Pacific Gas and Electric Company®

Yvonne J. Meeks
Site Remediation - Portfolio Manager
Environmental Affairs

6588 Ontario Road
San Luis Obispo, CA 93405
Mailing Address
4325 South Higuera Street
San Luis Obispo, CA 93401

805.546.5243
Internal: 664.5243
Fax: 805.546.5232
Internet: YJM1@pge.com

June 7, 2004

Norman Shopay
California Department of Toxic Substances Control
700 Heinz Avenue, Suite 200
Berkeley, CA 94710-2721

Subject: PG&E Topock Compressor Station - In Situ Test Plan, Core Test Plan, and Geotechnical Test Plan

Dear Norman:

Four separate technical memorandum describing proposed procedures/analyses to be used for characterization of bore hole core samples, laboratory simulation of *in situ* remediation technologies, and geotechnical testing were submitted on April 13, 2004, along with the Sampling Plan Supplement to the Draft Interim Measures Work Plan.

Comments on the technical memoranda received from members of the Consultative Work Group (CWG) were discussed with the principal commenters, DTSC, USGS, and MWD in a conference call of May 26, 2004. The participants of the May 26 conference call were Norm Shopay (DTSC), Ken Stollenwerk, Ph.D. (USGS), Eric Fordham of Geopentech (representing MWD), and Dennis Maslonkowski and Jim Mavis of CH2M HILL (representing PG&E). The discussion led to resolution of all of the comments, which have now been incorporated into the test plans (no technical comments were received on the proposed geotechnical testing). By mutual agreement among the conference call participants, PG&E was verbally authorized to proceed with the planned laboratory tests without further delay. DTSC agreed to send a confirmatory letter.

Accordingly, PG&E is redistributing the final test plans (attached) that reflect the resolution of technical comments, and will proceed with the authorized tests.

Sincerely,

Terri Herson
for Yvonne Meeks

Hexavalent Chromium (Aerobic) Zone Core Testing, PG&E Topock Compressor Station, Needles, California

DATE: June 7, 2004

1.0 Background

Cores were collected from a number of borings inland and along the floodplain of the Colorado River, in the vicinity of the hexavalent chromium [Cr(VI)] plume. Two types of cores were collected:

- Aerobic cores, in which zone *Cr(VI) is present* in formation water.
- Anaerobic cores from the near-shore zone, in the zone lacking measurable concentrations of Cr(VI) and having a negative redox potential.

Cores from the aerobic zone were collected initially, followed by cores from the anaerobic zone. A test plan for Cr(VI)-bearing aerobic core tests, performed in the CVO Applied Sciences Laboratory, is outlined below. A single aerobic core was collected, and three zones were tested: a core section just below the water table, a core section at mid-depth between the water table surface and bedrock, and a core section just above bedrock.

(The test plan for anaerobic cores is included as a separate memorandum.)

2.0 General Objectives

The test objectives for aerobic core testing were to:

1. Estimate the mass of Cr(VI) that is bound to the formation material per unit mass of formation material at representative depths below water table (potential source of ongoing Cr(VI) release into the aquifer after dissolved mass removal).
2. Estimate how strongly trivalent chromium [Cr(III)] and Cr(VI) are bound to the formation material.

3.0 Specific Questions to be Addressed by Aerobic Core Tests

1. What is the grain size distribution of formation materials from each of the three zones?
2. What is the as-received Cr(VI) concentration in porewater in the three core segments?
3. What is the total organic carbon (TOC) concentration of formation solids from each of the three zones?

4. What are the water-rinsed, as-received dithionite/citrate/bicarbonate-extractable (DCB) aluminum, iron, manganese and chromium concentrations of the formation solids from the three zones? What percentage of the total concentration of each metal is removed with this extraction?
5. What is the as-received moisture content of formation solids from the three zones?
6. What mass of Cr(VI) is extractable by deionized water in each of two successive extractions? What percentage of the total chromium content does deionized-extractable Cr(VI) represent?
7. What mass of Cr(VI) is extractable by phosphate buffer in each of two successive extractions, following the two deionized water extractions? What percentage of the total chromium does phosphate-extractable Cr(VI) represent?
8. How much aluminum, iron, manganese, and chromium are extracted in each step of sequential batch extraction tests? What is their relative binding strength to the formation materials? How are extractable metals distributed among the various sequentially extracted fractions?
9. Is deionized water-extractable Cr(VI) distinguishable from Cr(VI) in porewater?
10. What is the phosphate-extractable Cr(VI) loading per unit surface area (assuming spherical particle geometry) of formation solids from each zone?
11. Is there an apparent correlation between Cr(VI) loading capacity and the TOC of formation solids?
12. Is there evidence of Cr(III) on formation solids based on DTC and/or hydroxylamine hydrochloride-extraction (amorphous metal oxides) results?
13. How good is the reproducibility of deionized- and phosphate buffer-extracted formation solids, as indicated by a replicate test?

4.0 Task Descriptions

4.1 Preparations

4.1.1 Task 1 – Test Plan Review and Material Acquisition

- Review test plan and specific test procedures.
- Obtain materials needed to perform tests.

4.1.2 Task 2 – Initial Sample Preparation, Grain Size Analysis, Total Metals, and TOC

- Form three composite 1,000-gram samples from each zone by selecting representative materials from each core section (avoid moisture loss to the extent possible).
- Homogenize each composite sample and determine the moisture content by drying a small aliquot at 103 to 107° C. (Perform one duplicate moisture analysis.) (Question 5)

- Perform TOC analysis on dried, homogenized formation material from each zone. (Perform on duplicate analysis.) (Question 3)
- Perform a lithium *meta*borate digestion. Dissolve and analyze for total aluminum, iron, manganese, and chromium (to be performed by ALS Chemex). (Perform one duplicate analysis.) (Questions 4 and 7)
- Screen-out solids over ~0.25 millimeter (mm) in each dried aliquot and weigh each screened fraction.
- Perform grain size analysis on small aliquot from each size range (≥0.25 mm and <0.25 mm) (Question 1).
- Screen sufficient homogenized bulk formation material from each zone to produce 150 grams of the <0.25-mm fraction, which will be used in tests outlined in the following tasks. Preserve both screened fractions in a cold, humid condition to prevent biological growth and dehydration.

After this point, perform tests only with material with a grain size less than 0.25 mm, which has a high specific surface area.

4.2 Extraction Tests

4.2.1 Task 3 – Deionized Water Extraction

Cr(VI) in Porewater

- Weigh out 150 grams (dry basis) (~97 cc) of formation material from each zone, add 30.0 milliliters (mL) of deionized water (assumes 2.2 theoretical mineral density and 30-percent pore volume), and gently mix for 10 minutes. Centrifuge each sample, remove and measure the volume of the free liquid, and run ion chromatography scans for Cr(VI). Back-calculate the concentration of Cr(VI) that was present in porewater. (Back-calculate porewater concentration as diluted by the added deionized water. Perform one duplicate analysis.) (Question 2)

First Deionized Water Extraction

- Continue working with formation material from the preceding step, add deionized water at a ratio of 3:1 (water weight-to-solids dry weight), and agitate for 10 minutes. (Perform one duplicate test and carry it though the entire deionized water and phosphate extraction process.)
- Centrifuge and decant liquid for analysis of Cr(VI) at a target detection of 0.2 micrograms per liter (µg/L); aluminum, iron, and manganese each at a target detection of 10 µg/L; and total chromium at a target detection of 1 µg/L.
- Reanalyze solids for water content.

Second Deionized Water Extraction

- Continue working with formation material from the preceding step, add deionized water at a ratio of 3:1 (water weight-to-solids dry weight), and agitate for 10 minutes.

- Centrifuge and decant liquid for analysis of Cr(VI) to a target detection of 0.2 µg/L; aluminum, iron, and manganese each at a target detection of 10 µg/L; and total chromium at a target detection of 1 µg/L.
- Reanalyze solids for water content. (Questions 6 and 9)
- Wash each sample three times with deionized water (2:1 weight ratio water-to-solids), centrifuging after each wash cycle, including the final wash; combine the first two rinses; measure the combined volume and analyze for Cr(VI). Analyze the third rinse separately to serve as a "zero" baseline for phosphate extractions in Task 6.
- Split the sample into two approximately equal portions and weigh each.
- Send the largest portion out for sequential extraction tests and retain the other portion for use in DCB extractions in Task 4 and determinations of extractable Cr(VI) in Task 6.

4.2.2 Task 4 – Dithionite/Citrate/Bicarbonate Extraction

- Weigh out 5 grams of each formation material, plus one duplicate (dry-weight basis) and perform DCB extraction.
- Follow the procedure in Attachment A for DCB-extractable iron (and aluminum and manganese), except use the 0.25 mm screened formation material; do not grind or sieve.
- Analyze the extract for aluminum, iron, and manganese and report the results as "DCB-extractable iron and manganese." (Question 4)

4.2.3 Task 5 – Sequential Batch Extractions

- Ship the aliquots to be tested for extraction tests, including one duplicate, to ALS Chemex for sequential batch extractions.
- Sequential batch extraction will include at least the following extractions. Wash the solids before beginning each successive extraction step.
    - **Water soluble** aluminum, iron, manganese, and chromium measured in Task 3.
    - **Exchangeable (sodium acetate at pH 8.2 for 1 hour)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Carbonate-bound (sodium acetate at pH 5.0 for 1 hour)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Amorphous (0.05 M hydroxylamine hydrochloride and 0.01 M nitric acid for 1 hour)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Organically-bound (0.1 M sodium pyrophosphate at pH 10 for 1 hour)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Crystalline (0.25 M hydroxylamine hydrochloride and 0.25 M hydrochloric acid for 1 hour at 60° C)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).

  - **Residual refractory (lithium *meta*borate fusion, dissolution and analysis)** aluminum, iron, manganese, and chromium (target detection – 10, 10, and 1 µg/L, respectively).

- Laboratory results from sequential batch extraction provide information about relative strength of formation-associated iron, manganese, and chromium. (Question 8)

4.2.4 Task 6 – Exchangeable Cr(VI)

First Phosphate Extraction

- Prepare 1.5 liters of buffer solution containing 0.871 g/L of K_2HPO_4 (5 m Molar) and 0.68 g/L of KH_2PO_4 (5 mMolar) (pH ~7.2).
- Weigh out 10 grams (dry-weight basis) of each deionized water-extracted formation material (plus duplicate) from Task 3 and add 100 ml of phosphate buffer solution.
- Gently shake or tumble for 18 hours, then centrifuge the solids and analyze aqueous phase for Cr(VI), total chromium, aluminum, iron, and manganese (target detection limits, 0.2, 1.0, 10, 10, and 10 µg/L, respectively)
- Recover centrifuged solids, drain free water, and analyze the moisture content of each sample.

Second Phosphate Extraction

- Weigh out 10 grams (dry-weight basis) of each first-step phosphate buffer-extracted formation material and add 100 ml of phosphate buffer solution.
- Gently shake or tumble for 18 hours, then centrifuge the solids and analyze aqueous phase for Cr(VI), total chromium, aluminum, iron, and manganese (target detection limits, 0.2, 1.0, 10, 10, and 10 µg/L, respectively).
- Recover centrifuged solids, drain free water, and analyze the moisture content of each sample (Question 7).

5.0 Data Reduction, Analysis and Reporting

5.1 Cr(VI) Loading per Unit Specific Surface Area

- Calculate the specific surface area for formation material less than 0.25 mm, then divide the combined phosphate-extractable Cr(VI) mass from the combined phosphate extractions into the specific surface area, after subtracting the soluble Cr(VI) mass from the deionized water extractions.
- Report the net mass of exchangeable Cr(VI) per unit specific area for each formation sample. (Question 10)

5.2 Exchangeable Cr(VI) *versus* Soil TOC

Plot phosphate buffer-extractable Cr(VI) versus TOC and evaluate whether there is an apparent correlation between exchangeable Cr(VI) and solids surface area. (Question 11)

5.3 Sequential Extractions

Plot cumulative extracted iron, manganese, and chromium masses per unit solids weights ("specific mass") as percentages of the cumulative total specific masses of the respective metals. (Question 8)

5.4 DCB vs. Exchangeable, Carbonate-bound, and Amorphous Metal Oxides

Compare the percent (of total) extractable aluminum, iron, manganese, and chromium from DCB extractions with corresponding total percent aluminum, iron, manganese, and chromium extractions during exchangeable, carbonate-bound, and amorphous steps of sequential batch extraction procedures. (Question 12)

5.5 Comparison of Replicate Phosphate Extraction Tests

- Compare duplicate grain size analyses, deionized water, phosphate buffer, and sequential batch extractions and evaluate reproducibility. (Question 13)

5.6 Reporting

- Provide test results to the Technical Work Group as they become available.
- Participate in a Technical Work Group meeting, as appropriate.
- Prepare an interpretive report and present the data.

Anaerobic Core Hexavalent Chromium Uptake Capacity, PG&E Topock Compressor Station, Needles, California

DATE: June 7, 2004

1.0 Background

Cores were collected from a number of borings inland and along the floodplain of the Colorado River, in the vicinity of the hexavalent chromium [Cr(VI)] plume. Two types of cores were collected:

- Cores in which Cr(VI) is present in associated formation water.
- Anaerobic cores from the near-shore zone, in the zone lacking measurable concentrations of Cr(VI) and having a negative redox potential.

Cores from anaerobic zones of three borings were collected in April 2004. Selected sections from three borings in the anaerobic floodplain were sent to the CVO Applied Sciences Laboratory for characterization and testing. Three sections from each boring were selected for testing: a core section just below the water table; a core section at mid-depth between the water table surface and bedrock; and a core section just above bedrock. Water-bearing sections containing sands, silts, and clays are preferred to sections where coarse or monolithic material dominates or sections where the material is so fine-grained that transmissivity is extremely low.

2.0 General Objectives

The test objectives for anaerobic core testing were to:

1. Estimate the mass of Cr(VI) per unit mass of formation material that is reversibly bound to anaerobic formation solids at representative depths below water table (desorption).
2. Estimate the "total" (acidic) and Cr(VI) (pH-neutral)-reducing capacity of anaerobic formation solids (reduction).
3. Estimate how strongly trivalent chromium [Cr(III)] and Cr(VI) are bound to the anaerobic formation solids (sequential extraction).
4. Evaluate whether Cr(VI)-saturated anaerobic solids "rebound" in their ability to adsorb more Cr(VI) following a quiescent period to allow microorganisms to regrow (incubation "rebound").

3.0 Specific Questions to be Addressed by Anaerobic Tests

1. What is the grain size distribution of formation materials from the zones of each of the anaerobic cores that will be evaluated in the following procedures?
2. What is the as-received Cr(VI) concentration in porewater in each core sample? How do corresponding zones compare from core to core?
3. What is the total organic carbon (TOC) concentration of formation solids from each core sample to be tested?
4. What is the as-received moisture content of each core sample to be tested?
5. What is the total reducing capacity of the formation material from each core sample to be tested (Walkley-Black Method)?
6. What are the as-received dithionite/citrate/bicarbonate-extractable (DCB) aluminum, iron, manganese, and chromium concentrations of the formation solids? What percentage of the total concentration of each metal is removed with this extraction?
7. What mass of Cr(VI) is extractable by phosphate buffer? What percentage of the total chromium does phosphate-extractable Cr(VI) represent?
8. What is the phosphate-extractable Cr(VI) loading per unit surface area, if any, (assuming spherical particle geometry) of formation solids from each zone?
9. How much iron, manganese, and chromium are extracted in each step of sequential batch extraction tests? What is their relative binding strength to the formation solids? How are extractable metals distributed among the various sequentially extracted fractions?
10. How much Cr(VI) is taken up per unit weight/mass of anaerobic formation solids, from "low TDS" (low total dissolved solids) and "high TDS" groundwater from the Topock site?
11. Does anaerobic formation material reestablish its capacity to remove Cr(VI) when it has once been saturated with Cr(VI) but after an extended relaxation period?
12. Is there an apparent correlation between Cr(VI) loading capacity and the TOC content of formation solids?
13. Is there an apparent correlation between Cr(VI) loading capacity and the extractable aluminum, iron, or manganese, as characterized by sequential batch extraction tests?
14. How much of the Cr(VI) that is taken up by formation solids is extractable with phosphate buffer?
15. How good are the reproducibilities of the DCB and sequential batch extraction methods?

4.0 Task Descriptions

All work with anaerobic core sections was performed under nitrogen, in an enclosed glove box, to preserve the cores and sub-samples in anaerobic conditions throughout the entire testing and post-testing period.

4.1 Preparations

4.1.1 Task 1 – Test Plan Review and Material Acquisition

- Review test plan and specific test procedures.
- Obtain materials needed to perform tests.

4.1.2 Task 2 – Initial Sample Preparation, Moisture, Grain Size, Total Metals, and TOC

- Consult boring logs and involve project hydrogeologists to select core sections that are most representative of water- and Cr(VI)-bearing zones in the project area across all three borings, and to select a core from one location for which all three depth zones will be investigated for a total of five core sections (three depths from one boring and two additional sections; one from each of the other two cores).

  Formation materials must be handled under anaerobic conditions except during acidic extractions at pH greater than 6.0 or complete digestion with lithium metaborate.

- Form 1,000-gram composite samples from each of the five selected core segments by selecting representative materials from each core section (avoid moisture loss to the extent possible).

- Homogenize each composite sample and determine the moisture content by drying a small aliquot at 103 to 107° F (perform one duplicate moisture analysis). (Question 4)

- Perform TOC analyses on dried, homogenized material from each sample. (Perform one duplicate analysis.) (Question 3)

- Perform lithium *meta*borate digestion; dissolve and analyze for total iron, manganese, and chromium (to be performed by ALS Chemex). (Questions 6 and 7)

- Screen out solids over ~0.25 millimeter (mm) in each dried aliquot, and weigh each screened fraction.

- Perform grain size analysis on small aliquot from each size range (≥0.25 mm and <0.25 mm). (Question 1)

- Screen sufficient homogenized bulk core material to produce greater than 150 grams (dry basis) of the less than 0.25 mm fraction from each of the five zones. These screened sub-samples will be used for testing outlined in the following tasks. Preserve both large- and small-grain screened fractions in a cold, humid, anaerobic environment (wrapped in two layers of Saran Wrap and cooled to approximately 40° F) to inhibit biological growth and prevent dehydration.

After this point, perform tests only with material with a grain size less than 0.25 mm, which has a high specific surface area.

4.2 Extraction Tests

4.2.1 Task 3 – Deionized Water Extraction

This test must be run under anaerobic conditions.

Cr(VI) in Porewater

- Weigh out 150 grams (dry basis) (~97 cc) of each formation material sample, add 30 milliliters (mL) of deionized water (assumes 2.2 theoretical mineral density and 30-percent pore volume), and mix gently for 10 minutes. Centrifuge each sample, remove and measure the volume of the free liquid, and run ion chromatography scans for Cr(VI). Back-calculate the concentration of Cr(VI) that was present in porewater. (Back-calculate porewater concentration as diluted by the added deionized water. Perform duplicate analyses.) (Question 2)
- **NOTE: If Cr(VI) is detected in any deionized extract solution, contact Brian Schroth and Jim Mavis before proceeding further with tests of that particular core, pending resolution by appropriate technical advisors. (Continue testing other cores in which no Cr(VI) is detected.)**

4.2.2 Task 4 – Dithionite/Citrate/Bicarbonate Extraction

This test must be run under anaerobic conditions.

- Weigh out 5 grams of each formation material, plus one duplicate (dry-weight basis) and perform DCB extraction.
- Follow the Walkley-Black procedure for DCB-extractable iron (and aluminum and manganese), except use the 0.25 mm screened formation material; do not grind or sieve.
- Analyze the extract for aluminum, iron, manganese, and chromium, and report the results as DCB-extractable aluminum, iron, manganese, and chromium. (Question 6)

4.2.3 Task 5 – Phosphate Extraction

This test must be run under anaerobic conditions.

- Prepare 1.5 liters of buffer solution containing 0.871 grams per liter (g/L) of K_2HPO_4 (5 mMolar) and 0.68 g/L of KH_2PO_4 (5 mMolar) (pH ~7.2).
- Weigh out 10 grams (dry basis) aliquots of each fine-screened core sub-sample (plus duplicate) and add 100 mL of phosphate buffer solution.
- Gently shake or tumble for 18 hours, then centrifuge the solids and analyze aqueous phase for Cr(VI), total chromium, aluminum, iron, and manganese (target detection – 0.2, 1.0, 10, 10, and 10 micrograms per liter [µg/L], respectively). (Questions 7 and 8)

4.2.4 Task 6 – Sequential Batch Extractions

- Ship ~75 gram aliquots of each screened fraction, including one duplicate, to ALS Chemex for sequential batch extractions.

- Sequential batch extractions should include at least the following extractions. Wash the solids before beginning each successive extraction step.
    - **Water soluble** aluminum, iron, manganese, and chromium measured in Task 3.
    - **Exchangeable (sodium acetate at pH 8.2 for 1 hour – *run under anaerobic conditions to the extent possible*)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Carbonate-bound (sodium acetate at pH 5.0 for 1 hour)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Amorphous (0.05 M hydroxylamine hydrochloride and 0.01 M nitric acid for 1 hour)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Organically-bound (0.1 M sodium pyrophosphate at pH 10 for 1 hour – *run under anaerobic conditions to the extent possible*)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Crystalline (0.25 M hydroxylamine hydrochloride and 0.25 M hydrochloric acid)** aluminum, iron, manganese, and chromium (target detection – 10, 10, 10, and 1 µg/L, respectively).
    - **Residual refractory (lithium *meta*borate fusion, dissolution and analysis)** aluminum, iron, manganese, and chromium (target – 10, 10, 10, and 1 µg/L, respectively).
- Laboratory results from sequential batch extraction provide information about relative strength of formation-associated aluminum, iron, manganese, and chromium. (Question 9)

4.3 Anaerobic Core Cr(VI) Uptake

4.3.1 Task 7 – Total Reducing Capacity

Follow Walkley-Black testing procedures, except **DO NOT** grind the sample; use the screened aliquots as-received. (Question 5)

4.3.2 Task 8 – Cr(VI) Uptake from *Low*-TDS Topock Groundwater

Low-TDS Water Source (TW-1)

This test must be run under anaerobic conditions.

- Calculate the mass of Cr(VI) that was consumed in the Walkley-Black test (Task 7) and calculate the volume of TW-1 water that contains an equivalent mass of Cr(VI).
- Weigh out three 100-gram (dry basis) aliquots of each screened core sub-sample, plus a duplicate, and place each into an extraction vessel (flask, tumbler, or other device).
- Charge each 100-gram split with groundwater from TW-1 that contains 0.1 percent, 1 percent, and 10 percent of the Cr(VI) mass, respectively.

- Gently agitate for 30 minutes, allow solids to settle, decant and filter liquid.
- Save the solids from each extraction for further testing in Task 10.
- Analyze initial TW-1 water and filtrate from each sample for Cr(VI).
- Calculate the loss in Cr(VI) and interpret the loss as uptake by anaerobic core solids. (Question 10)

4.3.3 Task 9 – Cr(VI) Uptake from *High*-TDS Topock Groundwater

This test must be run under anaerobic conditions.

- Calculate the mass of Cr(VI) that was consumed in the Walkley-Black test (Task 7) and calculate the volume of water from 20-Cluster Groundwater Holding Tank that contains an equivalent mass of Cr(VI).
- Weigh out three 100-gram (dry basis) aliquots of each screened core sub-sample, plus a duplicate, and place each into an extraction vessel (flask, tumbler, or other device).
- Charge each 100-gram split with groundwater from the 20-Cluster Groundwater Holding Tank that contains 0.1 percent, 1 percent, and 10 percent of the Cr(VI) mass, respectively.
- Gently agitate for 30 minutes, allow solids to settle, decant and filter liquid.
- Save the solids from each extraction for further testing in Task 10.
- Analyze initial 20-Cluster Groundwater Holding Tank water and filtrate from each sample for Cr(VI).
- Calculate the loss in Cr(VI) and interpret the loss as uptake by anaerobic core solids. (Question 10)

4.3.4 Task 10 – Phosphate Extraction of Cr(VI) from Groundwater-treated Core Solids

This test must be run under anaerobic conditions.

- Select the sample from the "low-TDS" and the "high-TDS" extractions in which Cr(VI) uptake was most clearly evident (from Tasks 8 and 9).
- Split each 100-gram sample into two ~10-gram sub-samples and one 80-gram sample. Store one 10-gram aliquot from each split under anaerobic conditions at room temperature for further testing under Task 11. Store the 80-gram sample under anaerobic conditions at room temperature for use in Task 12.
- Wash each 10-gram aliquot three times with 2:1 (weight ratio) of deionized water to remove porewater; discard the first two washes and analyze the third wash for Cr(VI).
- Drain free water from the 10 grams aliquots of each fine-screened core sub-sample and add 100 mL of phosphate buffer solution (same phosphate buffer used in Task 5).

- Gently shake or tumble for 18 hours, then centrifuge the solids and analyze aqueous phase for Cr(VI), total aluminum, iron, manganese, and chromium (target detection – 0.2, 1.0, 10, 10, and 10 µg/L, respectively). (Question 14)
- Rinse each solid sample twice (2:1 water-to-solids weight ratio) with deionized water; dry and weigh the solids.

NOTE: The solids content of sample to be used below (Task 11) will be calculated by subtracting the weight of solids from this phosphate extraction from the original 20-gram aliquots used in Tasks 8 and 9.

4.3.5 Task 11 – Rebound in Reduction Capacity of Anaerobic Formation Solids

This test must be run under anaerobic conditions.

- Store unused portions of split solids from Task 10 at ambient temperature but under anaerobic conditions for 6 weeks.
- Charge each unused portion with groundwater from TW-1 or the 20-Cluster Groundwater Holding Tank, respectively, that contains 1.25 times the mass of Cr(VI) that was taken up previously in Task 8 or 9.
- Gently agitate for 30 minutes, allow solids to settle, decant and filter liquid.
- Save the solids from each extraction for further testing in Task 10.
- Analyze the filtrate from each sample for Cr(VI).
- Calculate the loss in Cr(VI), interpret the loss as uptake by anaerobic core solids, and report the uptake as "Rebound" Cr(VI) uptake capacity. (Question 11)

4.3.6 Task 12 – Cr(VI) Decay Rate in Groundwater-treated Anaerobic Core Solids

- Keep the 80-gram samples from Tasks 8 and 9 under anaerobic conditions at ambient temperature.
- Select the 80-gram samples from the "low-TDS" and "high-TDS" contacting in which Cr(VI) uptake was most clearly evident (from Tasks 8 and 9).
- If contacting the core samples with Cr(VI)-containing groundwater did not result in residual detectable Cr(VI) in the free-aqueous phase, carefully add more groundwater until residual Cr(VI) remains.
- At intervals of 0, 1, 3, and 6 weeks after Cr(VI) contacting, remove a small amount of porewater and analyze for Cr(VI).
- Plot the Cr(VI) concentration as a function of elapsed time since initial Cr(VI) addition.
- Discontinue analyzing porewater when no more Cr(VI) is detected or if no reduction in Cr(VI) has occurred after 6 weeks. (Question 11)

5.0 Data Reduction, Analysis, and Reporting

5.1 Cr(VI) Loading per Specific Surface Area

- Calculate the specific surface area for formation materials less than 0.25 mm, then divide the Cr(VI) that was taken up during Tasks 8 and 9 for "low-TDS" and "high-TDS" groundwater, respectively.
- Report the net mass of Cr(VI) removed per unit specific surface area for each formation material, for "low-TDS" and "high-TDS" groundwater, respectively. (Question 8)

5.2 Cr(VI) Uptake Rebound Capacity

- Calculate the Cr(VI) uptake capacity (milligrams Cr(VI)/gram solids) as determined in Tasks 8 and 9. (Questions 8 and 9)
- Calculate the Cr(VI) uptake capacity (milligrams Cr(VI)/gram solids) as determined in Task 11. (Question 11)
- Report the relative Cr(VI) uptake capacities of the two Cr(VI) challenges.

5.3 Sequential Extractions

Plot cumulative extracted aluminum, iron, manganese, and chromium masses per unit solids weights ("specific mass") as percentages of the cumulative total specific masses of the respective metals. (Question 9)

5.4 Cr(VI) Uptake versus TOC

Plot Cr(VI) taken into each sample (Cr(VI) mass/solids mass) versus TOC content of each sample (mass TOC/solids mass) and evaluate whether there is an apparent correlation. (Question 12)

5.5 Cr(VI) Uptake versus Extractable Iron or Manganese

Compare Cr(VI) taken up onto solids with (sequential batch- and DCB-) extractable aluminum, iron, and manganese and evaluate whether there is a correlation between Cr(VI) and these extractable metals. (Question 13)

5.6 Comparison of Replicate DCB and Sequential Batch Extraction Methods

- Compare replicated results by DCB extraction and by sequential batch extraction and evaluate the reproducibility of each method. (Question 15)

5.7 Reporting

- Provide test data to the Technical Work Group as results are received from the laboratories.
- Participate in a Technical Work Group meeting to discuss the test results.
- Prepare an interpretive report that addresses all the questions and presents the data.

Data Collection for Evaluating Potential *In-situ* Remediation Technologies, PG&E Topock Compressor Station, Needles, California

DATE: June 7, 2004

1.0 Introduction

This memorandum presents the proposed field data collection plans for evaluation of potential *in-situ* remediation technologies for chromium in groundwater at the Pacific Gas and Electric Company (PG&E) Topock Compressor Station (the Topock site) located near Needles, California. Bench-scale laboratory tests (treatability tests) are proposed to evaluate a number of reagents used for *in-situ* remediation of hexavalent chromium [Cr(VI)]. The findings from these tests will be integrated into the development of a long-term remedial strategy for groundwater containing Cr(VI) at the Topock site.

1.1 Purpose and Objectives

The goal of the testing program is to generate data to evaluate the potential application of *in-situ* technologies, given the specific geochemical conditions at the Topock site.

Specific objectives are to:

- Conduct bench-scale testing of potential *in-situ* technologies based on available data on the subsurface conditions at the Topock site.
- Evaluate performance of select chemical reductants (dithionite and polysulfide) and biological substrates (methanol, ethanol, acetate, lactate [MEAL], emulsified vegetable oil, and molasses) in a controlled laboratory environment.
- Evaluate the potential use of zero-valent iron (ZVI) at the Topock site.
- Identify promising *in-situ* technologies based on the bench-scale results and on operational factors that may influence the successful performance of the select *in-situ* technologies.
- Assess the potential for these *in-situ* technologies as part of a full-scale alternative for management of Cr(VI) in the groundwater at the Topock site.

1.2 Tasks and Activities

This memorandum summarizes the tasks and activities required to collect field representative samples of aquifer soils and groundwater and the use of the samples for construction of bench-scale microcosms and other bench-scale tests. The activities include:

- Reviewing data that are currently being collected as part of the interim remediation system. These data include pumping tests, aquifer boring logs, and geochemical data. This information will be used to estimate the potential influencing factors on application of *in-situ* technologies at these locations.
- Collecting field samples for construction of microcosms to be set up for treatability testing.
- Conducting treatability testing for evaluation of chemical and biological reduction of Cr(VI) to trivalent chromium [Cr(III)].
- Conducting treatability testing for evaluation of Cr(VI) to Cr(III) treatment using ZVI.
- Preparing a report that documents the results of the bench-scale testing.

The following presents a description of the activities.

2.0 Bench-scale Treatability Testing

Bench-scale testing will be conducted on microcosms constructed using site aquifer materials and groundwater collected from locations where *in-situ* technologies are likely to be implemented. This will simulate the subsurface conditions at the site and be used to:

- Assess the potential of the existing geochemical conditions to reduce Cr(VI) to Cr(III) and precipitate Cr(III) (following the amendment with dithionite, polysulfide, and soluble organic carbon substrates such as molasses, MEAL, emulsified vegetable oil, and ZVI).
- Identify reaction rate kinetics for the conversion of Cr(VI) to Cr(III) using the above-identified chemical and biological substrates.
- Identify and select the most appropriate chemical and/or biological substrate for further evaluation based on performance and reaction rate kinetics data and site conditions.

Methods for soil and groundwater sample collection, construction, and incubation of microcosms and the proposed sampling and analysis plan are presented in the following sections.

2.1 Soil and Groundwater Sample Collection and Handling Procedures

Representative aquifer soil and groundwater samples will be collected to construct the microcosms. Soil samples will be collected from a predetermined location by drilling to obtain discrete soil samples from Cr(VI)-bearing zones. Proposed drilling and sampling locations for collecting samples for use in the laboratory testing include:

- Near the Colorado River from the proposed well and boring at MW-39.
- Near the Compressor Station at MW-38.

Groundwater will be collected from each of select wells (MW-39-100, MW-38-S). Twenty gallons per well will be collected, field tested (pH, specific conductance, dissolved oxygen, oxidation-reduction potential [ORP], and temperature), and shipped to the CH2M HILL

laboratory in Corvallis, Oregon for baseline characterization and construction of microcosms. The 20-gallon samples will remain at the Corvallis laboratory and will be used for bench-scale testing as needed. The groundwater will be analyzed upon receipt at the Corvallis laboratory for characterization of Cr(VI), total chromium [Cr(T)] and the remaining parameters listed in Table 1.

TABLE 1
Summary of Baseline Characterization of Groundwater Samples Used for Bench-Scale Testing
PG&E Topock Compressor Station, Needles, California

Parameter	Methods	Number of Samples
Hexavalent Chromium (VI)	USEPA 7199	4
Dissolved Chromium	USEPA 200.7	4
Total Chromium [Cr(T)]	USEPA 200.7	4
Total Organic Carbon	USEPA 415.1	2
Dissolved Organic Carbon	USEPA 415.1	2
Bicarbonate Alkalinity	USEPA 310.1	2
Phosphate	USEPA 300	2
Dissolved aluminum	USEPA 200.7	2
Chloride	USEPA 300	2
Ammonia/Nitrate/Nitrite	USEPA 350.1/300/300	2
Sulfate/Sulfide	USEPA 300/376.1	2
Dissolved Iron (Fe^{2+})	USEPA 200.7	2
Methane	RSK175 Mod.	2
Dissolved Manganese	USEPA 200.7	2
pH, EC, ORP, temperature, dissolved oxygen	Field methods	2

Aquifer soil samples will be collected from four to five discrete depths throughout the saturated zone as the drilling advances. Twenty pounds of soil from the select boring locations will be collected, placed in coolers on ice, and shipped to the Corvallis laboratory for bench-scale testing. Upon receipt of the aquifer soils at the laboratory, the cores will be subsampled and combined to create a representative soil for use in bench-scale testing. The soil will be analyzed for those parameters listed in Table 2.

TABLE 2
Summary of Baseline Characterization of Aquifer Solids Used for Bench-scale Testing
PG&E Topock Compressor Station, Needles, California

Parameter	Methods	Number of Samples
Hexavalent Chromium (VI)	USEPA 7199 (after digestion by USEPA 3060A)	2
Total Chromium [Cr(T)]	USEPA 200.7	2
Total Organic Carbon	USEPA 9060	2
Total aluminum	USEPA 200.7	2
Chloride	USEPA 300	2
Total Nitrogen (TKN)	USEPA 351.4	2
Total Phosphorus	USEPA 365.1	2
Total Iron	USEPA 200.7	2
Manganese	USEPA 200.7	2
Specific conductance	USEPA 9050A	2
pH	USEPA 9045C	2

In addition to the *in-situ* characterization, test results used for evaluating the anoxic core will also be used to evaluate natural attenuation of Cr(VI). Analyses will be conducted using published United States Environmental Protection Agency (USEPA) analytical methods.

2.2 Chemical and Biological Microcosm Incubation, Sampling, and Analysis

Laboratory bench-scale testing is proposed to evaluate Cr(VI) reduction to Cr(III) using site aquifer materials and groundwater and to aid in the selection of *in-situ* reagents (chemical or biological reagents) that may be used for full–scale application of *in-situ* remediation. The testing will be conducted in two phases including:

- Phase 1: assessment of the capacity of chemical and biological to reduce Cr(VI) to Cr(III).
- Phase 2: assessment of the potential for re-oxidation of Cr(III) to Cr(VI) following the initial reduction to Cr(III) by exposing the microcosms to air.

Bench-scale testing will make use of established procedures using microcosms. Microcosms consisted of vessels containing soil and groundwater from the site and included treatment systems (amended with reagents to enhance Cr(VI) reduction) and unamended control systems. The microcosm vessels for this study will include 1-liter amber glass bottles with butyl rubber septa and screw caps. Each microcosm bottle will be filled with soil (approximately 400 grams [wet weight]) and groundwater (approximately 900 mL), allowing for less than 25 mL of headspace. Three replicate microcosms will be set up for each test condition. Treatment and control microcosms will be incubated in the laboratory. Anaerobic treatment and control microcosms will be constructed and incubated in an anaerobic chamber (glove box) purged with nitrogen gas to limit oxygen intrusion into the

bottles, while aerobic systems were set up in the laboratory (atmosphere conditions). Following Phase 1, the microcosm bottles will be opened to the air in the laboratory to evaluate re-oxidation of Cr(III) to Cr(VI).

The length of microcosm incubation will be based on the rates of Cr(VI) to Cr(III) conversion kinetics. Incubation periods are typically on the order of a few weeks for the Phase 1 testing and for an additional 2 months for Phase 2 reoxidation testing.

Microcosms will be analyzed between six to eight times over the incubation period. Chromium [Cr(VI), Cr(T), and Cr(dissolved)], pH, oxidation-reduction potential, dissolved oxygen, alkalinity, methane, and reagents will be analyzed at every sampling event, while geochemical parameters (remaining parameters in Table 1) will be characterized during at least four sampling events. Reagents added to the microcosms to effect chromium reduction will be analyzed to track persistence and reagent degradation kinetics. Sample intervals may vary by treatment, and sampling events will be selected based on substrate consumption and chemical and biological activity.

2.3 ZVI Treatability Testing

Treatability testing using ZVI will be conducted to evaluate the Cr(VI) treatment performance of ZVI under site-specific geochemical conditions simulated in microcosms. In addition to Cr(VI) treatment testing, precipitation of secondary minerals in the ZVI will also be examined.

2.4 Treatability Testing Report

A treatability testing report will present the results of the bench testing and recommend potential *in-situ* technologies to be further evaluated in a field pilot test.

Geotechnical Sampling and Testing Methodology, PG&E Topock Compressor Station, Needles, California

DATE: June 7, 2004

1.0 Background

This memorandum was prepared to provide detailed guidance for field personnel for initial geotechnical sampling and testing for the PG&E Topock site near Needles, California. The overall sampling plan is described in the *Sampling Plan Supplement* (Sampling Plan Supplement), *PG&E Topock Interim Measures Drilling Program*, dated April 13 2004.

The Sampling Plan Supplement directs field staff to select formation samples from all borings from the saturated Alluvial Aquifer[1], and in selected Red Fanglomerate locations, for sieve and grain-size and hexavalent chromium analyses. Two borings have been proposed to provide additional information to evaluate the constructability of a subsurface barrier wall at the Topock site. Details are provided below.

2.0 Geotechnical Objectives

At two select locations, geotechnical sampling and analysis have been proposed to aid in characterization of soil and rock materials. The remainder of this memorandum details the additional sampling and testing that is proposed to support evaluation of the constructability of a subsurface barrier wall such as a soil-bentonite cutoff wall. The properties of the unsaturated and saturated portions of the dredged sand, the alluvial aquifer, and underlying consolidated formation (Red Fanglomerate) are required for this evaluation.

The two proposed alluvial borings (MW-28D and MW-36) will provide samples for geotechnical analysis. The geotechnical data are intended to provide:

- Properties of the dredged sand and alluvial soils to assess trench stability and excavatability.
- Soil gradations to assess the suitability of the *in-situ* soil for backfill for the wall and to assess trenching and excavating requirements and limitations (i.e., equipment needs and anticipated work duration).
- Depth to and geotechnical properties of the Red Fanglomerate to assess the potential to construct a "key" into this unit.

[1] For complete clarity, the dredged sand is called out separately from the Alluvial Aquifer so that there is no doubt that the sampling and testing recommended herein should include the dredged sand.

2.0 Geotechnical Sampling and Testing

Geotechnical sampling and testing will proceed as follows:

1. Core will be collected continuously from the ground surface to bedrock (using rotosonic drilling methods). Standard penetration testing (SPT) in accordance with ASTM D1586 is proposed at 10-foot intervals throughout the boring. Borings will be logged in accordance with CH2M HILL guidelines using visual-manual procedures (ASTM D2488).
2. All borings will be advanced a minimum of 5 feet into the Red Fanglomerate. A 5-foot (minimum) core or sample (depending on the bedrock soundness) of Red Fanglomerate will be collected from all borings, if possible.
3. Table 1 summarizes the proposed geotechnical data collection efforts. These recommendations are not intended to limit any other sampling or testing that is being performed for any other purpose.

TABLE 1
Subsurface Soil Geotechnical Sample Analysis Methods
PG&E Topock Compressor Station, Needles, California

Parameter	ASTM Method	Comment
Continuous sampling	--	From ground surface to bottom of each boring regardless of depth to water.
Logging of Soil	D2488	At a minimum of 5-foot intervals and at all lithologic changes.
SPT	D1586	Proposed at 10-foot intervals between the ground surface and the Red Fanglomerate.
Soil gradations (particle size)	D422	Representative samples will be composited from rotosonic core on minimum 10-foot intervals. Hydrometer analysis on percent passing No. 200 sieve not required, only washed percent passing No. 200 sieve.
Moisture Content	D2216	To be performed on SPT samples only. Samples obtained during rotosonic drilling are not anticipated to retain their *in-situ* moisture content.
Atterberg Limits	D4318	To be performed on fine-grained SPT samples only. Determination to suitability of samples to be made by lab.
Rock Quality Designator	D6032	Determine only in Red Fanglomerate when applicable.
Unconfined Compressive Strength	D2938	Determine only in Red Fanglomerate, only if intact samples are obtained. Not necessary if rock crumbles during sampling.

The samples obtained during rotosonic drilling will be used to assess the site hydrostratigraphy and formation changes and to provide samples for gradation analysis. The objective of the gradation analysis is to be able to assess the anticipated typical

gradation of the excavated materials when these materials are blended on the surface into a single mixture for a slurry wall backfill. Representative samples will be composited and analyzed at 10-foot intervals. The results will be combined mathematically to predict the typical gradation. Using this approach, any materials determined to be unsuitable for backfill can be mathematically omitted in developing the overall expected gradations without additional laboratory testing.

The "N-value" obtained during SPT is will be used as an indicator of soil density and excavatability. In addition, "undisturbed" samples from the standard-diameter, split-barrel sampler will be used to obtain *in-situ* moisture content of the soil and to provide Atterberg Limits. (Atterberg Limits will be performed only on fine-grained soils.) One half of each SPT sample will be sent for laboratory analysis, and the remainder will be labeled and stored in a jar or ziplock bag in a manner that will preserve the sample for further testing, if required. The determination suitability for testing for Atterberg Limits will be made by the laboratory performing the geotechnical testing. The combined results of the geotechnical testing will provide an indication of trench stability.

Drilling will continue until refusal is met or until penetration has advanced 5 feet into the Red Fanglomerate. If intact samples of the Red Fanglomerate are obtained, the core should be logged and some estimate of Rock Quality Designator should be made. If of acceptable dimension and quality, the samples will be submitted for testing unconfined compressive strength in accordance with ASTM D2938.

Additional assessment of excavatability and constructability of a subsurface physical barrier may be made at a later date using geophysical methods.