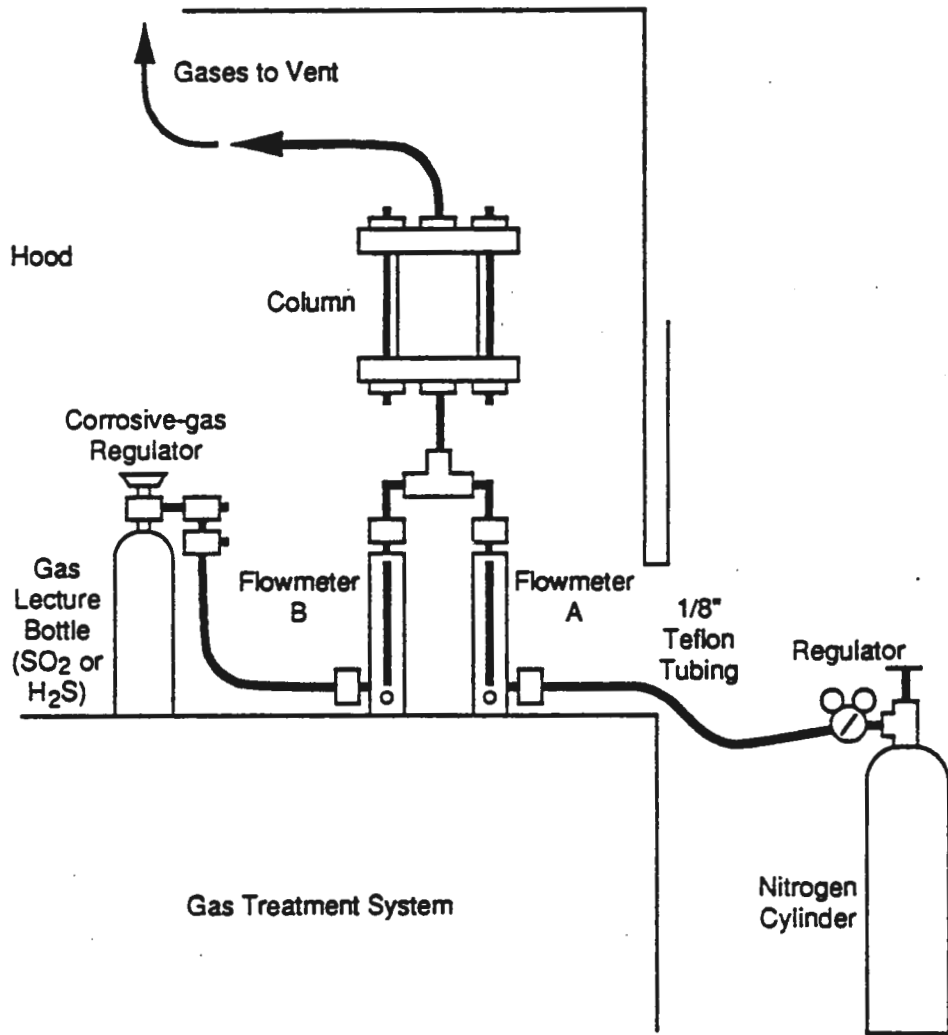
3. For soils to be tested for immobilization of 200 ppm chromium, add 50 g of a sodium chromate solution containing 4,000 ppm Cr(VI) to the soil sample and mix thoroughly. For soils to be tested for immobilization of 200 ppm uranium, add 50 g of a uranium nitrate solution containing 4,000 ppm U(VI) to the soil sample and mix thoroughly. For soils to be tested for the effect of additional nitrate (5,000 ppm total), add 98 g of a sodium nitrate solution containing 50,000 ppm nitrate to the soil sample and mix thoroughly.
4. Allow the Cr(VI) or U(VI) wetted sample to dry for 15 hours (overnight) within the hood. Application of heat should not be attempted since this may overdry the soil and result in chemical or mineralogical changes (Bartlett and James, 1979). A portion of the sample will be tested per GEL-14 (WHC 1990a) to obtain an estimate of the actual moisture content.
5. Pack the contaminated soil in a leach column following GEL-22, Section 6.3.1 (WHC 1990).
6. Complete the assembly of the column per GEL-22 and remove the column from the hood.
7. Weigh the assembled column following GEL-22.

The Column Leach - Compacted Sample Data Sheet (Figure 1 of GEL-22) is completed while preparing the contaminated soil and column packing. The data sheet is a record of the weight and pore volume of the sample. These data are required to complete the activities described in the following section. Note that the particle density of the soil also must be determined (see WHC 1990a).

4.3 GAS TREATMENT

The components and configuration of the gas treatment system are illustrated in Figure 2. The primary function of the gas treatment system is to prepare a known gas mixture and provide a flow of this mixture at a known, controlled rate. The reactive gas (hydrogen sulfide) is mixed with an inert carrier gas (nitrogen) and delivered to the packed soil column. The reactive gas is introduced to the soil column in a hood to eliminate possible inhalation. The gas treatment will be conducted by Process Chemistry laboratories and Geotechnology Group personnel in the 377 Laboratory.

The nitrogen source is a standard size compressed gas cylinder and regulator. The regulator must be multi-stage and capable of maintaining a continuous flow of nitrogen gas at a delivery pressure of less than 10 psi. The nitrogen source is connected to flowmeter A (see Figure 2) by 1/8 in. O.D. teflon tubing (teflon is a trademark of E.I. DuPont De Nemours). Flowmeter A regulates the flow of nitrogen and is connected to a mixing tee by teflon tubing. The nitrogen flow rate depends on the test as described in Section 5.0.



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Figure 2. Schematic Illustration of Gas Treatment System (not to scale).

A gas lecture bottle (pure hydrogen sulfide) or small cylinder (1% hydrogen sulfide in nitrogen) shall be used as a source for the hydrogen sulfide gas. The hydrogen sulfide must be vented into a hood during soil treatment to prevent exposure of the operator to toxic fumes. A corrosive gas regulator shall be utilized in association with the hydrogen sulfide source as specified by the manufacturer. The regulator must be capable of providing a delivery pressure of less than 10 psi. The hydrogen sulfide gas source is connected to flowmeter B (Figure 2) by teflon tubing. This flowmeter regulates the flow rate of the hydrogen sulfide gas (suggested range: 1-10 ml/min) and is connected to the mixing tee opposite the connection to the nitrogen gas source. Since brass corrodes in the presence of hydrogen sulfide, brass fittings must not be used in any part of the apparatus contacting the hydrogen sulfide gas. Stainless steel and teflon are compatible with hydrogen sulfide gas and are acceptable materials of construction for the system.

Steps in the gas treatment of the contaminated soil follow:

1. Assemble the equipment as shown in Figure 2 and attach the leach column containing the contaminated soil.
2. Open the main valve of the nitrogen cylinder and adjust the regulator outlet to obtain a pressure of several psi.
3. Increase the flow rate of nitrogen through flowmeter A to achieve the flow rate required for the particular test (note: nitrogen pressure should not exceed 10 psi). Adjust the regulator as necessary to achieve a stable nitrogen flow rate. Record the nitrogen flow rate and delivery pressure on the Gas Treatment Data Sheet (Figure 1).
4. Open the main valve of the hydrogen sulfide gas lecture bottle or cylinder and open the regulator valve slightly. Adjust the outlet and flowmeter B to achieve the desired flow rate. Record the hydrogen sulfide gas flow rate and the current time on the Gas Treatment Data Sheet.
5. At the end of the treatment period, close the main valve of the hydrogen sulfide gas lecture bottle and record the current time on the Gas Treatment Data Sheet. The nitrogen should continue to flow an additional minute to purge residual hydrogen sulfide gas from the system and then the main valve of the nitrogen cylinder should be closed.
6. Disconnect the leach column from the treatment line and remove the column from the hood.

The gas mixtures used in the treatment step will be prepared by adjusting flowmeters A and B. The actual gas flow rates utilized in the tests will be measured as accurately as possible. The data shall be recorded on the Gas Treatment Data Sheet (Figure 1). Calibration documentation and information regarding conversion of meter readings to flow rates shall be obtained from the manufacturer of the flowmeters.

4.4 PERFORMANCE OF COLUMN LEACH TESTS

Column leach tests shall be performed according to procedure GEL-22, "Leaching Saturated Soil and Sediment Samples Using the Column Method" (WHC 1990a) by Geotechnical Engineering and Process Chemistry Laboratory personnel. Specific requirements associated with column leach tests performed under this test plan are provided in the following paragraphs and in Section 5.0. An illustration of the column leaching system is presented in Figure 3.

The primary objective of performing the column leach tests is to assess the degree of immobilization of chromium and uranium attained by the hydrogen sulfide treatment. The degree of success of the treatment will be based on the amount of chromate and total chromium, and the amount of uranium measured in the leachate. Leach tests will be performed on uncontaminated and untreated, contaminated and untreated, and uncontaminated but treated (by hydrogen sulfide) soils to establish a baseline for comparison of the treatment approaches.

Characterized uncontaminated groundwater from the Hanford Site unconfined aquifer will be the leaching agent in the tests involving the Hanford soil. Deionized water will be the leaching agent for the tests involving the Sandia and Fernald soils. The selection of deionized water for the latter is based on the assumption that the pore fluids present at the Sandia and Fernald waste sites will be of low total dissolved solids content and close to neutral in pH. The groundwater at the Hanford Site is also of low total dissolved solids content and typically has a pH of about 7.5 to 8, but has concentrations of cations and anions that may not be similar to that of pore fluids associated with Sandia and Fernald soils.

The leaching solutions will be delivered to the column by a peristaltic pump. The pump flow rate shall be adjusted to deliver about a pore volume of leachate per day. Leachate samples shall consist of the first, second, and third column pore volumes (approximately 115 ml each). A fourth sample consisting of several more column pore volumes will also be collected (i.e., accumulated over the weekend). The Column Leach Effluent Collection Data Sheet (procedure GEL-22, Figure 3 of WHC 1990a) shall be used to record sampling times and volumes.

Leachate samples collected during the leach tests shall be analyzed for the primary solutes of interest (pH, chromate, total chromium, uranium, and nitrate). In addition, the first leachate sample collected in each test shall be analyzed for all cations and anions. Each leachate sample will be filtered through 0.45 μm pore-size filter and transferred to a polyethylene bottle. The filtered sample will be analyzed by inductively coupled plasma for total chromium and other cations (LA-505-151), by laser fluorimetry for total uranium (LA-925-106), and by ion chromatography for nitrate and other anions (LA-533-105) using the 222-S Analytical Laboratory. Hexavalent chromium will be analyzed spectrophotometrically in the Geotechnical Engineering 377 Laboratory by use of the diphenylcarbazide colorimetric indicator (procedure GEL-25 of WHC 1990a). The pH values of the leachate samples also will be determined in the 377 Laboratory (procedure GEL-26 of WHC 1990a).

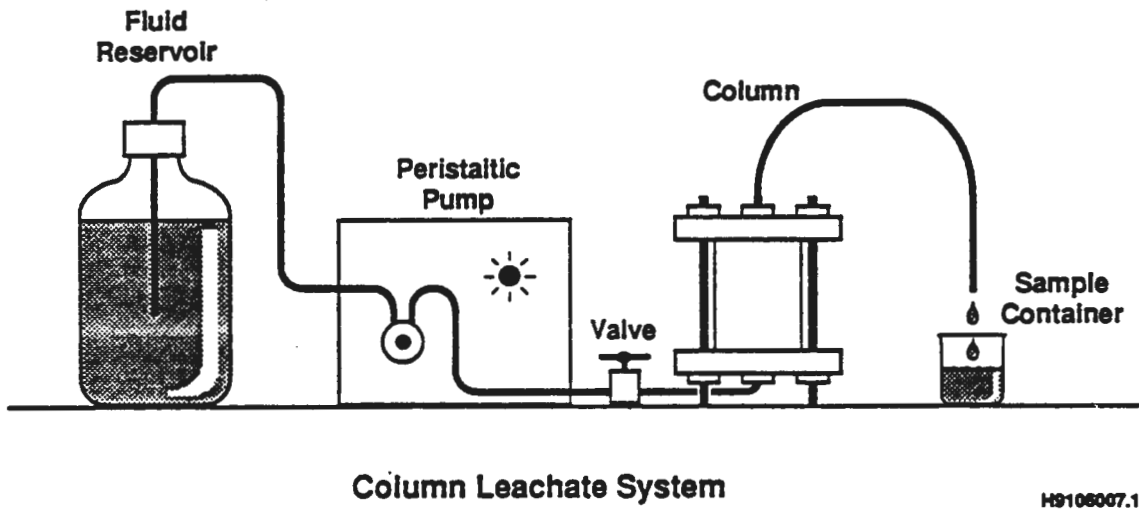


Figure 3. Schematic Illustration of Column Leachate System (not to scale).

5.0 IDENTIFICATION AND DESCRIPTION OF TESTS TO BE PERFORMED

An uncontaminated sample of sand from the Hanford formation will be collected and used in conducting tests related to chromate, uranium, and nitrate contamination of 100 Area soils. This sample shall be collected as indicated in procedure EII 5.2, Soil and Sediment Sampling (WHC 1989). A second uncontaminated soil sample has been obtained from the Sandia Laboratory near Albuquerque, New Mexico, and will be used to study the effect of hydrogen sulfide treatment on chromate-contaminated soils associated with the MWLID. An uncontaminated soil sample has also been obtained from the Fernald Site which will be used to assess the effectiveness of hydrogen sulfide treatment on uranium-contaminated soil. The Fernald soil is anticipated to behave in a manner unlike that of the Hanford soil since the Fernald soil is expected to have a different mineralogy and texture and may have a higher organic carbon content. Hydrogen sulfide treatment tests will also be conducted on chromate-contaminated Fernald soil to assess the extent to which the character of this soil may influence treatment effectiveness relative to that of the Hanford and Sandia chromate-contaminated soil samples.

Descriptions of the tests to be performed are provided in the test matrix presented in Table 2. The proposed test schedule is presented in Table 3.

Tests 1, 8, and 13 involve leaching uncontaminated Hanford, Sandia, and Fernald soils. Tests 2, 9, and 14 involve gas treatment of uncontaminated soils followed by leaching. The purpose of the latter tests is to determine what effect, if any, the hydrogen sulfide treatment has on uncontaminated soils. Tests 3, 10, and 15 will examine the mobility of the contaminants in the absence of treatment. Tests 1, 2, 3, 8, 9, 10, 13, 14, and 15 will serve as a baseline to the treatment tests involving contaminated soils.

Several combinations of contaminants will be introduced to the soils. The soils will then be treated with two concentrations of hydrogen sulfide treatment gas. For the Hanford soil, a 2x2 full-factorial test matrix will be performed using nitrate and hydrogen sulfide concentrations as the test variables and chromium and uranium as the contaminant constituents (tests 4 through 7). In order to determine the effect of water on the reaction, two tests (18 and 19) will be conducted with uranium and chromium as the contaminant constituents and treated with 2,000 ppm hydrogen, and percent moisture will be varied. The test for the effect of moisture is critical, as the reduction reaction may be mediated by moisture. For the Fernald soil, a test will be performed using hydrogen sulfide concentrations as the test variable and chromium and uranium as the contaminant constituents (tests 16 and 17). For the Sandia soil, two tests will be conducted using two different hydrogen sulfide concentrations as the test variables and chromium as the contaminant constituent (tests 11 and 12).

Table 2. Test Matrix.

Experiment number	Soil type	Contaminants	H ₂ S treatment concentration (ppm)	H ₂ S required (μmoles*)
1	Hanford	none	none	none
2	Hanford	none	2,000	16,440
3	Hanford	200 ppm Cr/200 ppm U/ 100 ppm NO ₃ ⁻	none	none
4	Hanford	200 ppm Cr/200 ppm U/ 100 ppm NO ₃ ⁻	2,000	16,440
5	Hanford	200 ppm Cr/200 ppm U/ 100 ppm NO ₃ ⁻	100	16,440
6	Hanford	200 ppm Cr/200 ppm U/ 5,000 ppm NO ₃ ⁻	2,000	16,440
7	Hanford	200 ppm Cr/200 ppm U/ 5,000 ppm NO ₃ ⁻	100	16,440
8	Sandia	none	none	none
9	Sandia	none	2,000	13,460
10	Sandia	200 ppm Cr	none	none
11	Sandia	200 ppm Cr	2,000	13,460
12	Sandia	200 ppm Cr	100	13,460
13	Fernald	none	none	none
14	Fernald	none	2,000	2,980
15	Fernald	200 ppm U/200 ppm Cr 100 ppm NO ₃ ⁻	none	none
16	Fernald	200 ppm U/200 ppm Cr 100 ppm NO ₃ ⁻	2,000	16,440
17	Fernald	200 ppm U/200 ppm Cr 100 ppm NO ₃ ⁻	100	16,440
18	Hanford	200 ppm Cr/200 ppm U/ 100 ppm NO ₃ ⁻ 0% moisture	2,000	16,440
19	Hanford	200 ppm Cr/200 ppm U/ 100 ppm NO ₃ ⁻ 10% moisture	2,000	16,440

* Approximation based on 350 g of soil per test and molar treatment ratio of H₂S:Cr(VI)+U(VI) = 10.

Table 3. Proposed Test Schedule.

Test number	Initiate test	Complete test
1	1/28/93	2/10/93
2	1/28/93	2/10/93
3	1/28/93	2/10/93
4	1/28/93	2/10/93
5	2/18/93	3/1/93
6	2/18/93	3/1/93
7	2/18/93	3/1/93
8	2/18/93	3/1/93
9	3/10/93	3/25/93
10	3/10/93	3/25/93
11	3/10/93	3/25/93
12	3/10/93	3/25/93
13	4/15/93	4/29/93
14	4/15/93	4/29/93
15	4/15/93	4/29/93
16	4/15/93	4/29/93
17	5/10/93	5/25/93
18	5/10/93	5/25/93
19	5/10/93	5/25/93

In the Hanford and Fernald soil tests, both chromate and uranium will be added to the soil to achieve contaminant concentration levels of 200 ppm, by weight. The two different levels of nitrate added to the soil are 100 and 5,000 ppm, by weight. The 100 ppm level corresponds to the amount of nitrate accompanying the 200 ppm uranyl nitrate spike. Solutions of sodium nitrate salt will be added to the Hanford soil to achieve the 5,000 ppm nitrate level. The tests involving varying nitrate concentrations will help determine if nitrate acts as a sink for the hydrogen sulfide reductant. For tests involving the Sandia soil, chromium will be spiked at 200 ppm Cr(VI), no uranium or nitrate being added.

The hydrogen sulfide concentrations in the treatment gas mixture will be tested at 2,000 ppm and at 100 ppm for the soils. The total quantity of hydrogen sulfide gas introduced to each column will be 10 times the molar quantities of Cr(VI) plus U(VI). The quantity of hydrogen sulfide required for each treatment test is presented in Table 2, where it is assumed that 350 g of soil is used in each test. The total quantity of hydrogen sulfide required for the tests identified in Table 2 is 0.158 moles or about 5.4 g.

It is likely that slightly more than this will be required since some testing of the gas treatment system will be necessary. Selected tests will be also be duplicated to ensure the reproducibility of test results or to provide more information. The specific tests to be duplicated or repeated shall be determined in the course of the actual experimentation.

6.0 REFERENCES

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