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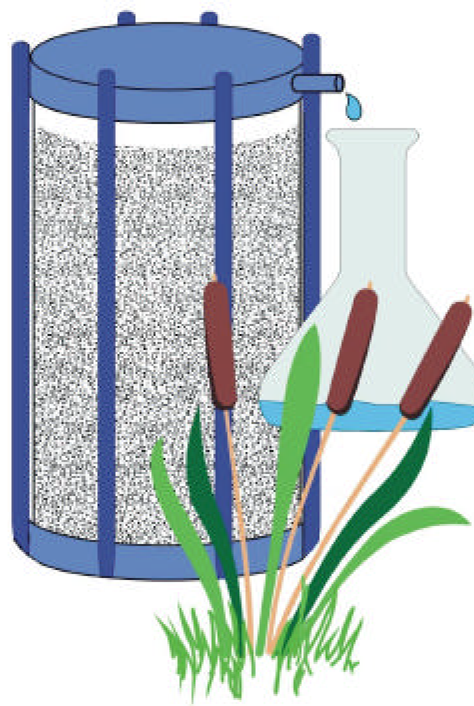
Environmental Sciences Laboratory

Final Report

Rejuvenating Permeable Reactive Barriers by Chemical Flushing

U.S. Environmental Protection Agency Region 8 Support

August 2004



Prepared for
U.S. Department of Energy
Grand Junction, Colorado

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Signature Page

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Acronyms

AFO	amorphous ferric oxyhydroxide
Ca	calcium
CBD	citrate bicarbonate dithionite
CCD	citrate carbonate dithionite
yd^3	cubic yards
°C	degrees Celsius
DOE	U.S. Department of Energy
DTPA	diethylenetriamine pentaacetic acid
EDTA	ethylenediaminetetraacetic
EPA	U.S. Environmental Protection Agency
ESL	Environmental Sciences Laboratory
Fe	iron
g	grams
g/mL	grams per milliliter
HCl	hydrochloric acid
in.	inch
L	liters
lb	pounds
µS/cm	microsiemens per centimeter
M	molar
mg	milligrams
mg/L	milligrams per liter
min	minutes
mL	milliliters
mL/min	milliliters per minute
mV	millivolts
µg/L	micrograms per liter
ORP	oxidation-reduction potential
pCi/L	picocuries per liter
PRB	permeable reactive barrier
U	uranium
UMTRA	Uranium Mill Tailings Remedial Action
ZVI	zero-valent iron

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Executive Summary

A permeable reactive barrier (PRB) is an engineered zone of chemically reactive material placed in the flow path of contaminated ground water to stabilize or degrade contaminants as ground water moves through the zone. The most common reactive material employed in PRBs is zero-valent iron (ZVI). PRBs are rapidly becoming widely used means to remediate ground water.

Unfortunately, reactions of ZVI with contaminants, dissolved oxygen, and water molecules result in an increase in pH values that cause carbonate minerals to precipitate in the ZVI. Alkalinity decreases of as many as several hundred milligrams per liter (as calcium carbonate) from influent to effluent indicate that large volumes of carbonate minerals have precipitated in the PRBs. In addition to carbonate precipitation, oxidation causes the precipitation of iron oxide minerals. The buildup of carbonate and oxide minerals within the reactive zone could disrupt the performance of the PRB by causing (1) preferential pathways within the reactive zone, (2) ground water mounding and bypassing the PRB, and (3) a reduction in the reactivity of the media because of mineral deposition on the ZVI surfaces.

The U.S. Environmental Protection Agency funded this project to investigate chemical methods of rejuvenating ZVI-based PRBs. The work was conducted by Environmental Sciences Laboratory personnel at the U.S. Department of Energy office in Grand Junction, Colorado. A portion of this work was reported previously in *The Final Report: Phase II, Performance Evaluation of Permeable Reactive Barriers and Potential for Rejuvenation by Chemical Flushing* (DOE 2004). For completeness, the results of the PRB rejuvenation project are reported here in their entirety.

Bench-scale tests were conducted to evaluate the potential for rejuvenation of ZVI PRBs using chemical solvent flushing. We tested various solvents, including free acid ammonium oxalate, diethylenetriamine pentaacetic acid (DPTA), disodium EDTA, ethylenediaminetetraacetic acid (EDTA), hydroxylamine hydrochloride, sodium citrate, sodium dithionite, and tetrasodium EDTA, and the commercial products Lime Away, and Lime Out (designed to remove calcium and iron scale deposits). Some tests were conducted with combinations of these solvents and sometimes included bicarbonate or carbonate as a pH buffer. Rejuvenation agents are considered more favorable if they dissolved more calcite (a ZVI corrosion product that occludes porosity), less ZVI, and have low toxicity. The ability to dissolve amorphous ferric oxyhydroxide, hematite and magnetite was also considered favorable. Most of the individual solvents and combinations of solvents were able to dissolve some calcite, but they also dissolved some ZVI.

Of the solvents tested in batch mode, disodium EDTA and tetrasodium EDTA were considered to be the most suitable and were selected for preliminary column tests. The column test results indicate that EDTA was able to remove all the calcite deposited during ZVI corrosion and some of the calcite initially present in the column fill material. While some iron was also dissolved by EDTA, the change in the iron inventory was insignificant.

A portion of ZVI from the Monticello, Utah, PRB was treated with disodium EDTA to determine if the treatment would improve the ability of the ZVI to remove uranium. Unfortunately, the results were inconclusive because the ZVI in both the treated and untreated columns removed uranium at a fast rate. Longevity calculations made by Morrison (2003) assumed that the ZVI would lose reactivity at a much higher rate than indicated by the column results. The data on the untreated column presented in this report suggest that the PRB continues to remove uranium at a high rate, implying that the previous calculations of longevity should be revisited.

This project was funded through an Interagency Agreement with the Environmental Protection Agency. DOE would like to thank Charles Sands and Robin M. Anderson from the Office of Emergency Response and Remediation, Response Decision Team for their support in this project. DOE would also like to acknowledge the efforts of Paul Mushovic, Richard Muza, and Jay Silvernale from the Office of Ecosystems Protection and Remediation, Region VIII, for their technical support and review of this document.

1.0 Introduction

A permeable reactive barrier (PRB) is a zone of chemically reactive material placed in the flow path of contaminated ground water to stabilize or degrade contaminants as ground water moves through the zone. The most common reactive material employed in PRBs is zero-valent iron (ZVI). PRBs are rapidly becoming a widely used means of remediating ground water. ZVI can treat both organic and inorganic contaminants including uranium.

Unfortunately, reactions of ZVI with contaminants, dissolved oxygen, and water molecules result in an increase in pH values that cause carbonate minerals to precipitate within the ZVI. Alkalinity decreases of as many as several hundred milligrams per liter (mg/L) (as calcium carbonate) from influent to effluent indicate that large volumes of carbonate minerals have precipitated in the PRBs. In addition to carbonate precipitation, oxidation causes the precipitation of iron oxide minerals. The buildup of carbonate and oxide minerals within the reactive zone could disrupt the performance of the PRB by causing (1) preferential pathways within the reactive zone, (2) ground water mounding and bypassing the PRB, and (3) a reduction in the reactivity of the media because of mineral deposition on the ZVI surfaces.

The U.S. Environmental Protection Agency (EPA) funded this project to investigate chemical methods of rejuvenating ZVI-based PRBs. The work was conducted by Environmental Sciences Laboratory (ESL) personnel at the U.S. Department of Energy (DOE) office in Grand Junction, Colorado. A portion of this work was reported previously in *The Final Report: Phase II, Performance Evaluation of Permeable Reactive Barriers and Potential for Rejuvenation by Chemical Flushing* (DOE 2004). For completeness, the results of the PRB rejuvenation project is reported here in its entirety. Appendix A contains copies of the ESL notes.

Ground water chemistry data for samples from the PRB at Monticello, Utah, show that reactivity has decreased and that carbonate minerals and iron oxide corrosion products have precipitated in the ZVI matrix (Morrison 2003). Other PRB sites have experienced similar decreases in reactivity and precipitation of corrosion products (e.g., Wilkin and Puls 2003). Data presented by DOE (2004) indicate that some reactive ZVI is still present in zones that have lost reactivity. If the corrosion products could be removed while leaving ZVI intact, the reactivity of the PRB may be improved. In the present study, preliminary laboratory tests were conducted to evaluate the efficiency of several solvents for rejuvenating ZVI-based PRBs. Batch tests using standard specimens of ZVI and corrosion products were performed to determine the amount of mineral dissolution after a specific time period. Subsequently, two solvents were used in column tests to determine rejuvenation efficiency under flow conditions.

1.1 Background on Permeable Reactive Barriers Used To Treat Ground Water for Uranium

More than 150 million tons of uranium mill tailings have been removed from 22 former uranium ore-processing sites in the United States. Remediation of ground water at these sites is mandated by Congress and was formerly conducted by the DOE Uranium Mill Tailings Remedial Action (UMTRA) Ground Water Project (NRC 1980) and now is being performed by the DOE Office of Legacy Management. EPA promulgated a ground water concentration limit of 30 picocuries per liter (pCi/L) (approximately 44 micrograms per liter [µg/L]) for uranium (U) to ensure protection of human health and the environment near these sites (EPA 1995); this U concentration is also being used as a ground water cleanup goal at a former uranium-ore processing site near Monticello, Utah. At many of these former ore processing sites, U has entered the ground water system and has contaminated more than 10 billion gallons of ground water (DOE 1996). Uranium ore processing outside the United States, particularly in Australia, Canada, South Africa, and Europe, has also resulted in significant ground water with U contamination. In addition to tailings sites, U has been reported in ground water at 12 of 18 major DOE facilities because of contamination from the weapons production cycle (Riley et al. 1992).

Cost-effective means of cleaning up ground water contaminated by U are needed. Ground water at some of the tailings sites is being extracted and treated ex situ, but costs for ex situ treatment are high and no site has yet been remediated to EPA's prescribed standards. PRBs to treat ground water contaminated by U are currently being tested at four sites (Monticello, Utah; Fry Canyon, Utah; Durango, Colorado; and Oak Ridge National Laboratory Y-12 Plant, Tennessee) as a low-cost alternative to pumping and treating ground water.

ZVI, a scrap-metal product that is available from the automotive industry, is being used as a reactive material in the PRBs at these four sites. Contact with ZVI causes U concentrations in ground water to decrease to a few micrograms per liter. Results of numerous laboratory experiments have confirmed the ability of ZVI to remove U from ground water. Because of the promising results of laboratory and field studies, project managers are expressing increasing interest in using ZVI to treat U contamination in ground water. Research is still needed, however, to understand the mechanisms of U uptake to support optimal designs for remediation systems and to make accurate predictions of the length of time that PRBs will remain effective.

1.2 Background on Monticello Permeable Reactive Barrier

This project is directly applicable to the Monticello, Utah, Mill Tailings National Priorities List site. An in situ PRB was installed hydraulically downgradient of the Monticello site in 1999; it is a funnel-and-gate system with a three-zone PRB (Morrison et al. 2002). The furthest upgradient zone (the pretreatment zone) has 13-percent ZVI by volume mixed with pea gravel. Downgradient from the pretreatment zone is a zone of 100-percent ZVI, followed by the third zone that contains 100-percent gravel and an air sparging unit (Figure 1).

2.0 Batch Test Results

Bench-scale tests were conducted to evaluate the potential for rejuvenation of ZVI PRBs using chemical solvent flushing. We tested various solvents, including free acid ammonium oxalate, diethylenetriamine pentaacetic acid (DPTA), disodium EDTA, ethylenediaminetetraacetic acid (EDTA), hydroxylamine hydrochloride, sodium citrate, sodium dithionite, and tetrasodium EDTA, and the commercial products Lime Away, and Lime Out (designed to remove calcium and iron scale deposits). Some tests were conducted with combinations of these solvents and sometimes included bicarbonate or carbonate as a pH buffer. Rejuvenation agents are considered more favorable if they dissolved more calcite (a ZVI corrosion product that occludes porosity), less ZVI, and have low toxicity. The ability to dissolve amorphous ferric oxyhydroxide, hematite and magnetite was also considered favorable. Most of the individual solvents and combinations of solvents were able to dissolve some calcite, but they also dissolved some ZVI.

Batch tests were conducted by combining a solid material (Table 1) with a solvent, agitating for a period of time, filtering, and then analyzing the filtrate for calcium (Ca) and iron (Fe). The amount of Ca or Fe removed was used as an indication of the amount of solid material that dissolved. Except where indicated otherwise, standard batch tests were conducted by combining 40 milliliters (mL) of the solvent with 0.5 gram (g) of a standard specimen of either powdered amorphous ferric oxyhydroxide (AFO), calcite, hematite, magnetite, or granular ZVI. Agitation was conducted in 50-mL glass Erlenmeyer flasks using an orbital motion in a temperature-controlled bath (Precision Model 25) at 25 °C. The mixtures were centrifuged, and the supernatant solution were analyzed for Ca and Fe by flame atomic absorption spectroscopy (DOE 2003; ESL Procedures AP[Ca-1] and AP[Fe-1]).

2.1.1 Sodium Acetate Buffer

Sodium acetate (NaOAc) adjusted to a pH value of 5 using glacial acetic acid (NaOAc buffer) is often used to remove calcite from clay samples in preparation for hydrometer tests (Jackson 1979). The NaOAc buffer is used because it can remove calcite without affecting the clay mineral composition.

NaOAc buffer (50 mL) was combined with 50 milligrams (mg) of powdered calcite in a 50-mL plastic centrifuge tube. Three additional tubes contained 2 grams (g) of the fresh (this report uses the term ‘fresh’ to indicate material that has not been used in a PRB) gravel/ZVI mixture used in the gravel/ZVI zone in the Monticello PRB and 50 mL of NaOAc buffer. The tubes were agitated end-over-end for various periods of time at room temperature. After agitation, they were centrifuged, decanted, preserved with nitric acid, and analyzed for Ca and Fe.

Gas was generated, and, consequently, an increase in pressure occurred during agitation in the tests with gravel/ZVI; a small amount of liquid was lost by leakage. The pressure increase was probably due to generation of hydrogen (H_2) gas from ZVI corrosion. About 88 percent of the calcite was dissolved, indicating that NaOAc was effective at removing calcite from the mixture (Table 2). Calcium was also removed from the three gravel/ZVI samples in approximately equivalent amounts, suggesting that all the calcite in the samples was dissolved. As much as 301 mg of ZVI was also dissolved. For the sample that agitated for 39 hours, about 86 percent of the ZVI was dissolved based on 13 percent by volume ZVI in the gravel/ZVI; the density of the

ZVI was 2.4 grams per milliliter (g/mL), and the density of the gravel was 1.7 g/mL. Because NaOAc dissolves ZVI, it is not suitable as a chemical rejuvenation agent.

2.1.2 EDTA and DTPA

Ethylenediaminetetraacetic acid (EDTA) and diethylenetriamine pentaacetic acid (DTPA) are industrial organic chelating agents used in cleansers, vegetable oils, and pharmaceuticals; they are also used to decontaminate radioactive surfaces and to remove insoluble deposits of Ca. The use of EDTA and DTPA in many consumable items suggests low toxicity. Free acid EDTA with a formula $C_{10}H_{16}O_8N_2$ (Fisher Scientific, BP118), tetrasodium EDTA with a formula of $(NaOCOCH_2)_2NCH_2CH_2N(CH_2COONa)_2 \bullet H_2O$ (Baker Chemicals I693-7), disodium EDTA with a formula of $C_{10}H_{14}N_2Na_2O_8 \bullet 2H_2O$ (Baker Chemicals, 4040-01), and DTPA with a formula $C_{14}H_{23}N_3O_{10}$ (Acros Organics) were tested in this study as potential ZVI PRB rejuvenation agents. The 0.1 molar (M) solutions of EDTA, tetrasodium EDTA, disodium EDTA, and DTPA have pH values of about 2.8, 10.8, 5.3, and 2.5, respectively. All chemicals were reagent grade.

In one set of tests, 50 mL of 0.1 M tetrasodium EDTA was combined with 2 g of fresh gravel/ZVI samples and various amounts of concentrated hydrochloric acid (HCl) to adjust the pH value (Table 3; tests 1 through 3). The mixtures were agitated end-over-end for 9 hours, centrifuged, and decanted. Values of final pH ranged from 8.25 to 11.02. The amount of Ca removed from the samples was similar at all three pH values and was more than the amounts removed by NaOAc (Table 2). The amount of Fe removed from the gravel/ZVI mixtures increased significantly as pH decreased (Table 3; tests 1 through 3).

Another set of tests evaluated the ability of 0.1 M tetrasodium EDTA to dissolve powdered calcite, hematite, magnetite, and ZVI from standard specimens. The tetrasodium EDTA was moderately effective at dissolving calcite, but it did not dissolve much of the Fe minerals (Table 3; tests 4 through 7). In another set of tests, the effect of the tetrasodium EDTA concentration on dissolution of a mixture of calcite and ZVI was examined. Lowering the tetrasodium EDTA concentration from 0.1 to 0.025 M caused a decrease in the dissolution of both calcite and ZVI (Table 3; tests 7 through 10). The combined results indicate that tetrasodium EDTA in a solution with a high pH value may be beneficial for ZVI PRB rejuvenation because it can remove large amounts of calcite while leaving most of the ZVI intact.

Table 4 displays the results of the dissolution of standard test specimens by free acid EDTA. A 0.1 M solution of free acid EDTA was oversaturated, as indicated visually by the occurrence of residual powder and by low electrical conductivity values of the solutions. Values of electrical conductivity in the free acid EDTA solutions ranged from 227 to 662 microsiemens per centimeter (µS/cm) (Table 4) compared to a range of 10,940 to 11,160 µS/cm in a 0.1 M solution of disodium EDTA (Table 5). Thus, the free acid EDTA tests were conducted with a saturated solution. The saturated solution of free acid EDTA did not remove much calcite and would not be effective for PRB rejuvenation.

A 0.1 M solution of disodium EDTA was effective in removing at least 80 percent of the calcite (Table 5). There was a small amount of solution loss, and it is likely that all the calcite was actually dissolved. About 3 percent of the ZVI was dissolved by the disodium EDTA. The much higher removal of calcite compared to ZVI suggests that disodium EDTA may be effective in rejuvenating PRBs. Unfortunately, only a small amount of the Fe oxides was dissolved.

Similar to free acid EDTA, a 0.1 M solution of DTPA could not be made because its solubility is too low. A saturated solution of DTPA dissolved about 13 percent of the calcite and 3 percent of the ZVI (Table 6). The results suggest that DTPA is less efficient than disodium EDTA and tetrasodium EDTA for removing calcite.

2.1.3 Ammonium Oxalate

Ammonium oxalate [$(NH_4)_2C_2O_4 \bullet H_2O$] solution buffered with oxalic acid to a pH value of 3 is used to selectively remove amorphous, or poorly crystalline ferric oxides from soil samples (Smith and Mitchell 1987). The extractant is prepared by mixing 0.2 M ammonium oxalate solution with 0.2 M oxalic acid in the proportion 4:3 by volume.
Despite the low pH value, the buffered ammonium oxalate extractant was ineffective at removing calcite (Table 7). In the rejuvenation tests with iron-based materials, it was most effective in removing Fe from AFO and magnetite. In addition to its ineffectiveness at removing calcite, ammonium oxalate is one of the most toxic of the compounds tested. Ammonium oxalate is not suitable for ZVI PRB rejuvenation.

2.1.4 Buffered Sodium Dithionite and Sodium Citrate

Sodium dithionite ($Na_2S_2O_4$, also called sodium hydrosulfite) is commonly used with sodium citrate and sodium bicarbonate to selectively remove crystalline ferric iron from soil samples (Jackson 1979). This extractant is often referred to as citrate bicarbonate dithionite (CBD). Each constituent of the CBD extractant has a purpose in helping to dissolve ferric minerals; the citrate chelates Fe, dithionite chemically reduces ferric iron to ferrous iron, and bicarbonate buffers the pH. Citrate, by itself, is often used to chelate metals and is also used in many consumable products. Citrate and bicarbonate are nontoxic.

Injection of a dissolved form of sodium dithionite has also been used for remediating chromium contamination in ground water (Vermeul et al. 2002). As with CBD, sodium dithionite causes reduction of ferric iron to ferrous iron. In the subsurface, sodium dithionite decomposes rapidly to compounds with low toxicities.

Extraction of Fe and Ca was tested with mixtures containing sodium dithionite, sodium citrate, and/or sodium bicarbonate using the previously described standard procedure. Sodium dithionite, by itself, removed 6.8 mg Ca from the calcite sample (about 3 percent of the calcite) and 5.6 mg of Fe from the ZVI sample (Table 8; tests 1 through 5). It removed as much as 17.4 mg of Fe from AFO, 8.2 mg of Fe from magnetite, and 10.4 mg of Fe from hematite. Although the sodium dithionite was able to preserve the ZVI reasonably well, it was unable to dissolve a significant portion of calcite.

Sodium citrate, combined with sodium bicarbonate, extracted 10.0 mg of Ca from the calcite sample (about 5 percent of the calcite) and minimal Fe (Table 8; items 6 through 10). However, the dissolution of iron-based materials was highest for ZVI. These results suggest that sodium citrate may be useful in removing a small portion of calcite from a PRB but may dissolve some ZVI while leaving Fe corrosion products intact.

A solution of CBD (0.27 M sodium citrate, 0.11 M sodium bicarbonate, and 0.1 M sodium dithionite), mixed in the same proportions as used in Jackson (1979), was tested for extraction of calcite, Fe oxides, and ZVI (Table 8, items 11 through 15). CBD removed 8.4 mg of Ca from the calcite sample (about 4 percent of the calcite). It also removed some of the ferric oxides, but the ZVI was least affected. The results with CBD are similar to those with dithionite alone, but more AFO was removed with CBD.

Results of some of the previous tests suggest that an increase in pH values in a CBD extractant may have a positive effect on calcite dissolution. Therefore, an extractant using citrate, carbonate, and dithionite (CCD) was designed that used carbonate (as potassium carbonate [K_2CO_3]) instead of bicarbonate to buffer pH. The pH value of CCD is 9.70 compared to 6.98 for CBD. Tests were conducted in the same manner as previous tests, and the results are presented in Table 8 (items 16 through 20). Less calcite was dissolved using CCD than with CBD; no other significant changes were apparent.

2.1.5 Sodium Dithionite and Tetrasodium EDTA

The CBD extractant previously discussed combines a chelating agent (citrate) with a chemical reductant (dithionite). The effects of using tetrasodium EDTA instead of citrate as the chelating agent were investigated. The same standard test procedure as described previously was used.

A solution containing low-strength 0.038 M tetrasodium EDTA and 0.025 M sodium dithionite was used. For additional control, tests were conducted simultaneously using only tetrasodium EDTA (Table 9). By comparing the Ca and Fe removal with tetrasodium EDTA alone (Table 9, tests 1 through 5) to tests with tetrasodium EDTA and dithionite (Table 9, tests 6 through 10), it was determined that the presence of dithionite had little effect on mineral dissolution.

Some of the previous test results suggest that the pH values of extractants composed of EDTA and dithionite may have an effect on the mineral dissolution capability. A titration of tetrasodium EDTA with a sodium dithionite solution was conducted to determine a solution composition that has a pH value greater than the 9.17 used in the previous tests. The selected high-strength solution contains 0.16 M tetrasodium EDTA and 0.057 M sodium dithionite and has a pH value of 9.69 (Table 10; tests 6 through 10). Tests were conducted with this high-strength mixture in the same manner as described for the low-strength tetrasodium EDTA and sodium dithionite solution.

Table 10 presents the results of the high-strength sodium dithionite and tetrasodium EDTA solution tests and the tests with 0.1 M tetrasodium EDTA. The test solutions containing sodium dithionite (Table 10; tests 6 through 10) dissolved essentially the same amounts of calcite as 0.1 M tetrasodium EDTA alone (Table 10; tests 1 through 5). Slightly more iron oxide minerals were dissolved by the high-strength solution than by the low-strength solution (compare data in Table 10 to data in Table 9). However, the results of the high-strength mixture do not represent a significant improvement over the low-strength mixture.

2.1.6 Hydroxylamine Hydrochloride

Hydroxylamine hydrochloride ($NH_2OH \bullet HCl$) mixed with acetic acid (CH_3COOH) has been used as an extractant to selectively remove iron and manganese oxides from soil samples (Landa 1982). The solution used for the tests in this study was made by combining 1 M hydroxylamine hydrochloride solution with 25 percent (by volume) concentrated glacial acetic acid, the same as used by Landa (1982). The tests were conducted using the previously described standard batch test method.

The pH value of the hydroxylamine hydrochloride solution is 1.48. The solution removed 224.8 mg of Ca from the calcite sample, which is essentially total dissolution (Table 11). Unfortunately, this extractant also dissolved 86.4 mg of Fe from the ZVI sample (about 17 percent of the sample). The ferric oxides were relatively unaffected, bringing into question the use of this extractant for selective removal of ferric oxides. Hydroxylamine hydrochloride is more toxic than most of the other extractants tested.

2.1.7 Lime Away and Lime Out

Lime Away (Reckitt Benckiser, Inc., Parsippany, New Jersey) and Lime Out (Iron Out, Ft. Wayne, Indiana) are commercial cleansers designed to remove calcium and iron scale in the home. These products were used full strength as purchased at a retail hardware store. The composition of these products is unknown. The pH values of Lime Away and Lime Out are 1.34 and 1.12, respectively. The tests were conducted using the previously described standard batch method.

Both products removed a significant amount of calcite (Table 12). The powdered calcite effervesced when contacted by both Lime Away and Lime Out, indicating the outgassing of carbon dioxide caused by the low pH. A small amount of material was spilled during the rapid effervescence, and the Ca values in Table 12 are probably low; it appeared that all the calcite dissolved. Lime Away was also able to dissolve a small amount of AFO. Both products dissolved a small amount of ZVI. Because of their ability to dissolve a large quantity of calcite while dissolving only a small amount of ZVI, these products may be suitable for PRB rejuvenation.

2.1.8 Summary of Batch Rejuvenation Tests

All results reported in this section were conducted suing the same test method. This method consists of orbital agitation of a 40-mL sample in a 50-mL glass volumetric Erlenmeyer flask in temperature controlled bath at 25 °C for 2 hours. Figure 1 presents a summary of the results of these tests that had the same methodology. Extractants that remove large amounts of calcite and small amounts of ZVI are considered most favorable for rejuvenating a ZVI PRB. Removal of large amounts of AFO, hematite, and magnetite is also considered beneficial.

Hydroxylamine hydrochloride, disodium EDTA, DTPA, Lime Away, and Lime Out were the most effective extractants tested for the removal of calcite. Unfortunately, these same extractants also dissolved slightly more ZVI than other extractants; hydroxylamine hydrochloride removed the most ZVI. Tetrasodium EDTA removed some calcite while leaving the ZVI relatively intact. Therefore, disodium EDTA and tetrasodium EDTA were selected for preliminary column tests. Unfortunately, tetrasodium EDTA was relatively ineffective in removing ferric oxides. Disodium EDTA was also used in column tests to determine its ability to improve the reactivity of ZVI for uranium removal.

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3.0 Column Test Results

Preliminary column tests were conducted to better evaluate the effectiveness of disodium EDTA and tetrasodium EDTA as extractants to rejuvenate ZVI PRBs.

3.1.1 Column Test 1, Ca and Fe Removal by Tetrasodium EDTA

This test was designed to determine if tetrasodium EDTA could effectively remove Ca from the Monticello PRB gravel/ZVI mixture.

3.1.1.1 Methods

This test was conducted in a 2-inch (in.)-diameter clear acrylic column packed with a fresh gravel/ZVI mixture (the same material that was emplaced in the gravel/ZVI zone of the Monticello PRB). The gravel/ZVI is a mixture of 2,500 pounds (lb) of –4 +20 Peerless ZVI to 9 cubic yards (yd^3) of 3/8-in. pea gravel. Dry weight of the gravel/ZVI in the column was 1,602.5 g. A peristaltic pump was used to flow influent solution from the bottom to the top of the column.

Influent to the column was synthesized to be similar in major ion composition to the ground water collected from well R1-M3, located immediately hydraulically upgradient of the Monticello PRB. Table 13 presents the compositions of the R1-M3 ground water and the synthesized column influent water. Influent solution was stored in a 20-liter (L) plastic carboy feed tank that was constantly stirred with a magnetic stir bar. Gaseous carbon dioxide (CO_2) flowing into the feed tank through a gas diffuser stone was used to control influent pH and alkalinity values. Flow rates for CO_2 ranged from 0 to 20 milliliters per minute (mL/min).

A programmable fraction collector was used to collect samples for analysis. Calcium and Fe concentrations were determined by atomic absorption on samples preserved with nitric acid. Alkalinity was measured by titration with sulfuric acid. Inline probes measured pH and oxidation-reduction potential (ORP) values, and data were fed to an automated data collection system.

3.1.1.2 Results

Synthetic ground water flowed through the column at 0.7 mL/min for about 12 days. During that time, the mean influent and effluent pH values were about 6.8 and 7.5, respectively (Figure 2). The mean ORP values were about +180 and less than –400 millivolts (mV), respectively. The Ca inventory in the column solids increased by about 1 g because of Ca-mineral precipitation (Figure 3). The Ca inventory was determined from the difference in influent and effluent Ca concentrations. There was no significant change in the Fe inventory during the flow of EDTA-free water (Figure 3).

After about 12 days (32 pore volumes), the influent was changed to 0.1 M tetrasodium EDTA and the flow rate was increased to 10 mL/min for about 5 pore volumes before switching back to synthetic ground water. During EDTA treatment, the pH value in the column effluent increased to about 11.5, similar to the EDTA influent value of about 11 (Figure 3). Calcium and Fe concentrations in the effluent EDTA solution increased to maximum values of 2,000 and 2,520 mg/L, respectively (Figure 2).

Although the concentration of Fe in the effluent increased substantially during EDTA injection, the inventory of Fe decreased only slightly (Figure 3). In contrast, all the Ca precipitated from the influent water and an additional 1.2 g (total of 2.2 g) was removed by the EDTA solution (Figure 3). The additional Ca was present in calcium carbonate minerals in the gravel. These results are encouraging for the use of tetrasodium EDTA as a rejuvenation agent.

3.1.2 Column Test 2, Ca and Fe Removal by Disodium EDTA

This test was designed to determine if disodium EDTA could effectively remove Ca from the Monticello PRB gravel/ZVI mixture.

3.1.2.1 Methods

Column test 2 was conducted using the same apparatus and methods as column test 1 (see previous section). The test was conducted in a 2-in.-diameter clear acrylic column packed with a fresh gravel/ZVI mixture (the same material that was used in the gravel/ZVI zone of the Monticello PRB). The gravel/ZVI is a mixture of 2,500 lb of –4 +20 Peerless ZVI to 9 yd^3 of 3/8-in. pea gravel. Dry weight of the gravel/ZVI in the column was 1,537.6 g. A peristaltic pump was used to flow influent solution from the bottom to the top of the column.

Influent to the column was synthesized to be similar in major ion composition to the ground water collected from well R1-M3, located immediately hydraulically upgradient of the Monticello PRB. Table 13 presents the compositions of the R1-M3 ground water and the synthesized column influent water. Influent solution was stored in a 20-L plastic carboy feed tank that was constantly stirred with a magnetic stir bar. Gaseous CO_2 flowing into the feed tank through a gas diffuser stone was used to control influent pH and alkalinity values. Flow rates for CO_2 ranged from 0 to 20 mL/min.

A programmable fraction collector was used to collect samples for analysis. Calcium and Fe concentrations were determined by atomic absorption on samples preserved with nitric acid. Alkalinity was measured by titration with sulfuric acid. Inline probes measured pH and ORP values, and data were fed to an automated data collection system.

3.1.2.2 Results

Synthetic ground water flowed through the column at 0.7 mL/min for about 7 days. During that time, the mean influent and effluent pH values were about 6.7 and 7.2, respectively (Figure 4). The mean ORP values were about +180 and less than –400 mV, respectively. The Ca inventory in the column solids increased by about 300 mg because of Ca-mineral precipitation (Figure 4). The Ca inventory was determined from the difference in influent and effluent Ca concentrations. There was no significant change in the Fe inventory during the flow of EDTA-free water (Figure 4).

After about 7 days (19 pore volumes), the influent was changed to 0.1 M disodium EDTA, and the flow rate was increased to 10 mL/min for about 5 pore volumes before switching back to synthetic ground water. The pH value in the column effluent decreased to about 5.3, similar to the EDTA influent value of about 5.1 (Figure 4). Calcium and Fe concentrations in the effluent EDTA solution increased to maximum values of 1,300 and 4,400 mg/L, respectively. Interestingly, the maximum effluent Ca concentration (1,300 mg/L) in the disodium EDTA column test was significantly less than the maximum effluent Ca concentration (2,000 mg/L) in the tetrasodium EDTA column test despite the greater effectiveness of the disodium form to dissolve calcite in the batch tests (Figure 1).

Although the concentration of Fe in the effluent increased substantially during EDTA injection, the inventory of Fe decreased only slightly (Figure 4). In contrast, all the Ca precipitated from the influent water and an additional 14,00 mg (a total of 1,700 mg) was removed by the EDTA solution (Figure 4). The additional Ca was present in calcium carbonate minerals in the gravel. These results are encouraging for the use of disodium EDTA as a rejuvenation agent.

3.1.3 Column Tests 3 and 4, Uranium Reactivity of a Column Treated With EDTA

These tests were designed to determine if the addition of disodium EDTA could improve the reactivity of ZVI for uranium removal. Two columns, one treated and one not, were run in parallel to evaluate the effectiveness of the treatment.

3.1.3.1 Methods

Column tests 3 and 4 were conducted in small (15-mm long, 22-mL volume) glass columns packed with a ZVI sample (sample PE-11-5, from about 6 in. from the contact with the gravel/ZVI zone) collected by coring the Monticello PRB in August 2003. The ZVI has been resident in the ground and reacting with ground water since 1999. A peristaltic pump was used to flow influent solution from the bottom to the top of the columns.

Column 3 was treated with 120 mL of 0.1 M disodium EDTA. The EDTA was flowed through the column at 3 mL/min. After this treatment, U-spiked synthesized ground water was flowed initially at 1.2 mL/min (residence time of about 13 min) through both columns in parallel. Because both columns removed U to less than detection, the flow rate was increased to 3.6 mL/min (residence time of about 4 min); about 90 percent of the test used a flow rate of 3.6 mL/min. The synthesized ground water had a major ion composition similar to ground water obtained from well R1-M3, located immediately hydraulically upgradient of the Monticello PRB. In contrast to column tests 1 and 3, this test used synthesized R1-M3 water that was spiked with about 1 mg/L U. Table13 presents the compositions of the R1-M3 ground water and the synthesized column influent water (without the U).

A programmable fraction collector was used to collect samples for analysis. Calcium and Fe concentrations were determined by atomic absorption on samples preserved with nitric acid. Uranium concentrations were determined by kinetic phosphorescence analysis (DOE 2003; ESL Procedure AP[U-2]).

3.1.3.2 Results

Figure 5 presents the results of the uranium removal column tests. During EDTA treatment, 478 mg of calcite was removed from the ZVI sample in column 3. Most of the effluent U concentration were less than 10 µg/L, considerably less than the influent concentration of 1,000 µg/L. Despite the relatively short residence time of 4 min in contact with the ZVI, both columns removed more than 99 percent of the U for more than 300 pore volumes. The differences observed between the treated and untreated columns were insignificant. The U removal efficiency in the untreated column is too high to evaluate the effects of the EDTA treatment properly and, thus, the results are inconclusive. It is interesting, however, that the U removal rates in this sample from the PRB are still very high, suggesting that the PRB longevity may exceed the values estimated by Morrison (2003).

4.0 Conclusions and Recommendations

This report presents data from a preliminary evaluation of ZVI rejuvenation using various chemical solvents. Suitable rejuvenation agents were defined by the high ability to dissolve calcite (a ZVI corrosion product that occludes porosity), low dissolution of ZVI, and low toxicity; the ability to dissolve AFO, hematite, and magnetite was also considered favorable. Most of the solvents and combinations were able to dissolve some calcite, but most also dissolved some ZVI. Column test results indicate that both tetrasodium EDTA (pH about 11) and disodium EDTA (pH about 7) were able to remove all the calcite deposited during ZVI corrosion and some of the calcite initially present in the column fill material. While some ZVI was also dissolved by EDTA, the change in the Fe inventory was insignificant. Dissolved EDTA has minimal negative effects on human health. The results indicate that EDTA may be an effective chemical agent for PRB rejuvenation. A field pilot study is recommended to determine if rejuvenation with EDTA can improve PRB performance.

End of current text

5.0 References

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Table 1. Standard Materials Used in Batch Tests

Name	Vendor	Description
Calcite Powder	Aldrich	Reagent grade. Calcite only.[a]
AFO	Noah Industries	AFO slurry dried at room temperature.
Hematite	Fisher Scientific I-116-3	Reagent grade. Hematite only.[a]
Magnetite	American Chemical Enterprises A-310	Reagent grade. Magnetite only.[a]
ZVI	Fisher 40 Mesh	Reagent grade ZVI. Sieved to about 40 mesh.
Gravel/ZVI	ZVI = Peerless –8 +20	Mixture used in Monticello gravel/ZVI zone.

[a]The mineralogy of these materials was identified by X-ray diffraction analysis.

Table 2. Batch Tests Using 50 mL of Sodium Acetate Buffer

Gravel/ZVI[a] (g)	Calcite Powder (mg)	Shake Time (hours)	Ca Removed (mg)	Fe Removed (mg)	Final pH
0	50	14	17.6[b]	0.1	5.01
2	0	14	5.1	132.5	5.10
2	0	20	4.6	163.5	5.14
2	0	39	3.8	301.0	5.36

[a]Fresh gravel/ZVI material used in the Monticello PRB.
[b]20 mg of Ca is equivalent to 100 percent removal of the calcite.

Table 3. Batch Tests Using 50 mL of Tetrasodium EDTA

Test	Solids	Tetrasodium EDTA Concentration	Concentration HCl (μL)	Shake Time (hours)	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	2 g gravel/ ZVI[a]	0.1 M	0	9	10.85	11.02	7.3	4.2
2	2 g gravel/ ZVI[a]	0.1 M	270	9	9.05	9.70	12.1	49.5
3	2 g gravel/ ZVI[a]	0.1 M	550	9	6.88	8.25	7.1	58.3
4	1 g Calcite	0.1 M	0	7.5	10.85	11.11	191	0.0
5	1 g ZVI[b]	0.1 M	0	7.5	10.85	11.13	0.1	4.0
6	1 g Hematite	0.1 M	0	7.5	10.85	10.76	0.1	0.1
7	1 g Magnetite	0.1 M	0	7.5	10.85	10.85	0.8	0.2
8	1 g Calcite + 1 g ZVI[b]	0.1 M	0	28	10.85	11.11	172.5	0.3
9	1 g Calcite + 1 g ZVI[b]	0.05 M	0	28	nm[c]	11.06	98.5	0.2
10	1 g Calcite + 1 g ZVI[b]	0.025 M	0	28	nm	10.90	46	0.1

[a]Fresh gravel/ZVI material, same as used at Monticello.
[b] –6 + 10 mesh Peerless ZVI.
[c] nm = not measured.

Table 4. Batch Tests Using Free Acid EDTA

Test	Solids[b]	Final Electrical Conductivity (μS/cm)	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	227	2.94	4.49	1.4	1.0
2	Hematite	584	2.94	2.88	0.0	0.1
3	Magnetite	595	2.94	2.90	0.3	0.3
4	Fisher 40 Mesh ZVI	662	2.94	2.80	0.0	2.9
5	Calcite	454	2.94	6.68	8.0[d]	0.0

[a] Tests conducted with 40 mL of saturated free acid EDTA and 0.5 g of solids; 2-hour agitation at 25 ºC.
[b] Descriptions are provided in Table 1.
[c] μS/cm = microsiemens per centimeter.
[d] 200 mg of Ca is equivalent to 100 percent removal of the calcite.

Table 5. Batch Tests Using Disodium EDTA

Test	Solids[b]	Final Electrical Conductivity (μS/cm)	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	11,120	5.04	5.32	3.5	2.4
2	Hematite	10,940	5.04	5.12	0.1	0.1
3	Magnetite	10,960	5.04	5.13	0.4	0.3
4	Fisher 40 Mesh ZVI	11,040	5.04	5.30	0.1	16.4
5	Calcite	11,160	5.04	6.20	159.2[d]	0.0

[a] Tests conducted with 40 mL of 0.1 M disodium EDTA and 0.5 g of solids; 2-hour agitation at 25 ºC.
[b] Descriptions are provided in Table 1.
[c] μS/cm = microsiemens per centimeter.
[d] 200 mg of Ca is equivalent to 100 percent removal of the calcite.

Table 6. Batch Tests Using DTPA

Test	Solids[b]	Final Electrical Conductivity (μS/cm)	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	2,170	2.27	2.39	2.5	3.8
2	Hematite	2,780	2.27	2.28	0.1	0.2
3	Magnetite	2,760	2.27	2.27	0.3	0.6
4	Fisher 40 Mesh ZVI	2,580	2.27	2.31	0.1	14.1
5	Calcite	1,774	2.27	5.79	66.4[d]	0.0

[a] Tests conducted with 40 mL of saturated DTPA and 0.5 g of solids; 2-hour agitation at 25 ºC.
[b] Descriptions are provided in Table 1.
[c] μS/cm = microsiemens per centimeter.
[d] 200 mg of Ca is equivalent to 100 percent removal of the calcite.

Table 7. Batch Tests Using 0.2 M Ammonium Oxalate

Test	Solids[b]	Start Electrical Conductivity (μS/cm)[c]	Final Electrical Conductivity (μS/cm)	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	20,300	20,100	3.00	3.18	0.0	15.8
2	Hematite	20,300	20,100	3.00	3.05	0.0	0.8
3	Magnetite	20,300	20,100	3.00	3.13	0.0	13.8
4	Fisher 40 Mesh ZVI	20,300	20,200	3.00	3.12	0.0	3.9
5	Calcite	20,300	20,200	3.00	3.14	0.0	0.0

[a] Tests conducted with 40 mL of 0.2 M ammonium oxalate buffered to a pH 3 value with 0.2 M oxalic acid and 0.5 g of solids; 2-h agitation at 25 ºC.
[b] Descriptions are provided in Table 1.
[c] μS/cm = microsiemens per centimeter.

Table 8. Batch Tests Using Citrate Bicarbonate Dithionite-Type Solutions

Test	Solids[b]	Solvent[c]	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	D	4.53	5.85	0.8	17.4
2	Hematite	D	4.53	4.83	0.0	10.4
3	Magnetite	D	4.53	4.11	0.1	8.2
4	Fisher 40 Mesh ZVI	D	4.53	3.75	0.0	5.6
5	Calcite	D	4.53	4.22	6.8	0.0
6	AFO	CB	8.16	8.49	2.5	0.1
7	Hematite	CB	8.16	8.41	0.1	0.0
8	Magnetite	CB	8.16	8.44	0.7	0.1
9	Fisher 40 Mesh ZVI	CB	8.16	8.57	0.1	2.7
10	Calcite	CB	8.16	8.68	10.0	0.0
11	AFO	CBD	6.98	7.44	2.5	42.0
12	Hematite	CBD	6.98	7.19	0.1	16.2
13	Magnetite	CBD	6.98	7.08	0.5	9.4
14	Fisher 40 Mesh ZVI	CBD	6.98	7.08	0.1	6.0
15	Calcite	CBD	6.98	7.13	8.4	0.0
16	AFO	CCD	9.70	8.58	1.5	15.1
17	Hematite	CCD	9.70	8.56	0.1	2.8
18	Magnetite	CCD	9.70	8.63	0.2	3.0
19	Fisher 40 Mesh ZVI	CCD	9.70	8.83	0.0	4.0
20	Calcite	CCD	9.70	8.44	1.0	0.0

[a]Tests conducted with 40 mL of solution, 0.5 g of solids, and 2-h agitation at 25 ºC.
[b]Descriptions are provided in Table 1.
[c]D = 0.1 M sodium dithionite; CB = 0.27 M sodium citrate with 0.11 M sodium bicarbonate; CBD = same as CB but with 0.1 M sodium dithionite; and CCD = 0.27 M sodium citrate with 0.11 M potassium carbonate and 0.1 M sodium dithionite.

Table 9. Batch Tests Using Low-Strength Sodium Dithionite and Tetrasodium EDTA

Test	Solids[b]	Solvent[c]	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	E	10.93	10.61	2.1	0.4
2	Hematite	E	10.93	10.79	0.1	0.0
3	Magnetite	E	10.93	10.82	0.3	0.0
4	Fisher 40 Mesh ZVI	E	10.93	10.88	0.1	1.0
5	Calcite	E	10.93	10.92	11.5	0.0
6	AFO	ED	9.17	9.30	1.7	0.5
7	Hematite	ED	9.17	9.25	0.1	0.0
8	Magnetite	ED	9.17	9.28	0.2	0.1
9	Fisher 40 Mesh ZVI	ED	9.17	9.37	0.0	4.3
10	Calcite	ED	9.17	9.36	9.0	0.0

[a] Tests conducted with 40 mL of solution, 0.5 g of solids, and 2-h agitation at 25 ºC.
[b] Descriptions are provided in Table 1.
[c] E = 0.05 M tetrasodium EDTA; ED = 0.038 M tetrasodium EDTA with 0.025 M sodium dithionite.

Table 10. Batch Tests Using High-Strength Sodium Dithionite and Tetrasodium EDTA

Test	Solids[b]	Solvent[c]	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	E	10.95	10.66	2.4	0.4
2	Hematite	E	10.95	10.80	0.1	0.0
3	Magnetite	E	10.95	10.85	0.4	0.1
4	Fisher 40 Mesh ZVI	E	10.95	10.97	0.1	1.1
5	Calcite	E	10.95	10.88	10.1	0.0
6	AFO	ED	9.69	9.21	1.5	6.7
7	Hematite	ED	9.69	9.19	0.1	4.8
8	Magnetite	ED	9.69	9.16	0.3	4.7
9	Fisher 40 Mesh ZVI	ED	9.69	9.16	0.1	4.2
10	Calcite	ED	9.69	9.20	7.3	0.0

[a] Tests conducted with 40 mL of solution, 0.5 g of solids, and 2-h agitation at 25 ºC.
[b] Descriptions are provided in Table 1.
[c] E = 0.1 M tetrasodium EDTA; ED = 0.16 M tetrasodium EDTA with 0.057 M sodium dithionite.

Table 11. Batch Tests Using Hydroxylamine Hydrochloride

Test	Solids[b]	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	1.48	1.52	1.9	7.4
2	Hematite	1.48	1.32	0.0	0.1
3	Magnetite	1.48	1.33	0.1	0.5
4	Fisher 40 Mesh ZVI	1.48	2.20	0.0	86.4
5	Calcite	1.48	2.67	224.8	0.1

[a]Tests conducted with 40 mL of solution, 0.5 g of solids, and 2-h agitation at 25 ºC.
[b]Descriptions are provided in Table 1.

Table 12. Batch Tests Using Lime Away and Lime Out

Test	Solids[b]	Solvent	Start pH	Final pH	Ca Removed (mg)	Fe Removed (mg)
1	AFO	Lime Away	1.34	1.47	2.9	16.4
2	Hematite	Lime Away	1.34	1.40	0.1	0.7
3	Magnetite	Lime Away	1.34	1.39	0.4	1.4
4	Fisher 40 Mesh ZVI	Lime Away	1.34	1.37	0.1	24.0
5	Calcite	Lime Away	1.34	1.37	131.2	0.3
6	AFO	Lime Out	1.12	1.25	1.2	1.5
7	Hematite	Lime Out	1.12	1.22	0.9	0.4
8	Magnetite	Lime Out	1.12	1.23	1.0	0.4
9	Fisher 40 Mesh ZVI	Lime Out	1.12	1.24	1.9	15.2
10	Calcite	Lime Out	1.12	1.27	146.8	0.2

[a] Tests conducted with 40 mL of full-strength Lime Away or Lime Out and 0.5 g of solids; 2-h agitation at 25 ºC.
[b] Descriptions are provided in Table 1.

Table 13. Composition of Well R1-M3 Ground Water (sampled January 14, 2003) and Synthesized Ground Water Used in Column Tests

Constituent	Units	Actual Concentration	Synthesized Concentration
Na	mg/L	111.00	150.60
K	mg/L	11.3[a]	11.22[a]
Ca	mg/L	213.00[a]	222.88[a]
Mg	mg/L	52.80	53.28
SO_4	mg/L	677.00	655.21
Cl	mg/L	76.60	76.09
TIC[b]	mg/L	76.00[c]	78.57
pH	s.u.	6.65	6.65[d]
Alkalinity	mg/L as $CaCO_3$	317.00	279[d]

[a] Corrected from DOE 2004
[b] Total inorganic carbon.
[c] Estimated from alkalinity.
[d] Varies with CO_2 flow.

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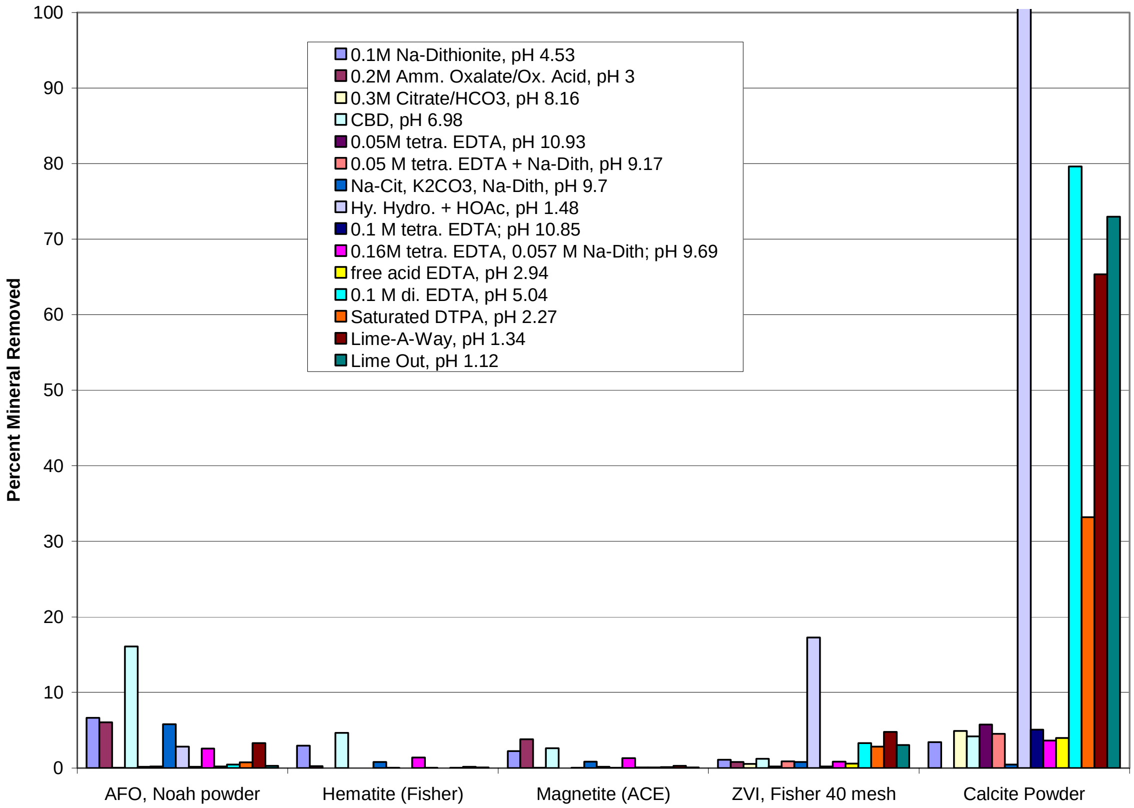

Figure 1. Results of Batch Tests

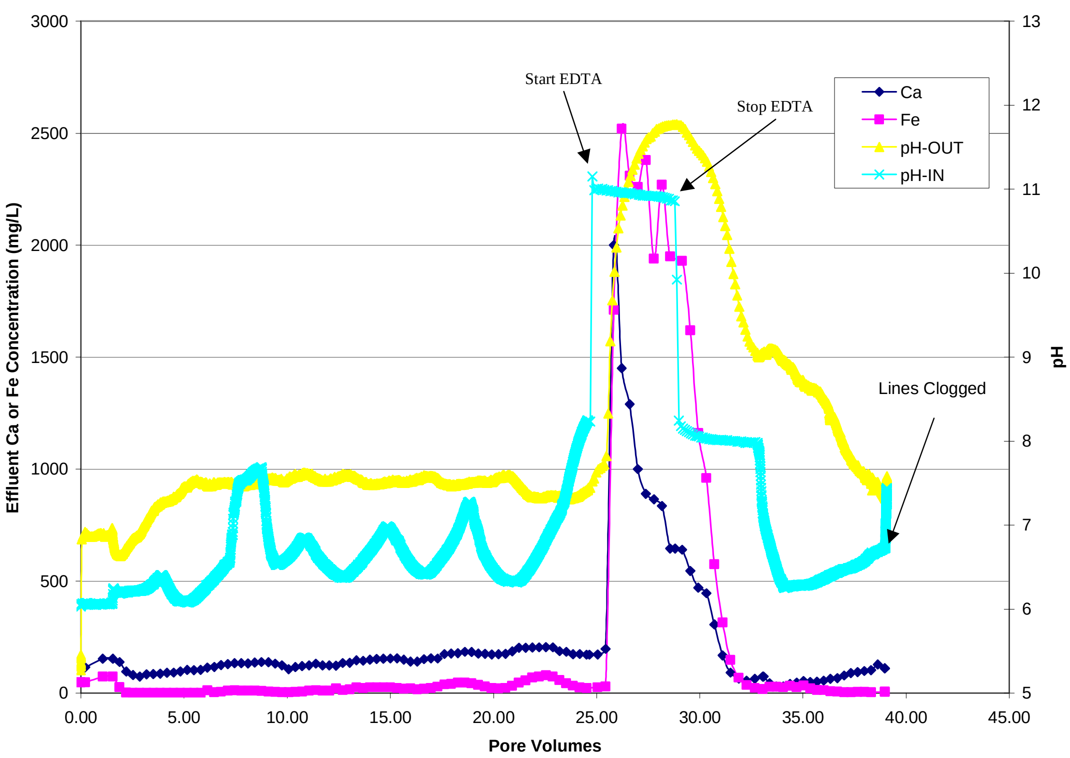

Figure 2. Results of Column 1: Tetrasodium EDTA

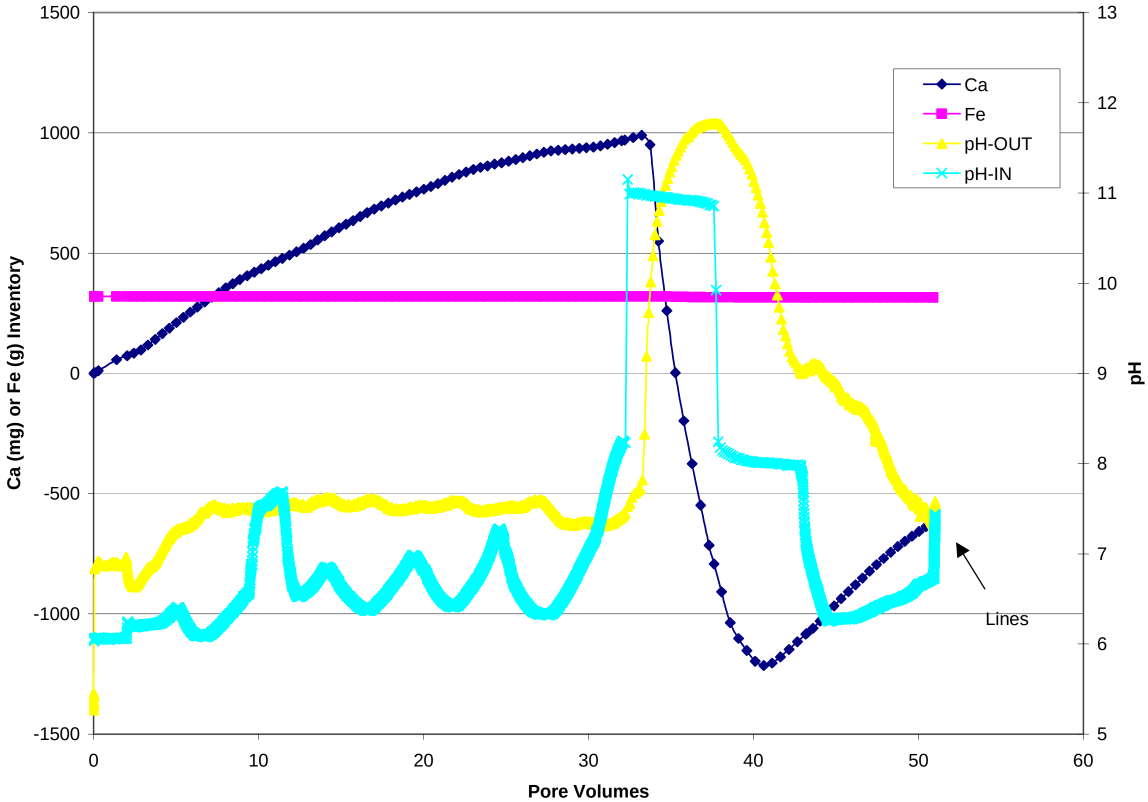

Figure 3. Chemical Inventory in Column Test 1

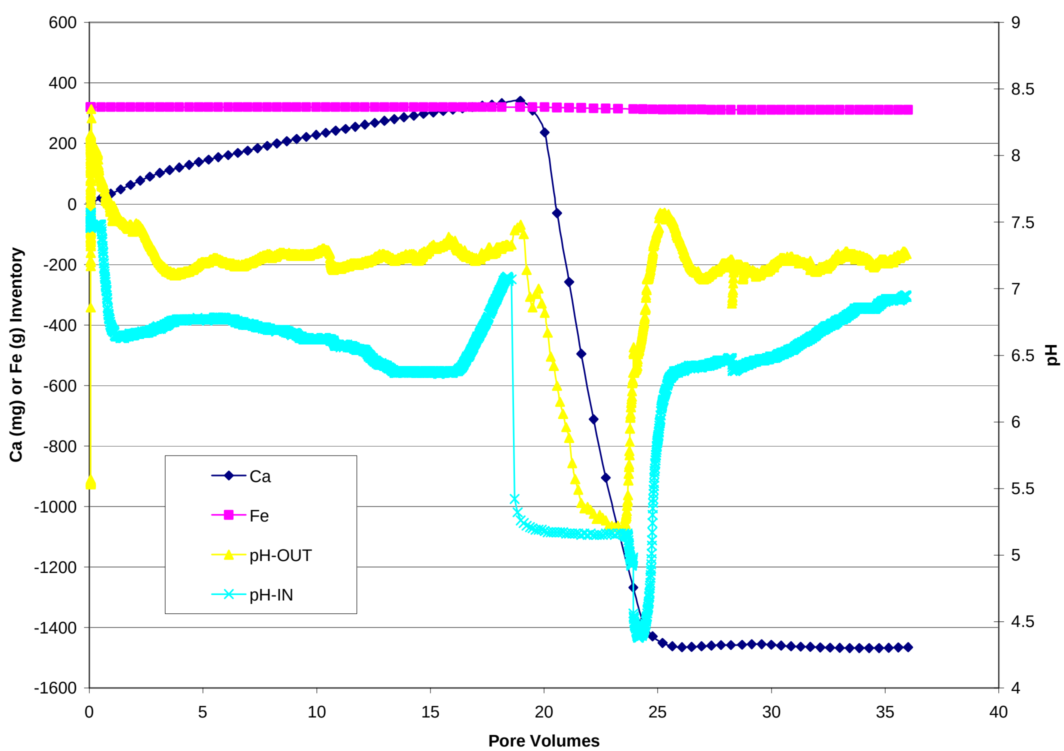

Figure 4. Chemical Inventory in Column Test 2: Disodium EDTA

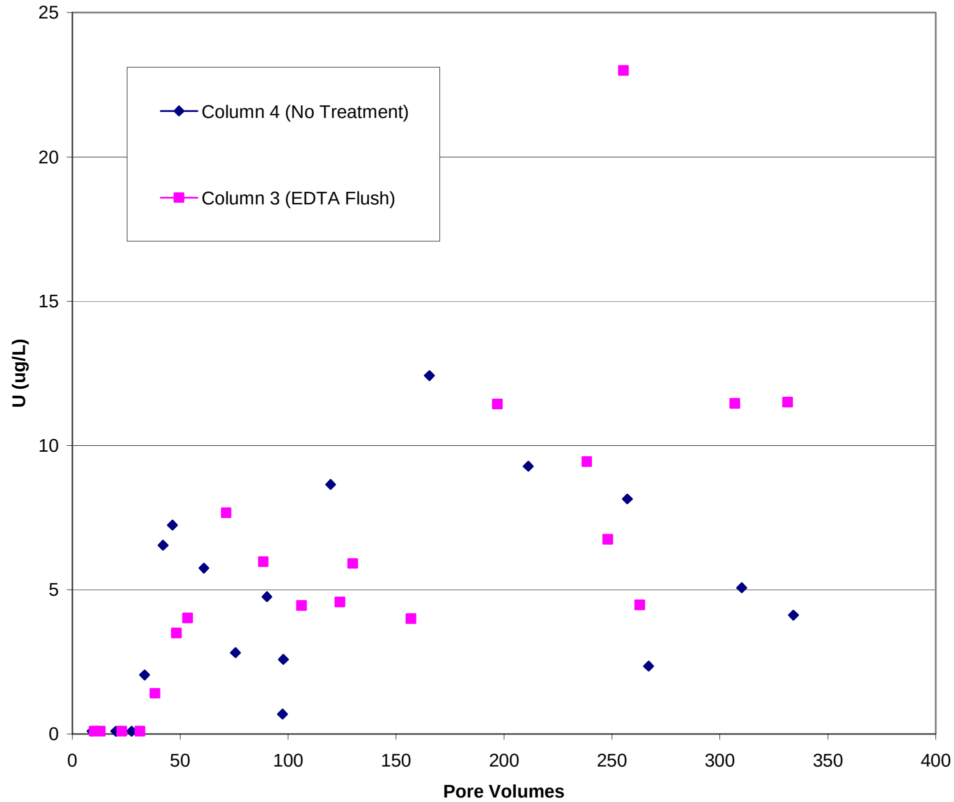

Figure 5. Effluent Uranium in Column Tests 3 and 4. Column 3 was flushed with disodium EDTA.

Appendix A

Environmental Sciences Laboratory Notes

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EFFICIENCY LINE® 22-210

Monticello PeRT Rejuvination MPERT01-09-01

4/30/03 Pack a 2" diameter column with fresh ZVI/gravel mix like that used in the ZVI/gravel zone @ PeRT wall in Monticello. It is a mix of ~ 2500 lbs -4/+20 iron to 9 cubic yards of gravel. Net weight of fill is 1602.5g

Prepare 20L RIM3 synthetic water by adding chemical solids to MilliQ water

	1L (g)	20L (g)
$NaHCO_3$	0.55	11.0
$CaSO_4 \cdot 2H_2O$	0.7725	15.45
$MgSO_4 \cdot 7H_2O$	0.54	10.8
$CaCl_2 \cdot 2H_2O$	0.1576	3.15
K_2SO_4	0.025	0.5

320g ZVI (assuming ~20% by weight)

1600 Place carboy on stir plate. Entrain CO_2 @ ~60cc/min. Monitor pH @ data logger initial pH = 6.89

1630 CO_2 flow ↓ to 20 mL/min

pH and ORP probes calibrated prior to monitoring. pH used 7 and 10 buffer. ORP used zobell @ +230.

5/1/03 0745 Check pH of source tank. pH = 5.908. DC CO_2 flow. alk = 279 mg/L as $CaCO_3$

0850 pH now @ 5.94

Note that data logger channels are:
1 ORP inlet
2 ORP outlet
3 pH inlet
4 pH outlet

file: APR30_03 is monitoring 20L source tank of influent c̄ CO_2 entrained pH only

Slope-intercept for ORP probe calibration (from factory) = intercept -389.76, slope 222.6

EFFICIENCY LINE® 22-210

Monticello PeRT Rejuvination MPERT 01-09-02

5/1/03 0915 Start flow to column @ 4ml/min

1123 First outflow. Collect in 50ml cent tube. That is 129 minutes till first programmed collect on fraction collector.

Fraction collector is set @ 234 minute drain, 6 minute collect.

Note outflow pH has not increased as expected.

1446 Change pump heads to size 16. Flow set @ 0.7ml minute. Will reprogram frac collector to 35 minute collects, 205 minute drain. 176 minutes left on drain.

flow changed @ [redacted] on data logger pH in 6.071 pH out 6.904

1130 * Flow starts to outflow sensors @ 11:30 [redacted] May01_03

1150 start drain to beaker.

1339 first collect on fraction collector 6" = 24ml rate = 4ml/min

PV = 512 ml (128 minutes x 4ml/minute)

1535 Start CO_2 flow @ 10 ml/min [redacted]

5/2/03 0847 pH inlet at 6.23. Turned off CO_2 gas (although it was barely flowing if at all). [redacted] MAY01_03

0852 Stopped data logger. Started new file

pH IN Intercept = 13.5801
Slope = -3.7123

0930 [redacted] ile (with time column)

Lost all data from 0852 – 0930

pH OUT Intercept = 13.1649
Slope = -3.7520

Had to [illegible]

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MPERT 01-09-03

5/2/03 1930 pH inlet is rising 6.375. Started 10 ml/min CO_2
Time = 9.833 hours
2019 Increased CO_2 to 20 ml/min

5/3/03 1028 Log time = 24.917 hrs. pH in was 6.094, Turned CO_2 off

5/4/03 1151 Log time = 50.333 hrs. pH in was 6.556, Turned CO_2 to 10 ml/min
Attached Aquarium pump with Aeration stone to add O_2 to source tank
ORP has drifted down slightly from about 200 to 179 mV

5/5/03 0701 pH in up to 7.675. Turned off air. Turned CO_2 on at 20 ml/min
Log time = 69.5 hours

1224 pH_{in} is 6.792. O/C CO_2 (log time = 74.917 hours)

1615 pH_{in} is 6.538 (logtime 78.667)

5/6/03 0632 Log = 93.000 pH in = 6.796 CO_2 turned on 10 ml/min

5/7/03 0643 Log = 117.25 hr. pH in = 6.075 CO_2 D/C

0 17:17 Stopped datalogger MAY02_03
17:20 Start MAY07_03 (0 hours)

5/8/03 0637 (logger 13.250 hrs) pH in at 6.896, turned CO_2 on 10 ml/min
Some tubes on fraction collector are not filling all the way. Some are only about 1/2 full. Lots of Fe staining (red) in lines.

5/9/03 0600 Turned CO_2 off. 100 ml Conc HNO_3 put in Tubes 31-54
(logger 36.007)

EFFICIENCY LINE® 22-210

MPERT 01-09-04

5/10/03	0748	CO_2 started 10 mL/min
	0906	Added 100 µL HNO_3 conc to tubes 50 – 74
	0906	Last tube collected was #57. Logger 63.750 hours
5/11/03	1213	D/C CO_2 Log time = [illegible] hr. ([illegible] 03) Last tube collected = #60
5/12/03	0619	Last Tube collected was [illegible] Log time = 109.000
		Added another 100 µL conc HNO_3 to tubes 50–74 (total 200 µL)
		Added 200 µL conc HNO_3 to tubes 75 – 9[illegible]
	0640	Started filling tube #65. Log time = 109.3[illegible]
	0653	Made 2L of 0.1 M EDTA (41.62 g/L) 83.24 g per 2L
	0712	Ended Tube #65 collect. Log Time = 109,833
	0717	Stopped File MAY07_03
	0718	Started File MAY12_03 (1000 hours)
5/13/03	0821	Start flow @ 10 mL/min of 0.1 M EDTA Log time @ 25.000. Change collect times to 18 min. drain / 2 min collect to get 20 mL split.
	0839	First collect (tube 72) after start of EDTA. Soln is clear. 0.2 mL HNO_3 in test tubes
	0859	tube 73 off - soln is clear
	0919-21	tube 74 off. soln is layered. bottom 1/3rd green, middle 1/3rd tan, top 1/3rd orange
	0939-41	75 as above
	0959-1001	76 as above
	1019-[illegible]	77 [illegible]

MPERT 01-09-05

5/12/03 1039-41 tube 78 off liquid on above
1059-1101 79 off no acid in test tube
5/13/03
1105 change outlet ORP probe. Check new probe prior to installation
[illegible] = +227

1119-21 tube 80 off. Note that tubes without acid are very pale orange with dark orange thin layer at top. Cloudy orange striation throughout tube

Prepare new 20L of R1 [illegible]3 using dissolved chemical soln to MQ H_2O

50g/L	$NaHCO_3$	220
1.5g/L	$CaSO_4 \cdot 2H_2O$	10300
200g/L	$MgSO_4 \cdot 7H_2O$	54
92.72g/L	$CaCl_2 \cdot 2H_2O$	5
100g/L	K_2SO_4	34
	MQH_2O	9387
		20,000

1153 Switch to new source tank = R1 [illegible]3 file time = 28.583 six minutes left on drain between tubes 81 and 82.

1159 Start tube 82 collect. file time 28.667

1400 Column appears to be running well. Samples are lightening in color

1420 Start CO_2 flow to source tank @ ~10 ml/min

1520 ↓ flow to 0.7 ml/min. Reprogram frac collector to 205 min drain/35 min collect
add acid [illegible]

MPERT 01-09-06

5/13/03 1655 (33.583 file time) Columns running well, will leave Co @ 10ml/min.

5/14/03 0723 Completed Tube 96. File time = 48.083

1123 complete tube 97 file time = 52.083

acidify tubes 79-92 (not acidified @ time of collection) to rerun Fe, Cu, use 0.5ml conc HNO_3
add addl HNO_3 to tubes 74-78 (pH was ~7 prior to addition) all now pH<2

1502 stop file collect MAY13-03 file time 55.750, restart MAY14-03 since clock time @ 1502
during RJV098 collect (20 minutes left to collect)

EFFICIENCY LINE® 22-210

Ca (source tank) = 219 mg/L (measured)
= 223 mg/L (calculated)

MPERT 01-09-07

Column Rejuvenation

Outflow/tube	Vol (ml)	Cum Vol (ml)	Rate (ml/min)	PV (1=512)	Color	Ca (mg/L)	Fe (mg/L)
1st outflow	25	25	4	0.05	Red	115	47.5
drain	516	541	4	1.06			
RJV001	24	565	4	1.10	Red	153	72.6
drain	224	789	4	1.54			
drain	18	807	0.7	1.58			
drain	102	909	0.7	1.78			
RJV002	23	932	0.7	1.82	Red	139	26.0
drain	144	1076	0.7	2.10			
RJV003	23	1099	0.7	2.15	clear	96	1.48
drain	144	1243	0.7	2.43			
RJV004	23	1266	0.7	2.47		80	0.91
drain	144	1410	0.7	2.75			
RJV005	23	1433	0.7	2.80		74	0.52
drain	144	1577	0.7	3.08			
RJV006	23	1600	0.7	3.13		84	0.43
drain	144	1744	0.7	3.41			
RJV007	23	1767	0.7	3.45		85	0.21
drain	144	1911	0.7	3.73			
RJV008	23	1934	0.7	3.78		87	0.23
drain	144	2078	0.7	4.06			
RJV009	23	2101	0.7	4.10		90	0.78
drain	144	2245	0.7	4.38			
RJV010	23	2268	0.7	4.43		91	0.99
drain	144	2412	0.7	4.71			
RJV011	23	2435	0.7	4.76		97	0.10
drain	144	2579	0.7	5.04			
RJV012	23	2602	0.7	5.08		103	0.12
drain	144	2746	0.7	5.36			
RJV013	23	2769	0.7	5.41		102	0.42
drain	144	2913	0.7	5.69			
RJV014	23	2936	0.7	5.73		104	0.85

EFFICIENCY LINE® 22-210

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Column Rejuvination

MPERT01-09-08

Outflow	Vol (mL)	Cum Vol (mL)	Rate (mL/min)	PV 1=512 (mL)	Color	Cu (mg/L)	Fe (mg/L)
drain	144	3080	0.7	6.02			
RJV015	23	3103	0.7	6.06	Red	114	12.3
drain	144	3247	0.7	6.34			
RJV016	23	3270	0.7	6.39		117	3.77
drain	144	3414	0.7	6.67			
RJV 017	23	3437	0.7	6.71		125	5.42
drain	144	3581	0.7	6.99			
RJV 018	23	3604	0.7	7.04		129	9.6
drain	144	3748	0.7	7.32			
RJV 019	23	3771	0.7	7.37		133	11.4
drain	144	3915	0.7	7.65			
RJV 020	23	3938	0.7	7.69		133	10.3
drain	144	4082	0.7	7.97			
RJV 021	23	4105	0.7	8.02		132	10.0
drain	144	4249	0.7	8.30			
RJV 022	23	4272	0.7	8.34		136	10.2
~~RJV 02~~ drain	144	4416	0.7	8.63			
RJV 023	23	4439	0.7	8.67		139	9.5
drain	144	4583	0.7	8.95			
RJV 024	24	4607	0.7	9.00		138	6.7
drain	144	4751	0.7	9.28			
RJV 025	24	4775	0.7	9.33		131	4.78
drain	144	4919	0.7	9.61			
RJV026	22	4941	0.6	9.65	pale yellow	124	4.00
drain	144	5085	0.7	9.93			
RJV 027	25	5110	0.7	9.98		107	3.72
drain	144	5254	0.7	10.26			
RJV 028	21	5275	0.6	10.30		116	4.15
drain	144	5419	0.7	10.58			
RJV 029	23	5442	0.7	10.63		120	6.60
drain	144	5586	0.7	10.91			

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MPERT01-09-09

Column Rejuvenation

Outflow/tube	1 Vol (ml)	2 Cum Vol (ml)	3 Rate (ml/min)	4 PV 1=512	5 Color	6 Ca (mg/L)	7 Fe (mg/L)	8	9	10	11	12	13
RJV030 1	23	5609	0.7	10.96	pale yellow	124	10.0						
drain 2	144	5753	0.7	11.24									
RJV031 3	22	5775	0.6	11.28		131	12.4						
drain 4	144	5919	0.7	11.56									
RJV032 5	23	5942	0.7	11.61		123	10.5						
drain 6	144	6086	0.7	11.89									
RJV 033 7	23	6109	0.7	11.93		123	10.5						
drain 8	144	6253	0.7	12.21									
RJV 034 9	16	6269	0.5	12.24	rust flocs	122	21.1						
drain 10	144	6413	0.7	12.53									
RJV 035 11	24	6437	0.7	12.57	pale yellow	134	12.6						
drain 12	144	6581	0.7	12.85									
RJV 036 13	26	6607	0.7	12.90		135	16.5						
drain 14	144	6751	0.7	13.[illegible]9									
RJV 037 15	17	6768	0.5	13.22	rust flocs	146	25.4						
drain 16	144	6912	0.7	13.50									
RJV 038 17	16	6928	0.5	13.53		144	22.3						
drain 18	144	7072	0.7	13.81									
RJV 039 19	24	7096	0.7	13.86		149	25.4						
drain 20	144	7240	0.7	14.14									
RJV 040 21	23	7263	0.7	14.19		152	25.2						
drain 22	144	7407	0.7	14.47									
RJV 041 23	21	7428	0.6	14.51		153	25.2						
drain 24	144	7572	0.7	14.79									
RJV 042 25	23	7595	0.7	14.83		154	25.2						
drain 26	144	7739	0.7	15.12									
RJV 043 27	24	7763	0.7	15.16		155	22.3						
drain 28	144	7907	0.7	15.44									
RJV 044 29	24	7931	0.7	15.49		149	19.0						
drain 30	144	8075	0.7	15.77									
RJV 045 31	15	8090	0.4	15.80	rust flocs	140	20.4						

EFFICIENCY LINE® 22-210

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MPERT01-09-10

Column Rejuvenation

outflow/tube	Vol (ml)	Cumul Vol (ml)	Rate (ml/min)	PV (1=512)	Color	La (mg/L)	Fe (mg/L)	8	9	10	11	12	13
drain	144	8234	0.7	16.08	clear								
RJV 046	17	8251	0.5	16.12		140	16.1						
drain	144	8395	0.7	16.40									
RJV 047	22	8417	0.6	16.44		151	18.9						
drain	144	8561	0.7	16.72									
RJV 048	24	8585	0.7	16.77		155	22.3						
drain	~~144~~ 144	8729	0.7	17.05									
RJV 049	~~144~~ 25	8754	0.7	17.10		154	28.2						
drain	144	8898	0.7	17.38									
RJV 050	23	8921	0.7	17.42		174 ~~210~~	38.0						
drain	144	9065	0.7	17.71									
RJV 051	21	9086	0.6	17.75		177	40.8						
drain	144	9230	0.7	18.03									
RJV 052	22	9252	0.6	18.07		178	46.5						
drain	144	9396	0.7	18.35									
RJV 053	23	9419	0.7	18.40		184	46.5						
drain	144	9563	0.7	18.68									
RJV 054	23	9586	0.7	18.72		183	42.8						
drain	144	9730	0.7	19.00									
RJV 055	25	9755	0.7	19.05		176	36.6						
drain	144	9899	0.7	19.33									
RJV 056	23	9922	0.7	19.38		175	28.9						
drain	144	10066	0.7	19.66									
RJV 057	17	10083	0.5	19.69		172	21.5						
drain	144	10227	0.7	19.97									
RJV 058	22	10249	0.6	20.02		173	20.5						
drain	144	10393	0.7	20.30									
RJV 059	22	10415	0.6	20.34		175	22.7						
drain	144	10559	0.7	20.62									
RJV 060	24	10583	0.7	20.67		186	32.0						
drain	144	10727	0.7	20.95									

EFFICIENCY LINE® 22-210

(5)

Column Rejuvenation

MPERT01-09-11

outflow/tube	Vol (mL)	Cum Vol (mL)	rate (mL/min)	PV (1=512)	Color	Ca (mg/L)	Fe (mg/L)	8	9	10	11	12	13
RJV 061 1	28	10755	0.8	21.01	clear	202	46.6						
drain 2	144	10899	0.7	21.29									
RJV 062 3	25	10924	0.7	21.34		202	56.6						
drain 4	144	11068	0.7	21.62									
RJV 063 5	24	11092	0.7	21.66		203	69.0						
drain 6	144	11236	0.7	21.95									
RJV 064 7	20	11256	0.6	21.98		204	73.2						
drain 8	144	11400	0.7	22.27									
RJV 065 9	23	11423	0.7	22.31		205	79.2						
drain 10	144	11567	0.7	22.59									
RJV 066 11	24	11591	0.7	22.64		204	73.6						
drain 12	144	11735	0.7	22.92									
RJV 067 13	25	11760	0.7	22.97		187	57.4						
drain 14	144	11904	0.7	23.25									
RJV 068 15	21	11925	0.6	23.29		184	42.4						
drain 16	144	12069	0.7	23.57									
RJV 069 17	23	12092	0.7	23.62		173	33.0						
drain 18	144	12236	0.7	23.90									
RJV 070 19	21	12257	0.6	23.94		174	24.6						
drain 20	144	12401	0.7	24.22									
RJV 071 21	23	12424	0.7	24.27		171	21.6						
drain 22	73	12497	0.7	24.41						only 105 min, Δ to EDTA, 10 mL			
drain 23	180	12677	10.0	24.76		~~172 5pm~~	~~25.4 5pm~~			max 18°	fileTime		
RJV 072 24	20	12697	10.0	24.80		172	25.4			2"	25,333+	@08:39	
drain 25	180	12877	10.0	25.15									
RJV 073 26	19	12896	9.5	25.19		197	29.1				25,667+	@08:59	
drain 27	180	13076	10.0	25.54									
RJV 074 28	19	13095	9.5	25.58	purpled green	2000	1710			Clump white ppt @ bottom			
drain 29	180	13275	10.0	25.93	brown								
RJV 075 30	20	13295	10.0	25.97	orange (top)	1450	2520			clump	26.333+	@ 9:59	
drain 31	180	13475	10.0	26.32									

Column Rejuvenation Rate

MPERT01-09-12

outflow/tube		Vol (mL)	Cum Vol (mL)	Time (min)	PV (1=512)	Color	Ca (mg/L)	Fe (mg/L)	8	9	10	11	12	13
RJV 076	1	20	13495	10.0	26.36	Beyond	1290	2310		clump white ppt.				
drain	2	180	13675	10.0	26.71									
RJV 077	3	20	13695	10.0	26.75		1000	2260		Clump white ppt				
drain	4	180	13875	10.0	27.10									
RJV 078	5	19	13894	9.5	27.14		890	2380		Clump white ppt				
drain	6	180	14074	10.0	27.49									
RJV 079	7	19	14093	9.5	27.53	cloudy orange	865	1940		@1059 – 27.667 + filltime ?/c acid in tubes				
drain	8	180	14273	10.0	27.88	rust ppt				Change ORP outlet probe cloudy orange				
RJV 080	9	20	14293	10.0	27.92		835	2270						
drain	10	180	14473	10.0	28.27									
RJV 081	11	19	14492	9.5	28.30		645	1950		@1139 – 28.333				
drain	12	180	14672	10.0	28.66					Change back to R1M3 @ 6" left on drain tube @28.58				
RJV 082	13	19	14691	9.5	28.69		640	1930						
drain	14	180	14871	10.0	29.04									
RJV 083	15	20	14891	10.0	29.08		545	1620						
drain	16	180	15071	10.0	29.44									
RJV 084	17	19	15090	9.5	29.47		470	1160						
drain	18	180	15270	10.0	29.82									
RJV 085	19	19	15289	9.5	29.86		446	960						
drain	20	180	15469	10.0	30.21									
RJV 086	21	20	15489	10.0	30.25		306	575						
drain	22	180	15669	10.0	30.60									
RJV 087	23	19	15688	9.5	30.64	clearing	168	315						
drain	24	180	15868	10.0	30.99	pale orange								
RJV 088	25	19	15887	9.5	31.03		91	128						
drain	26	180	16067	10.0	31.38									
RJV 089	27	16	16083	8.0	31.41		645	68						
drain	28	180	16263	10.0	31.76									
RJV 090	29	19	16282	9.5	31.80		55.9	35.5						
drain	30	180	16462	10.0	32.15									
RJV 091	31	19	16481	9.5	32.19	clear	63.5	22.8						

MPERT01-09-13

Column Rejuvenation

outflow/tube	#	Vol (mL)	Cum Vol (mL)	Rate (mL/min)	PV (1=512)	Color	Ca (mg/L)	Fe (mg/L)	8	9	10	11	12	13
drain	1	180	16661	10.0	32.54									
RJV 092	2	19	16688	9.5	32.59	clear	73.5	16.9		last collect @ ↑ flow				
drain	3	144	16824	0.7	32.86				filtrate =	Δ collect time	205 drain	/35 collect		↓ flow 0.7
RJV 093	4	23	16847	0.7	32.90		44	27.4	32,000	resume acid	addition			mL/min
drain	5	144	16991	0.7	33.19					0.2 mL HNO_3	in each tube			
RJV 094	6	23	17014	0.7	33.23		29.2	27.8						
drain	7	144	17158	0.7	33.51									
RJV 095	8	23	17181	0.7	33.56		29.3	25.9						
drain	9	144	17,325	0.7	33.84									
RJV 096	10	25	17350	0.7	33.89		39.7	31.6						
drain	11	144	17494	0.7	34.17									
RJV 097	12	23	17517	0.7	34.21		46.2	26.6						
drain	13	144	17661	0.7	34.49		53.5	33.3						
RJV 098	14	25	17686	0.7	34.54		55,750	5/12	stop @ 1502,	start 5/14 @ 1502		~~MAY 15~~	MAY14_0	
drain	15	144	17830	0.7	34.82									
RJV 099	16	19	17849	0.5	34.86		49.0	21.0						
drain	17	144	17993	0.7	35.14									
RJV 100	18	21	18014	0.6	35.18		51.0	13.9						
drain	19	144	18158	0.6	35.46									
RJV 101	20	18	18176	0.5	35.50		55.5	12.4						
drain	21	144	18320	0.7	35.78									
RJV 102	22	22	18342	0.6	35.82		64.0	7.4						
drain	23	144	18486	0.7	36.11									
RJV 103	24	25	18511	0.7	36.15		66.5	6.5						
drain	25	144	18655	0.7	36.44									
RJV 104	26	26	18681	0.7	36.49		78.0	3.95						
drain	27	144	18825	0.7	36.77									
RJV 105	28	25	18850	0.7	36.82		89.0	3.80						
drain	29	144	18994	0.7	37.10									
RJV 106	30	25	19019	0.7	37.15		94.0	4.50						
drain	31	144	19163	0.7	37.43									

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MPERT01-09-14

Column Rejuvenation Rate

aulfeed/tube	Vol (ml)	Cum vol(ml)	(ml/min)	PV (1=512)	Color	Ca (mg/L)	Fe (mg/L)						
RJV 107	15	19178	0.4	37.46	clear	98.5	4.75						
drain	82	19260	0.4	37.62									
RJV 108	15	19275	0.4	37.65		102.0	3.40						
drain	82	193[illegible]	0.4	37.81									
RJV 109	28	19385	0.8	37.86	rust ppt.	127.5	33.5						
drain	164	19549	0.8	38.18									
RJV 110	16	19565	0.4	38.21		110.0	4.45						
drain	82	19647	0.4	38.37									
RJV 111	3	19650	0.09	38.38		110.0	5.93		shut down - Column tubing plugged.				
drain	18	19668	0.09	38.41									
RJV 112	3	19671	0.09	38.42									
drain	18	19689	0.09	38.46									
RJV 113	2	19691	0.06	38.46									
drain	12	19703	0.06	38.48									
RJV 114	1	19704	0.03	38.48									
drain	6	19710	0.03	38.50									
RJV 115	1	19711	0.03										
drain	6	19717	0.03	38.51									
RJV 116	1	19718	0.03										
drain	6	19724	0.03	38.52									
RJV 117	1	19725	0.03										
drain	6	19731	0.03	38.53									
RJV 118	1	19732	0.03										
drain	6	19738	0.03	38.54									
RJV 119	1	19739	0.03										
drain	6	19745	0.03	38.56									
RJV 120	<1		Ø										
drain	—												
RJV 121	<1												
drain	—												
RJV 122	<1												

Rejuvenation Batch Tests

MPERT 01-10-01

4/2/03 5 — OSL — 19:41 Made 500 mL of NaOAc Buffer (Jackson recipe)
41 g NaOAc and 13.5 mL glacial Ac. Acid in 500 mL

20:00 pH of NaOAc Buffer = 4.95 (should be 5 - close enough)

Testing to see if NaOAc Buffer removes calcite from ZVI w/o removing Fe

	Tube #	Gran ZVI (g)	Calcite Powder (g)	NaOAc Buffer	Start Time		Stop Time		Time Shake (HR)	Ca mg/L	Fe mg/L	Final pH
RJV-B1	1	0	50 mg	50 mL	2016 4/2/03		4/3/03	10:22	~14	352*	1.01	5.01
RJV-B2	2	2.0	0	↓	↓		4/3/03	10:22	~14	102	2650	5.10
RJV-B3	3	2.0	0				4/3/03	16:54	~20	92	3270	5.14
RJV-B4	4	2.0	0				4/4/03	11:38	~39	76	6020	5.36

*Dissolved 17 mg (~35%) [illegible]

4/3/03 5 — Tubes # 2,3,4 (those w/ gran ZVI) had pressured up and leaked slightly
Removed # 1 and 2 Centrifuged @ 3820 rpm for 22 min. Decanted ~15 mL and Added 100 µL conc HNO_3
Appears to be a small amount of powder in Tube 1 (why didn't it pressure up?)
Must be H_2 that is causing P buildup.

pH of Conc. Glacial Acetic Acid ≈ 0.25. Does not fizz when powdered calcite added

16:54 Tubes 3 and 4 pressured up and leaked - released pressure
Centrifuged tube 3 20 min @ 3820 rpm. Preserved (after decanting) with 100 µl HNO_3

1731 Made EDTA Solution 100 mg EDTA in 50 mL DI (~0.005M). Looks like
RJV-B5 it dissolved immediately. pH = 10.57 Added 100 mg powdered $CaCO_3$.
Placed on stir bar 1737. Cloudy (i.e. $CaCO_3$ has not dissolved)

4/4/03 5 — 1138 Tube #4 had pressured up and leaked again
Centrifuged Tube #4, EDTA + $CaCO_3$, and HOAc + Calcite 20 min @ 3820 RPM

RJV-B5 EDTA + Calcite: There is undissolved powder in the EDTA [illegible] Ca = 175 mg/L Fe = 0.4[illegible]

RJV-B6 HOAc + Calcite: " " " " " " HOAc pH = 0.28 Ca = 50 mg/L Fe = 1.01

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MPERT 01-10-02

4/5/03 0701 Tubes 2, 3, 4 (w/ grav ZVI), after centrifugation have turned red

RJV-B1 * NaOAc Buffer (50 mL) had 352 mg/L Ca

$0.050\,L \times \frac{352\,mg}{L} = 17.6\,mg$

$17.6\,mg\,Ca \times \frac{100\,mg\,CaCO_3}{40\,mg\,Ca} = 44\,mg\,CaCO_3 \quad \left(\frac{44}{50} = \underline{88\%}\ \text{dissolved}\right)$

RJV-B5 EDTA (50 mL) had 175 mg/L Ca

$0.050\,L \times \frac{175\,mg}{L} = 8.75\,mg\,Ca$

$8.75\,mg\,Ca \times \frac{100\,mg\,CaCO_3}{40\,mg\,Ca} = 21.9\,mg\,CaCO_3 \quad \left(\frac{21.9}{100} = \underline{21.9\%}\ \text{Dissolved}\right)$

4/7/03 0645 Made Solution of EDTA, tetrasodium salt Baker I693-7

FW = 416.2 $(NaOCOCH_2)_2NCH_2CH_2N(CH_2COONa)_2 \cdot 2H_2O$

0.25 M = 0.25 × 416.2 = 104 g/L

0.10 M = 41.62 g/L

MADE 1 L of 0.10 M. Solution has yellow cast. pH = 10.85

	0.1M EDTA	gr Au/ZVI	START* Time 4/7/03	START** pH	Conc HCl added (mL)	END Time 4/9/03	FINAL pH	Ca (mg/L)	Fe (mg/L)	[illegible]
RJV B7	50 mL	2g	0753	10.85	0	1640	11.02	145	84	9
RJV B8	50 mL	2g	0753	9.05	270	"	9.70	242	940	"
RJV B9	50 mL	2g	0753	6.88	550	"	8.25	142	1165	"

* END over END

** Solution – no grav/ZVI

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MPERT 01-10-03

5/7/03 1640 Removed RJV B7 - B9 from shaker. Centrifuged 20 m at 3780 RPM
No pressuring (ie no leakage, no swelling seps.)

5/8/03 0725 Prepared new leach tests:

	0.1M EDTA	Material (1g each)	Start Time 5/8/03	Stop Time 5/8/03	Shake Time (hr)	Final pH	Ca (mg/L)	Fe (mg/L)
RJV B10	50 mL	$CaCO_3$ Powder	0742	01516	7.5	11.11	5820	0.36
RJV B11	↓	ZVI -6 +10 (Mont)	↓	↓	↓	11.13	1.67	80
RJV B12	↓	~~Fe_2O_3~~ Hematite Fisher	↓	↓	↓	10.76	2.38	1.21
RJV B13	50 mL	Magnetite ACE	↓	↓	↓	10.85	15.4	4.88

ALL tubes had residual solids after treatment.
1516 D/C samples from shaker. Centrifuged 3780 RPM 22 mins

0.1 M NaOAc FW = 82.03 (ALDRICH 241245) 8.2 g/L made 1L. pH = 7.32

5/9/03 Analyzed Ca and Fe:

5/10/03 0735 Made new batches with EDTA (different concentrations) + 1g Calcite powder + 1g ZVI -6 +10 (Mont). End over end

	50 mL	1g Calcite	1g ZVI	Start Time 5/10/03 0800	Stop Time 5/11/03 1215	Shake Time (HR)	Final pH	Ca (mg/L)	Fe (mg/L)
RJV B14	0.1 M EDTA	↓	↓			28	11.11	3430	5.20
RJV B15	0.05 M EDTA	↓	↓	↓	↓	↓	11.06	1970	4.10
RJV B16	0.025 M EDTA	↓	↓	↓	↓	↓	10.90	930	2.60
RJV B17	0.1 NaOAc*	↓	↓	↓	↓	↓	9.64	10.4	0.69

Ret

* pH 7.32 (no HOAc)

5/11/03 1215 D/C shaker. Centrifuged 40 min @ 3780 RPM

9/30/03 Set up. Precision Model 25 Temperature controlled shaker bath. Using 50 mL – glass erlenmeyer flasks. Bath will hold up to 16 flasks. Powdered NOAH AFO (DRIED) in mortar/pestle. This (DRIED NOAH AFO) WAS made 9/18/00 BY DRYING NOAH AFO slurry on bread pans. made solution of 0.2 M Ammonium oxalate and 0.2 M oxalic acid. Mixed these together (see ESL Proc.) to yield pH 3 (400 mL Am-ox + 300 mL Ox Acid). Call this Amm. OxALate-pH3. Set temperature bath to 25°C (distilled H_2O – SAYS not to use DEIONIZED H_2O). EDTA (0.1 M) Solution was made 5/7/03 from Baker EDTA Tetrasodium Salt Dihydrate. pH = 10.71; cond = 18,470 µS/cm

Amm Oxalate-pH3 cond = 20,300 µS/cm

Flask	Sample No. (40mL)	Liquid (40 mL)	Solid (0.5 g)	pH initial	pH final	Cond initial (µS/cm)	Cond final (µS/cm)	Ca (mg/L)	Fe (mg/L)	Eff. Color	Shake Time	Te
1	RE-1	0.1 M ~~EDTA~~ Na Dithionite	NOAH AFO Powder	~~10.74~~ 4.53	5.85	~~18,470~~ 15,650	16,130	21.0	434	clear	2 hr	[illegible]
2	2	↓	Hematite Powder	↓	4.83	↓	15,960	0.40	260	lgt susp. pink	↓	↓
3	3	↓	MAGNETITE powder	↓	4.11	↓	15,960	2.12	204	Clear	↓	↓
4	4	↓	Fisher 40 mesh ZVI	↓	3.75	↓	15,910	0.26	140	Clear	↓	↓
5	5	↓	CALCITE Powder	↓	4.22	↓	16,070	170	0.50	Clear	↓	↓
6	6	*0.2 M Amm. Oxalate -pH3 (in dark)	NOAH AFO Powder	3	3.18	20,300	20,100	<0.1	394	green/yel.	↓	↓
7	7	↓	Hematite Powder	↓	3.05	↓	20,100	0.31	~~<0.1~~ 19.1	pink	↓	↓
8	8	↓	magnetite powder	↓	3.13	↓	20,100	0.45	~~19.4~~ 344	lgt. green	↓	↓
9	9	↓	Fisher 40 mesh ZVI	↓	3.12	↓	20,200	0.18	98	"	↓	↓
10	10	↓	Calcite Powder	↓	3.14	↓	20,200	0.50	0.48	clear	↓	↓

STARTED SHAKE 10/1/03 @ 0644 — * Shaker bath keeps them in the dark

Stopped 10/1/03 @ 0844 — Shaken at 60 RPM

Solid MATERIALS

① NOAH AFO Powder – see above

② Hematite Powder – Fisher Fe_2O_3 Ferric Oxide Red-Anhydrous I116-3, XRD confirmed hematite.

③ MAGNETITE Powder – ACF A-310, magnetic ferric oxide, Black; XRD confirmed as magnetite

④ Fisher 40 mesh ZVI – Fisher Iron metal, Filings about 40 mesh, I57-500

⑤ CALCITE powder – Aldrich 23,921-6 $CaCO_3$; confirmed as calcite by XRD

Made 0.1 M Sodium Dithionite Cond = 15,650 µS/cm, pH = 4.53

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MPERT 01-10-05 P.2

10/1/03												
0844	Stopped shaker bath. Decant into 50mL plastic centrifuge tubes.											
	Centrifuged 20m @ 3500 RPM											
	Analyzed for Ca and Fe by AA											

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Rejuvenation Tests

MPERT 01-10-06

10/2/03

0813 Prepared 0.3 M Na-citrate (pH = 8.40); 1 M $NaHCO_3$ (pH = 7.95)

Mixture of 200 mL 0.3 M Na-citrate + [illegible] M $NaHCO_3$ (pH = [illegible])

Mixture of 200 mL 0.3 M Na-citrate + 25 mL 1 M $NaHCO_3$ + 3.917 g Na Dithionite (pH = 6.98)

Fizzes when dithionite is added (0.1 M [illegible])

Flask	Sample No.	Liquid (40 mL)	Ratio	Solid*	pH^i	pH^f	Ca (mg/L)	[illegible] (mg/L)	Shake Time (hr)	Controlled Temp.
1	RE-11	0.3 M Na-citrate + 1 M $NaHCO_3$	(40 mL) (5 mL)	NOAA AFO [illegible]	[illegible]	8.49	63.0	[illegible]	[illegible]	25°C
2	-12			Hematite Powder		8.41	2.08	0.58		
3	-13			Magn[illegible]		8.44	16.4	1.45		
4	-14			Fisher 40 mesh ZVI		8.57	1.44	68		
5	-15			Cal[illegible]		8.68	246.5	0.15		
6	-16	Same as RE-11 - 15 But + 3.917 g Na Dithionite		NOAA AFO Powder	6.98	7.44	62.8	1050		
7	-17			Hematite Powder		7.19	2.41	405		
8	-18			Magnetite Powder		7.08	13.0	235		
9	-19			Fisher 40 mesh ZVI		7.08	1.49	150		
10	-20			Calcite Powder		7.13	209	0.40		

* See previous day chart for product designations

0841 Start shaking in T-controlled bath 60 RPM

1021 Stopped shaker. Decanted into 50 mL plastic centrifuge tubes. Centrifuged 20 min @ 350 RPM

RE-16 AFO w/ dithionite turned brown (Fe being reduced); however, hematite RE-2 does not appear to have been reduced.

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MPERT 01-10-07

10/3/03

Made 0.05 M EDTA Tetrasodium salt solution (20.81 g/L) pH = 10.92

Made mixture of 0.05M EDTA 50% and 0.1 M Na-Dithion. 50% pH = 6.97

Flask	Sample No.	Liquid (40mL)	Solids*	pH	[illegible]	Ca mg/L	Fe mg/L	Shake T (hr)	Controlled Temp
1	RE-21	0.05M EDTA (Tetra)	NOAH AFO Powder	10.93	10.61	52.5	11.2	2	25°C
2	-22		Hematite Powder		10.79	1.88	0.66		
3	-23		Magnetite Powder		10.82	7.9	1.22		
4	-24		Fisher 40 ZVI		[illegible]	1.45	24.0		
5	-25		Calcite Powder		10.92	288	0.14		
6	-26	200 mL 0.05 EDTA + 65mL 0.1 M Na-DTH	NOAH AFO Powder	9.17	9.30	43	12.5		
7	-27		Hematite Powder		9.25	1.76	0.64		
8	-28		magnetite Powder		9.23	5.8	1.45		
9	-29		Fisher 40 ZVI		9.37	1.16	107.5		
10	-30		Calcite Powder		9.36	226	<0.1		

Started shaking (60 RPM) 0704 10/6/03. Stopped 0904 (2 Hr). Decanted Centri. @ 3500 RPM, 20 min. Decanted

10/03/03 0839 Titration of 0.1 M EDTA (Tetra) w/ 0.1 M Na Dithionite (Start w/ 200 mL of EDTA)

Added	DTH Na DTH (mL)	pH	Add.	Cum	pH	Add	cum	pH
0	0	10.74	5	20	10.13	10	90	9.36
1	1	10.70	10	30	9.97	10	100	9.28
2	3	10.60	10	40	9.84	10	110	9.20
2	5	10.53	10	50	9.73	20	130	9.02
5	10	10.36	10	60	9.63	20	150	8.83
5	15	10.24	10	70	9.54	20	170	8.59
			10	80	9.45	20	190	8.26
						10	200	8.07

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MPERT - 01 - 10 - 08

10/6/03 Made 200 mL 0.05 EDTA (Tetra-Na) + 65 mL 0.1 M Na-Dithionite (pH = 9.17)
This solution is based on the titration (see MPERT-01-10-07) to achieve
Approx. pH 9. The thought is that since EDTA (pH 10.93) is effective at
removing Ca but also removes some Fe, perhaps by adding a reductant
(Na-Dith) the proportion of Fe corrosion would be reduced.

10/7/03 Made solutions for next tests:
Soln 10-7-1 1M Hydroxylamine Hydrochloride in 25% Acetic acid (see Landa 1982)
pH = 1.48
Soln 10-7-2 200 mL 0.3 M Na Citrate, 25 mL 1M K_2CO_3, 3.917 g Na-Dithionite
pH (before Na Dith) = [illegible]
pH (After Na Dith) = 9.70

Start shaker @ 15:35 10/7/03
Stop shaker @ 17:35 10/7/03

Flask	Sample No	Liquid (40 mL)	Solid*	pH i	pH f	Ca (mg/L)	Fe (mg/L)	Shake Time	controlled T °C
1	RE-31	200 mL 0.3 M Na Citrate 25 mL 1M K_2CO_3 3.917 g Na-Dithionite	NOAH AFO Powder	9.70	8.58	36.6	378	2 hr	25
2	-32	↓	Hematite Powder	↓	8.56	1.46	69	↓	↓
3	-33	↓	Magnetite Powder	↓	8.63	4.38	75	↓	↓
4	-34	↓	Fisher 40 mesh ZVI	↓	[illegible]	0.85	100	↓	↓
5	-35	↓	Calcite Powder	↓	8.44	23.4	0.75	↓	↓
6	-36	1M Hydroxylamine hydrochloride in 25% Acetic Acid	NOAH AFO Powder	1.48	1.52	46.8	185	↓	↓
7	-37	↓	Hematite Powder	↓	1.32	0.96	2.51	↓	↓
8	-38	↓	Magnetite Powder	↓	1.33	3.37	13.3	↓	↓
9	-39	↓	Fisher 40 mesh ZVI	↓	2.20	0.33	2160	↓	↓
10	-40	↓	Calcite Powder	↓	2.67	5620	1.33	↓	↓

← Foamed - nearly overflowed

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MPERT-01-10-09

10-9-03

Results to date suggest that EDTA is good at removing calcite but not good at removing Fe-oxide corrosion products. Na-Citrate + Dithionite appears to be good at removing Fe oxides. High pH appears to minimize ZVI removal.

Therefore we will try mixing Na-Citrate EDTA and Dithionite.

10:03 Start with 200 mL EDTA (1M), pH = [illegible].85
Added 4g Na-Dith, pH = 9.13
Added 100 mL EDTA (1M), pH = [illegible]
Added 100 mL EDTA (0.1m), pH = 9.58
Added 1g EDTA, pH = [illegible]
Added 9g EDTA, pH = 9.69

Solution Composition:

400 mL EDTA, 41.62g/L = 16.648g
+10g = 26.648g EDTA

$\frac{26.648g}{.4L} = 66.62g/L \times \frac{mol}{416.2g} = 0.16m$ EDTA

$\frac{4g\ Dith}{.4L} \times \frac{mol}{174.1g} = 0.057M$ Dith

FLASK	Sample No	Liquid (40 mL)	Solid*	pH^i	pH^f	Ca mg/L	Fe mg/L	Shake Time	Controlled T.°C
1	RE-41	0.1M EDTA (Tetra Na)	NOAH AFO Powder	10.95	10.66	59.5	10.8	2 hr	25
2	-42	↓	Hematite Powder	↓	10.80	2.15	0.53	↓	↓
3	-43	↓	Magnetite Powder	↓	10.85	9.0	1.76	↓	↓
4	-44	↓	Fisher 40 mesh ZVI	↓	10.97	1.6	26.6	↓	↓
5	-45	↓	Calcite Powder	↓	10.88	253	0.34	↓	↓
6	-46	0.16M EDTA (Tet. Na) 0.057M Dith.	NOAH AFO Powder	9.69	9.21	37	167	↓	↓
7	-47	↓	Hematite Powder	↓	9.19	2.04	121	↓	↓
8	-48	↓	Magnetite Powder	↓	9.16	8.7	118	↓	↓
9	-49	↓	Fisher 40 mesh ZVI	↓	9.16	1.9	106	↓	↓
10	-50	↓	Calcite Powder	↓	9.20	182	0.53	↓	↓

Started shaker 12:41 10/9/03
Stopped " 14:41 10/9/03

5-13-04 Rejuvenation Column Test (#2) MPERT01-12-01

0700 Starting set up for a rejuvenation column test.

1310 Calibration of electrodes Logger Pro file = MAY13_04 (Folder = Rejuvenation Column 2)

Time #	TYPE	MANF	ESL #	Intercept	Slope	CALIB STDS	pH 4 Check (STAGNANT)	pH 7 Check (STAGNANT)	pH 10 Check (STAGNANT)	Zobell ORP mV	
13:16	pH gel	Orion	F9107	13.9249	-3.8460	4,7	3.95	7.00	10.14	–	* As per Vernier
"	pH gel	Orion	E9107	12.9397	-3.7980	4,7	4.08	6.99	10.04	–	
15:36	ORP	Cole-P	A05990-55	-389.76*	222.6*	–	–	–	–	194	
"	ORP		C45900-ORP	-389.76*	222.6*	–	–	–	–	189	

5/24/04 Pack a 2" diameter column with fresh ZVI/gravel mix like that used in the ZVI/gravel zone @ PeRT well in Monticello. It is a mix of 2500 lbs -4/+20 ZVI to 9 yd^3 gravel
Net weight of fill = 1537.6 g

5/25/04 Prepare 20L RIM3 synthetic water using the following

50 g/L	$NaHCO_3$	220 mL
1.5 g/L	$CaSO_4 \cdot 2H_2O$	10300 mL
200 g/L	$MgSO_4 \cdot 7H_2O$	54 mL
92.72 g/L	$CaCl_2 \cdot 2H_2O$	5 mL
100 g/L	K_2SO_4	34 mL
Milli-Q H_2O		9387 mL
		20,000 mL

Note: Error in making solution. Switched amts of 92.72 g/L $CaCl_2 \cdot 2H_2O$ with 100 g/L K_2SO_4. Result was ↓ amt Cl in solution and ↑ K in soln. See pg 8 for notes

0945 Start air (via aquarium pump/stone) and CO_2 (via tank/aquarium stone). CO_2 @ 10 cc/min. initial pH = 8.63. Start mag stir bar.

1000 fill inlet tubing.

1010 Start flow to column @ 0.7 mL/min using size 16 masterflex pump head, start logging point 1

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5/25/04 file name = May25_04a. Collect 12 points/hr. Set collect time @ 336 hrs (2 weeks)

pH and ORP probes checked prior to start.

	intercept	slope	pH 7	pH 10	zobell
pH 1	13.9249	-3.8460	7.07	10.13	
pH 2	12.9397	-3.7984	7.06	10.10	
ORP 1	-389.76	222.6			213
ORP 2	-389.76	222.6			189

1045 note pH checked in source tank = 7, pH @ pH1 = 8.60 (line 9). will monitor - pH should drop when CO_2 entrained H_2O fills tubing and reaches pH 1 probe

1420 pH is gradually dropping line 52 7.68 Columns ~ ½ full

1540 ↑ CO_2 flow to 20 ml/min pH is still ↑ (line 68) 7.66

1800 Program Gilson 206 fraction collector #518618, file #2. Drain × 205", Collect × 35".
Will collect 1st outflow in cent. tube then start frac. collector and use the 205" drain time to fill outflow tubing. (Drain is 1st cycle in program)

1807 line 97 pH still ↑ @ 7.60. ↑ CO_2 flow to 30 ml/min

1928 first outflow line 113 pH = 7.57 ORP = 159.13

9 hrs 18 minutes to fill = 558 minutes × 0.7 ml/min = 390.6 ml = 1 PV
Measure 35" = 24.5 ml = 0.7 ml/min

2003 Start fraction collector line 120 pH = 7.53 ORP 159.68

5/26/04 0710 Arrive to find leak @ connector to fraction collector. Water on counter. Tighten connection and ck all others. No samples (12) collected over night. at ~0730 3rd collection started. Not enough water in tubing to make sample #3.

Moved tubing around lines 256, 257. Leak was @ bad coupling to frac. collector. Replace. No leaks @ present

1100 Seems to be running well. Add 200 µl conc HNO_3 to each collection tube

1252 ↓ CO_2 flow to 10 ml/min pH = 7.09 line 322. Changed CO_2 diffuser (with the air diffuser) @ ~1030. Flat diffuser seems to work better.

1615 All appears to be running well line 363

	pH	ORP
out	7.52	-148.47
in	6.69	153.43

5/27/04 0930. Column appears to be running well. CO_2 flow @ 10 ml/min. line 549 parameters

	pH	ORP
in	6.69	163.48
out	7.32	-212.59

Transfer collected samples to cent. tubes to prevent evaporation.

Column has run well all day. Samples 1-10 + source analyzed for Ca, Fe

5/28/04 0755 Column appears to be running well CO_2 flow @ 10 ml/min line 839 parameters

	pH	ORP
in	6.77	156.14
out	7.22	-328.08

rust colored staining @ bottom of column
outflow is pale orange

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5/31/04 ~2100 Check column. Appears to be running well with one unexplained occurrence. Tube 17 was full to overflowing. ~~other~~ Subsequent tubes are filled appropriately. Clean ~10 ml from frac collector tray.

line 1864 parameters 155,250 hrs.

	pH	ORP
in	6.37	192.83
out	7.22	-389.76

↓ CO_2 flow slightly to ~8 ml/min.

Source tank contains ~13L

6/1/04 0730 Column running well. Discovered explanation of ? vol in tube 17. Frac collector malfunction. Tube 17 was filled to overflow with collection #37. Arrival last night to tend column was in between collects. Tubes were emptied, tube for next collection in supposed correct slot. Tubes 38, 39 in wrong position, lost to secondary containment. Tube 40 back on track. Will monitor throughout day.

line 1990 parameters

	pH	ORP
in	6.38	192.83
out	7.33	-389.76

↓ CO_2 flow to ~5 ml/min.

Rusted area at bottom of column has intensified in color, but not increased in area or changed shape.

Col. running well line 2034 parameters

	pH	ORP
in	6.38	190.92
out	7.34	-389.76

DC CO_2 flow

6/2/2004 0715 column running well pH of source tank has ↑ since D/C of CO_2 flow 6/1/04
Will restart CO_2 flow @ 10ml/min

line 2275 parameters @ 189.500 hrs

	pH	ORP
in	7.03	178.42
out	7.32	-389.76

0930 Col. running well line 2297 parameters @ 191.33 hrs

	pH	ORP
in	7.09	175.16
out	7.32	-389.76

1018 stop frac collector c̄ 69 min left of 205" drain =95 ml out during 136" of drain
Stop pump

1029 Restart pump @ 10 ml/min c̄ 0.1M EDTA (disodium) 37.224 g/L
restart frac collector @ 18" drain, 2 min collect
tube 47 will be first split out after start of EDTA

1340 Two L Disodium EDTA complete @ 6.24" left on 18" drain cycle
Tubes 47-55 collected during this time

line 2348 parameters @ 195.583 hrs

	pH	ORP
in	~~5.16~~	209.40
out	5.17	-232.43

12
205

1347 Reprogram frac collector 205" drain, 35" collect
restart pump c̄ RIM3 @ 0.7 ml/min

Took 191 minutes to pump 2000 ml EDTA soln, rate = 10.47 ml/min. Will use this instead of calc rate from tubes to estimate vol rate of outflow to drain
large amts of gas generated in column during EDTA pumping

6/2/2004 1347 cont. Crystals formed in bottom of test tubes in tubes 50→55, white, angular

1715 line 2390 parameters pH ORP time 199.083 start of tube 56

	pH	ORP
in	4.96	200.16
out	6.31	-276.72

Elevate outlet end of probe holders to allow EDTA to completely flush
will leave CO_2 flow @ ~10mL/min Tube 56 will probably be short.

1805 Col running well. inlet pH is still low. will make any changes in Am.

6/3/04 0730 Col. running well pH has gradually ↑ on influent
line 2563 parameters @ 213.500 hrs

	pH	ORP
in	6.29	155.06
out	7.55	-297.92

~10L in source tank, CO_2 @ 10mL/min

"rusted" area @ bottom of column (opposite inlet) has lightened significantly
sample tubes are layered in color. When transferred to cent. tubes, layering ceases.

6/4/04 0730 Col. running well pH grad ↑ on influent
CO_2 @ 10mL/min
line 2856 parameters @ 237.917

	pH	ORP
in	6.47	171.09
out	7.19	-322.01

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6/4/04 DC flow to column for probe check. Data logger continues to run.
Remove probes and place in cal solns.

0949 @ line 2878 time 239.750 | pH | ORP | end of tube 66 collect.

		pH	ORP
	in	6.48	178.36
	out	7.21	-370.74

D/C flow is @ end of tube 66 collect.

place probes in standards @ line 2879 (pH1, ORP1)

first data on all 4 @ line 2880 @ 239.917 time

	Std 7	Std 4		3 bel
pH1	7.01	4.07	ORP1	197.72
pH2	7.02	4.08	ORP2	183.04

switch buffer solns @ line 2882

1024 restart flow to column pump off 35" will adjust drain vol accordingly

6/7/04 0745 Column running well. CO_2 @ ~10ml/min

0800 @ line 3726 time 310.417

	pH	ORP
in	6.93	143.37
out	7.27	-389.76

0957 D/C flow to column, D/C data logger

line 3744 @ 311.917

	pH	ORP
in	6.94	148.10
out	7.26	-389.76

EFFICIENCY LINE® 22-210

6/14/2004 Discovered error in RIM3 solution preparation resulting in low Ca in source tank

Incorrect amounts of 92.72g/L $CaCl_2 \cdot 2H_2O$ and 100g/L K_2SO_4 were used. Amounts were switched in recipe:

1.7 mL/L (instead of 0.25mL/L) of 100g/L K_2SO_4 used resulting in

76.3 mg/L (instead of 11.22mg/L) K — 680% of desired K concentration from K_2SO_4
93.7 mg/L (instead of 13.78mg/L SO_4

0.25mL/L (instead of 1.7mL/L) of 92.72g/L $CaCl_2 \cdot 2H_2O$ used resulting in

6.3162 mg/L (instead of 42.95mg/L) Ca — 14.71% of desired Ca concentration from $CaCl_2 \cdot 2H_2O$
11.1898 mg/L (instead of 76.09 mg/L) Cl

	A	B	C	D	E	F	G	H	I	J	K
1	R1-M3, Samples 1/14/2003; for Rejuvenation Column Study initiated- 4/26/03										
2		mL per									
3		L of	Na	K	Ca	Mg	SO4	Cl	C	U	V
4		Soln	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
5	Liquid Components										
6	V2O5, V=998mg/L, 2%HNO3	0.00									0.00
7	V2O5, V=1000mg/L, 5%HNO3	0.00									0.00
8	V2O5, V=1000mg/L, 1.4%HNO3	0.00									0.00
9	V2O5, V=1000mg/L, 2%HNO3	0.00									0.00
10	Na2MoO4.2H2O, Mo=926mg/L	0.00	0.00								
11	Na2MoO4.2H2O, Mo=1000 mg/L,	0.00	0.00								
12	(NH4)2MoO4, 1006 mg/L Mo	0.00									
13	U3O8, U=10000mg/L, 3.5%HNO3	0.00								0.00	
14	U3O8, U=10000mg/L, 5.2%HNO3	0.00								0.00	
15	U Std, U=1000mg/L, 2% HNO3	0.00								0.00	
16	Ra Std, Ra=68.7pCi/mL, 2% HNO3	0.00									
17	As Std, As=999mg/L, 5%HNO3	0.00									
18	As Std, As=1000mg/L, 1.4%HNO3	0.00									
19	Se =999 mg/L 5% HNO3	0.00									
20	Cd Std ~ 1000mg/L, ~2% HNO3	0.00									
21											
22	100g/L K2CO3	0.00		0.00					0.00		
23	50g/L NaHCO3	11.00	150.60						78.57		
24	100g/L Na2SO4.10H2O	0.00	0.00				0.00				
25	100g/L Na2SO4.(0.00 H2O)	0.00	0.00				0.00				
26	500g/L (NH4)2SO4	0.00					0.00				
27	1.5g/L CaSO4.2H2O	515.00			179.83		430.98				
28	1.2 g/L CaSO4	0.00			0.00		0.00				
29	200g/L MgSO4.7H2O	2.70				53.28	210.48				
30	100g/L K2SO4	0.25		11.22			13.78				
31	conc H2SO4(36N)	0.00					0.00				
32	627.2 g/L CaCl2.2H2O	0.00			0.00			0.00			
33	82.72 g/L CaCl2.2H2O	1.70			42.95			76.09			
34	300g/L NaCl	0.00	0.00					0.00			
35	100g/L MgCl2.6H2O	0.00				0.00		0.00			
36	100g/L NH4Cl	0.00						0.00			
37	100g/L NaNO3	0.00	0.00								
38	2% HNO3	0.00									
39	mL 1N NaOH needed to neutralize	0.00	0.00								
40	66.67 g/L NaN3	0.00	0.00								
41											
42											
43											
44	Totals (mg/L)	530.65	150.60	11.22	222.88	53.28	655.21	76.09	78.67	0.00	0.00
45	ACTUAL (mg/L)		111.00	11.30	213.00	52.80	677.00	76.60	78.00	0.57	0.35
46	Totals (mol/L)		6.55E-03	2.87E-04	5.56E-03	2.19E-03	6.83E-03	2.15E-03	6.55E-03	0.00E+00	0.00E+00
47											
48	ACTUAL pH	6.85									
49	Measured pH(no acid or base)	?									
50	Measured pH (________ added)										
51	ACTUAL Alk (mg/L CaCO3)	317.00									
52	Measure Alk (no acid or base)	?									

AMPAD EFFICIENCY LINE® 22-210

MPERT01-12-09

outflow / tube	Vol (mL)	Cum Vol (mL)	Rate (mL/min)	PV 1=391	Color	Ca (mg/L)	Fe (mg/L)
1st outflow	24	24	0.7	0.06	orange	55.5	7.68
drain	0						
RC2-2	0						
drain	0	* filling tubing					
RC2-3	0						
drain	0						
RC2-4	12	36		0.09	clear	76.0	0.44
drain	144	180		0.46			
RC2-5	24	204		0.52		74.0	0.39
drain	144	348		0.89			
RC2-6	24	372		0.95		73.0	0.40
drain	144	516		1.32			
RC2-7	24	540		1.38		72.0	0.50
drain	144	684		1.75			
RC2-8	24	708		1.81		75.0	0.56
drain	144	852		2.18			
RC2-9	24	876		2.24		80.0	0.98
drain	144	1020		2.61			
RC2-10	24	1044		2.67		88.0	2.16
drain	144	1188		3.04			
RC2-11	24	1212		3.10		102.0	3.63
	144	1356		3.47			
RC2-12	24	1380		3.53		106.5	5.13
	144	1524		3.90			
RC2-13	24	1548		3.96		106.5	6.60
	144	1692		4.33			
RC2-14	24	1716		4.39		106.0	7.42
	144	1860		4.76			
RC2-15	24	1884		4.82		108.0	8.96
	144	2028		5.19			
RC2-16	24	2052		5.25		111.5	9.06

ST525 measured Ca source tank = 153 mg/L
calculated 223 mg/L
measured Fe source tank = 0 mg/L

* filling tubing. Small leak (<50mL)

AMPAD EFFICIENCY LINE® 22-210

MPERT01- 12-10

outflow/tube	#	Vol (ml)	Cum Vol (ml)	Rate (ml/min)	PV 1=391	Color	Cl (mg/L)	Fe (mg/L)	8	9	10	11	12	13
drain	1	144	2196	0.7	5.62									
RC2-17	2	29(24)	2220		5.68		121.0	13.45			* tube 17+37			
drain	3	144	2364		6.05									
RC2-18	4	24	2388		6.11		116.0	9.45						
	5	144	2532		6.48									
RC2-19	6	24	2556		6.54		114.0	9.65						
	7	144	2700		6.91									
RC2-20	8	24	2724		6.97		112.0	9.90						
	9	144	2868		7.34									
RC2-21	10	24	2892		7.40		111.0	10.30						
	11	144	3036		7.76									
RC2-22	12	24	3060		7.83		111.5	10.70						
	13	144	3204		8.19									
RC2-23	14	24	3228		8.26		116.0	12.10						
	15	144	3372		8.62									
RC2-24	16	24	3396		8.69		115.5	13.30						
	17	144	3540		9.05									
RC2-25	18	24	3564		9.12		117.5	13.95						
	19	144	3708		9.48									
RC2-26	20	24	3732		9.54		117.5	14.90						
	21	144	3876		9.91									
RC2-27	22	24	3900		9.97		117.0	16.70						
	23	144	4044		10.34									
RC2-28	24	24	4068		10.40		119.5	18.00						
	25	144	4212		10.77									
RC2-29	26	24	4236		10.83		121.0	18.90						
	27	144	4380		11.20									
RC2-30	28	24	4404		11.26		119.5	20.7						
	29	144	4548		11.63									
RC2-31	30	24	4572		11.69		118.5	21.1						
	31	144	4716		12.06									

EFFICIENCY LINE® 22-210

MPERT01-12-11

Outflow/tube		Vol (mL)	Cum Vol (L)	Rate (mL/min)	PV1=391	Color	Ca (mg/L)	Fe (mg/L)	8	9	10	11	12	13
RC2-32	1	24	4740	0.7	12.12		119.5	21.8						
	2	144	4884		12.49									
RC2-33	3	25	4909		12.55		122.0	24.4						
	4	144	5053		12.92									
RC2-34	5	24	5077		12.98		125.5	26.1						
	6	144	5221		13.35									
RC2-35	7	25	5246		13.42		125.5	25.7						
	8	144	5390		13.79									
RC2-36	9	27	5417	0.8	13.85		124.5	27.1						
	10	144	5561	0.7	14.22									
RC2-37	11	lost (24)	5585		14.28		—	—						
	12	144	5729		14.65									
RC2-38	13	lost (24)	5753		14.71		—	—						
	14	144	5897		15.08									
RC2-39	15	lost (24)	5921		15.14		—	—						
	16	144	6065		15.51									
RC2-40	17	24	6089		15.57		132.5	39.4						
	18	144	6233		15.94									
RC2-41	19	24	6257		16.00		137.5	42.0						
	20	144	6401		16.37									
RC2-42	21	26	6427		16.44		134.5	39.8						
	22	144	6571		16.81									
RC2-43	23	25	6596		16.87		132.0	36.4						
	24	144	6740		17.24									
RC2-44	25	25	6765		17.30		132.0	35.2						
	26	144	6909		17.67									
RC2-45	27	25	6934		17.73		132.5	34.6						
	28	144	7078		18.10									
RC2-46	29	25	7103		18.17		134.5	34.8						
	30	95/188	7386		18.89				switch to EDTA		@ 10 mL/min		18" drain/2" collect	
RC2-47	31	25	7411	10	18.95		153.0	42.8	clear					

AMPAD EFFICIENCY LINE® 22-210

MPERT01-12- 12

Outflow/tube		Vol (mL)	Cum Vol	Rate (mL/min)	PV1=391	5	Ca (mg/L)	Fe (mg/L)	Comments
drain	1	188	7599		19.43				
RC2-48	2	27	7626		19.50		345	945	yellow
	3	188	7814		19.98				
RC2-49	4	26	7840		20.05		1270	4300	yellow/green
	5	188	8028		20.53				
RC2-50	6	20	8048		20.58		1085	~~4300~~ 4410	yellow/green, lots of air bubbles ↓ vol, few white crystals
	7	188	8236		21.06				
RC2-51	8	18	8254		21.11		1130	~~3765~~ 4220	yellow/green
	9	188	8442		21.59				
RC2-52	10	16	8458		21.63		1030	~~4410~~ 3930	yellow/green
	11	188	8646		22.11				
RC2-53	12	28	8674		22.18		920	~~4220~~ 3820	yellow/green — significant amt (cover bottom of test tube) white crystals
	13	188	8862		22.66				
RC2-54	14	21	8883		22.72		810	3560	yellow/green
	15	188	9071		23.20				
RC2-55	16	28	9099		23.27		740	3080	yellow/green
	17	99/144	9342		23.89				outflow = 11" EOTA then 205" RIM3 switch to 205" drain/35" collects
RC2-56	18	10	9352		23.92		662	4730	yellow/green ē crystals (56)
	19	144	9496		24.29				
RC2-57	20	25	9521		24.35		454	3010	dark orange (57)
	21	144	9665		24.72				
RC2-58	22	25	9690		24.78		291	2260	layered yellow/black/yellow/orange (bottom) notes dark 5
	23	144	9834		25.15				
RC2-59	24	25	9859		25.21		224	1400	" " " " " notes dark 59
	25	144	10003		25.58				
RC2-60	26	25	10028		26.65		175.5	865	layered yellow/lt. black/yellow colors lightening
	27	144	10172		26.02				
RC2-61	28	25	10197		26.08		154	494	layered yellow to orange
	29	144	10341		26.45				
RC2-62	30	26	10367		26.51		144.5	302	layered yellow
	31	144	10511		26.88				

AMPAD EFFICIENCY LINE® 22-210

MPERT01- 12 - 13

outflow/tube		Vol (ml)	Cum Vol (ml)	Rate (ml/min)	PV 1=391	5	Ca (mg/L)	Fe (mg/L)	Comments	10	11	12	13
RC2-63	1	25	10536	0.7	26.95		146.5	188	yellow				
	2	144	10680		27.31								
RC2-64	3	25	10705		27.38		149.5	135.5	pale yellow				
	4	144	10849		27.75								
RC2-65	5	25	10874		27.81		156.5	99	Very pale yellow				
	6	144	11018		28.18								
RC2-66	7	26	11044				156	82.5	Very pale yellow				
	8	119	11163						D/c pump x ~~35~~" for make check				
RC2-67	9	25	11188				147	63	clear bottom pale yellow top				
	10	144	11332										
RC2-68	11	25	11357						Clear				
	12	144	11501										
RC2-69	13	20	11521										
	14	144	11665										
RC2-70	15	24	11689										
	16	144	11833										
RC2-71	17	25	11858										
	18	144	12002										
RC2-72	19	21	12023										
	20	144	12167										
RC2-73	21	24	12191										
	22	144	12335										
RC2-74	23	26	12361										
	24	144	12505										
RC2-75	25	25	12530										
	26	144	12674										
RC2-76	27	17	12691										
	28	144	12835										
RC2-77	29	24	12859										
	30	144	13003										
RC2-78	31	23	13026										

AMPAD EFFICIENCY LINE® 22-210

MPERT01-12-14

Outflow/tube#		1 Vol (mL)	2 Cum Vol (mL)	3 Rate (mL/min)	PV 1=351	5	6 Ca (mg/L)	Fe (mg/L)	8	9	10	11	12	13
drain	1	144	13170											
RC2-79	2	26	13196											
drain	3	144	13340											
RC2-80	4	26	13366											
drain	5	144	13510											
RC2-81	6	25	13535											
drain	7	144	13679											
RC2-82	8	23	13702											
drain	9	144	13846											
RC2-83	10	25	13871											
drain	11	144	14015											
RC2-84	12	24	14039											
	13	7	14046											
	14													
	15													
	16													
	17													
	18													
	19													
	20													
	21													
	22													
	23													
	24													
	25													
	26													
	27													
	28													
	29													
	30													
	31													

Col. Tests 3 & 4 EPA

MPERT01- 13-01

6/14/2004 Prepare 2 columns for testing using 15mm Omnifit columns. Each has frit @ inlet and none @ outlet

Col 3 = 41.33g net wgt. Mont PE-11-5. grab samples to fill column

Col 4 = 42.23g net wgt Mont PE 11-5 grab samples to fill column

Pump setup w/ size 13 head and tubing. Outflow to fraction collector.

Prepare 0.25L 0.1M EDTA, disodium salt dihydrate

Prepare 1L RIM3 synthetic water using freshly prepared standards

Prepare 2L RIM3 synthetic water spiked with 1mg/L U. 10,000 mg/L U Std (100ul) used for spike. As above, fresh standards used.

Measure parameters on all 3 solutions

	0.1 M EDTA	RIM3	RIM3 + 1mg/L U (1) (2L)	RIM3 + 1mg/L U (2) (2L)	RIM3 + 1mg/L U (3) (4L)	RIM3 + 1mg/L U (4) 4L
pH	5.07	7.86	7.87	7.91	8.04	7.97
ORP mV	168.2	123	126	127	133	129
Cond (uS/cm)	11390	1891	1896	1885	~~[illegible]~~ 1884	1905
alk (mg/L)	280	230	230	235	215	220
Ca (mg/L)	0.35	189.5	187.5	181.0	189.0	
U (mg/L)	<0.1	<0.1	941.7	993.5	967.2	1031.4

MPERT01- 13-02

6/15/2004 Start flow of 0.1M EDTA disodium salt to Col. 3 @ 3mL/min. Collect 12 ea 10mL splits. Total 120 mL EDTA through column.

0759 start flow @ 3mL/min
0804 1st outflow (4.5" @ 3mL/min) = PV = 13.5mL
0844 end EDTA, stop flow to column.

0848 start flow of RIM3 to Col 4 @ 1.2mL/min
0900 1st outflow (12" x 1.2 mL/min) = PV = 14.4mL
stop pumps.

Reprogram frac collector for RIM3 flow to both columns for 10mL collect.

0902 start flow of RIM3 to both columns @ 1.2mL minute Collect 5 ea 10mL splits for total of 50mL outflow of RIM3

0946 end flow of RIM3 to both colums @ 1.2mL min. Reset frac collectors, increase pump speed to 2.4mL/min

0947 start flow of RIM3 w/ 1mg/L U @ 2.4mL min. start frac collectors. 8.34 minute collect @ 2.4 mL/min for 20mL split. tubes 6 → 31

1350 ↑ rate to 3.6 mL/min, collect time 5.56" = 20mL split tubes 32 → 37

1430 maintain rate, ↑ collect time to 6.95" = 25mL split tubes 38 →

1600 Prepare RIM3 = 1mg/L U Batch 2 ck parameters. Start using @ start of tube 55

1750 reset frac collector w/ 44 acidified tubes (0.2mL conc HNO_3) Note: all tubes have been acidified prior to collection of sample.

22-210

MPERT01-13-03

6/15/04 1730 Columns running well.

2013 SHAN D/C Both columns.

6/16/04 0915 Send samples. Col 3 samples 3-66, 67, 74 and 83 required 100 µl additional conc HNO_3.
Soln was pale yellow in tubes, pH ck = 4.

Column 4 outflow was decreased. Check column and tubing. Insert needle into inlet and outlet of column. Flow appears increased.

1300 Will restart column c̄ size 14 heads @ 6 mL/min. Prepare new batch RIM3 c̄ 1mg/L U

1700 Finally restart columns c̄ size 13 heads @ 3.6 mL/min. Unable to make tight plumbing connections c̄ size 16 head (only 1 size 14 head available)

Set fraction collector to collect every ~~4th~~ 3rd sample. Now set @
~~14~~ 21 minutes drain = (~~50~~ 75 mL) then 7 minutes collect (≈25 mL)

New batch RIM3 = 1mg/L U checked (made 4L batch). Will switch to that @ tube #

First collect on new protocol is tube 85 collected

1820 Columns not running. Unable to program one of the frac colle

6/17/04 0700 Call Gilson re frac collector.

0800 Gilson - diagnose problem w/ drain. frac collectors running well now will restart column.

0830 Start 14" drain, 7" collect @ 3.6 mL/min. first tube off = 86

Monticello EPA Columns 3:4 MPERT01-13-04

6/17/04 1005 switch to RIM3 batch 3 (4L) source tank. Columns appear to be running well.

Switched inlet tubing between columns to check reason for ↓ outflow from Col 4. pumphead 1 (left) now supplying Col 4
2 (right) Col 3.

In addition, probed both top and bottom of Col 4 with 21ga needle through inlet and outlet. Will watch for change in outflow (at end tube 87)

1015 Col 3 outflow ↓. Will probe with needle. (drain p̄ tube 90)

1230 Col 3 outflow good til tube 95, ↓ 95, 96. Probe again with needle. flow ↑

1330 Col 3 short flow on tube 99, probe c̄ needle flow ↑

1500 Col 3 short flow tubes 106, 107, 108, probe c̄ needle during drain after 108. flow ↑

1700 Reset frac collector to start. Start RIM3 Batch 4 for overnight run

6/18/04 0728 Both columns had plugged overnight. D/C pump

Col 3 had essentially 0 flow (0.3 mL/7min) since tube 122 (~2200 6/17/04)
samples 121 → 150 not retained. 7 4% in tube = conc HNO_3

Col 4 had essentially zero flow (0.3/7min) since tube 143 (0530)
and severely reduced flow (0.6/7min) since tube 127 (2400)
samples 127 → 150 not retained.

Monticello EPA Col 3 ; 4 MPERT01-13-05

6/18/04 Check pump for flow - both heads = 3.6 ml/min
probe inlets and outlets of both columns. appears flow restored.
Note: if columns had been running well, ~3300 ml would have been
used from source tank. Only 1500 ml used.
0930 Restart flow to columns @ tube 151 drain first x 17" then 7 min collect
1500 D/C columns after tubes 165

22-210

Column 3 EDTA Monticello EPA

MPERT01-13-06

	Vol (ml)	Cum Vol (ml)	Rate (ml/min)	PV=135		Ca (mg/L)	Fe (mg/L)		Comments
3-E1	10	10	3.0			1895.0	5920		Clear pale yellow
E2	10	20	↓			1905.0	5780		" " "
E3	10	30				1650.0	5520		Dark greenish/brownish black
E4	10	40				1530.0	5200		↓
E5	10	50				1410.0	5180		
E6	10	60				1370.0	5280		
E7	10	70				1385	5380		
E8	10	80				1315	5180		
E9	10	90				1315	5220		
E10	10	100				1335	5220		
E11	10	110				1335	5540		
E12	10	120				1295	5260		
0.1M EDTA disodium salt, dihydrate						0.35	0.07		

Note: crystals noted in bottom of centrifuge tubes 6/18/04 – date samples analyzed for Ca

MPERT01-13-07

Monticello EPA Col 3 RIM3

	1 Vol (mL)	2 Cum Vol (mL)	3 Set Rate (mL/min)	4 Set Time (min)	5 Source Tank	6 pH (1=13.5)	7	Ca (mg/L)	U (ug/L)	Comments	12	13
3-1	8.5		1.2	8.34	RIM3			760		clear amber		
2	8.5							302		clear med. yellow		
3	8.5							207		clear light yellow		
4	8.5							174		clear pale yellow		
5	8.5							161		" " "		
6	19		2.4		RIM3EU					Clear light yellow		
7	19									Clear pale yellow		
8	19									" " "		
9	19									" " "		
10	19								<0.01	" " "		
11	19									Clear very pale yellow		
12	19									" " " "		
13	19									" " " "		
14	19									clear		
15	19											
16	19											
17	19											
18	19											
19	19											
20	19								<0.01			
21	19											
22	19											
23	19											
24	19											
25	19								<0.01			
26	19									{ Vol. through column, sample		
27	19									lost to frac collector, operator error		
28	19									5mL collect		
29	19											
30	19								1.41			
31	19		2.4	8.34								

22-210

MPERT01-13-08

Monticello EPA Col 3 RIM3

2

	Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Set Time (min)	Source tank	PV (1735)		Cl (mg/L)	U (ug/L)	Comments		12	13
3-32	19		3.6	5.56	RIM3ZU								
33	19												
34	19												
35	19												
36	19												
37	19								3.50				
38	24			6.95									
39	24												
40	24								4.02				
41	24												
42	24												
43	24												
44	24												
45	24												
46	24												
47	24												
48	24												
49	24												
50	24								7.66				
51	24												
52	24												
53	24												
54	16												
55	24									rate ok, inlet tube out of source			
56	24												
57	24												
58	24												
59	24												
60	24								5.97				
61	24												
62	24												

22-210

MPERT01-13-09

3

Col 3 RIM3 Mont EPA

	Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Time	Source Tank	PV 1=13.5	7	Ca (mg/L)	U (ug/L)	Comments	12	13
3-63	24		3.6	6.95	RIM3=U							
64	24											
65	24											
66	24											
67	24									pale yellow, 100 ul adtl HNO_3		
68	24									" " " " "		
69	24											
70	24								4.45			
71	24											
72	24											
73	24											
74	24											
75	24									pale yellow, 100 ul adtl HNO_3		
76	24											
77	24											
78	24											
79	24											
80	24								4.58			
81	24											
82	24											
83	24											
84	8								5.91	pale yellow, 100 ul adtl HNO_3 stop @ 1000 6/15/04		
drain	48			14						restart 1700 6/16/04		
85	24			7								
drain	48			14								
86	24			7								
drain	48			14								
87	24			7								
drain	~~24~~ 48			14								
88	24			7								
drain	~~24~~ 48			14								

22-210

MPERT01-13-10

Monticello EPA Col 3 RIM3EU

4

		Vol (mL)	Cum Vol (mL)	Set Rate (mL/m)	Collect Time	Source tank	PV (1=13.5)		Ca (mg/L)	U (ug/L)	10	11	12	13
3-89	1	19		3.6	7	RIM3EU								
drain	2	34			14									
90	3	15			7					4.00				
drain	4	36.5			14									
91	5	23			7									
	6	46			14									
92	7	23			7									
	8	46			14									
93	9	23			7									
	10	42			14									
94	11	19			7									
	12	30			14									
95	13	11			7									
	14	18.5			14									
96	15	7.5			7									
	16	30.5			14									
97	17	23			7									
	18	46			14									
98	19	23			7									
	20	31			14									
99	21	8			7									
	22	31			14									
100	23	23			7					11.44				
	24	46			14									
101	25	23			7									
	26	46			14									
102	27	23			7									
	28	46			14									
103	29	23			7									
	30	46			14									
104	31	23			7									

22-210

MPERT01-13-11

5

Monticello EPA Col 3 RIM3=U

		Vol(ml)	CumVol(ml)	Set Rate (ml/min)	Collect Time(min)	Source	pH (1=13.5	7	Ca (mg/L)	U (ug/L)	10	11	12	13
drain	1	45		3.6	14	RIM3-U								
3-105	2	22			7									
drain	3	33			14									
106	4	11			7									
	5	20			14									
107	6	9			7									
	7	17			14									
108	8	8			7									
	9	30			14									
109	10	22			7									
	11	44			14									
110	12	22			7					9.44				
	13	44			14									
111	14	22			7									
	15	44			14									
112	16	22			7					6.75				
	17	36			14									
113	18	14			7									
	19	22			14									
114	20	8			7									
	21	13			14									
115	22	5			7					23.0	Note short samples U# confirmed			
	23	9			14									
116	24	4			7									
	25	7			14									
117	26	3			7									
	27	6			14									
118	28	3			7									
	29	5.5			14									
119	30	2.5			7									
	31	5			14									

22-210

MPERT01-13-12

6

Monticello EPA Col 3 RIM3EU

	Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Collect	Source	N (1=13.5)		Ca (mg/L)	U (ug/L)				
3-120	2.5		3.6	7	RIM3-U								
	3.5			14									
121	1			7									
	1.3			14									
122	0.3			7									
	0.6			14									
123	0.3			7									
	0.6			14									
124	0.3			7									
	0.6			14									
125	0.3			7									
	0.6			14									
126	0.3			7									
	0.6			14									
127	0.3			7									
	0.6			14									
128	0.3			7									
	0.6			14									
129	0.3			7									
	0.6			14									
130	0.3			7									
	0.6			14									
131	0.3			7									
	0.6			14									
132	0.3			7									
	0.6			14									
133	0.3			7									
	0.6			14									
134	0.3			7									
	0.6			14									
135	0.3			7									

Monticello EPA Col 3 RIM3EU

	Vol (mL)	Cum Vol (mL)	Set Rate (mL/min)	Collect Time	Source	pH (1=135)		Ca (mg/L)	U (µg/L)				
drain	0.6		36	14	RIM3EU								
3-136	0.3			7									
	0.6			14									
137	0.3			7									
	0.6			14									
138	0.3			7									
	0.6			14									
139	0.3			7									
	0.6			14									
140	0.3			7									
	0.6			14									
141	0.3			7									
	0.6			14									
142	0.3			7									
	0.6			14									
143	0.3			7									
	0.6			14									
144	0.3			7									
	0.6			14									
145	0.3			7									
	0.6			14									
146	0.3			7									
	0.6			14									
147	0.3			7									
	0.6			14									
148	0.3			7									
	0.6			14									
149	0.3			7									
	0.6			14									
150	0.3			7									
	0.6			14									

22-210

MPERT01-13-14

8

Monticello EPA Column 3 RIM3=U

Sample		Vol (mL)	Cum Vol (mL)	Set Rate (mL/min)	Collect Time (min)	Source	PV (1=13.5)		Ca (mg/L)	U (µg/L)	Comments		12	13
3-151	1	22		3.6	7	RIM3				4.48				
	2	44			14									
152	3	22			7									
	4	44			14									
153	5	22			7									
	6	44			14									
154	7	22			7									
	8	44			14									
155	9	22			7									
	10	44			14									
156	11	22			7									
	12	44			14									
157	13	22			7									
	14	44			14									
158	15	22			7									
	16	44			14									
159	17	22			7									
	18	44			14									
160	19	22			7					11.45				
	20	44			14									
161	21	22			7									
	22	44			14									
162	23	22			7									
	24	44			14									
163	25	22			7									
	26	44			14									
164	27	22			7									
	28	44			14									
165	29	22			7					11.50				
	30	Off												
	31													

22-210

Mont EPA Column 4 RIM3

MPERT01-13-15

	Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Set Time (min)	Source Tank	PV (1=14.4)		Ca (mg/L)	U (ug/L)	Comments			
4-1	9		1.2	8.34	RIM3			1540		Clear very pale brown			
2	9		1.2	8.34				715		Clear			
3	9		1.2	8.34				479		"			
4	9		1.2	8.34				406		"			
5	9		1.2	8.34				376		"			
6	17.5		2.4	8.34	RIM3+U					Clear pale yellow			
7	17.5		2.4	8.34						Clear very pale yellow			
8	17.5		2.4	8.34						Clear			
9	17.5		2.4	8.34						"			
10	17.5		2.4	8.34									
11	17.5		2.4	8.34									
12	17.5		2.4	8.34					<0.01				
13	17.5		2.4	8.34									
14	17.5		2.4	8.34									
15	17.5		2.4	8.34									
16	17.5		2.4	8.34									
17	17.5		2.4	8.34									
18	17.5		2.4	8.34									
19	17.5		2.4	8.34					<0.01				
20	17.5		2.4	8.34									
21	17.5		2.4	8.34									
22	17.5		2.4	8.34									
23	17.5		2.4	8.34									
24	17.5		2.4	8.34									
25	17.5		2.4	8.34					<0.01				
26	17.5		2.4	8.34									
27	17.5		2.4	8.34									
28	17.5		2.4	8.34									
29	17.5		2.4	8.34									
30	17.5		2.4	8.34					2.05				
31	17.5		2.4	8.34									

22-210

MPERT01-13-16

Mont. EPA Column 4 RIM3

②

		Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Set Time (min)	Source Tank	PV (1=14.4)	7	Ci (mg/L)	U (ug/L)	Comments		12	13
4-32	1	17.5		3.6	5.56	RIM3EU								
33	2	17.5												
34	3	17.5												
35	4	17.5												
36	5	17.5												
37	6	17.5								6.54				
38	7	21			6.95									
39	8	21												
40	9	21								7.24				
41	10	21												
42	11	21												
43	12	21												
44	13	21												
45	14	21												
46	15	21												
47	16	21												
48	17	21												
49	18	21												
50	19	21								5.75				
51	20	21												
52	21	21												
53	22	21												
54	23	21												
55	24	21												
56	25	21												
57	26	21												
58	27	21												
59	28	21												
60	29	21								2.82				
61	30	21												
62	31	21												

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MPERT-01-13-17

3

Col 4 RIM3

		Vol (mL)	Cum Vol (mL)	Set (mL/min) Rate	Collect Time (min)	Source	PV (1=14.4 mL)		Ca (mg/L)	Cl (mg/L)	Comments		12	13
4-63	1	21		3.6	6.95	RIM3CV								
64	2	21												
65	3	21												
66	4	21												
67	5	21												
68	6	21												
69	7	21												
70	8	21								4.76				
71	9	21												
72	10	19									Column plugging			
73	11	15												
74	12	12												
75	13	10												
76	14	8												
77	15	7												
78	16	6												
79	17	4												
80	18	3								0.69				
81	19	2												
82	20	1.5												
83	21	1.5												
84	22	.5												
drain	23	42			14					2.59	clear plug		coll@ 8:15	6/15/04
85	24	21			7						start @ 1700 6/16/04			
drain	25	29			14									
86	26	8			7									
drain	27	18			14									
87	28	10			7									
drain	29	32			14									
88	30	22			7									
drain	31	44			14									

MPERT01-13-18

(4)

Mont. EPA Col 4 RIM3ZU

	Vol (mL)	Cum Vol (mL)	Set Rate (ml/min)	Collect Time (min)	Source	PV (1=14.4)		Ca (mg/L)	Cl (mg/L)	Comments		12	13
4-89	22		3.6	7	RIM3ZU								
	44			14									
90	22			7					8.65				
	44			14									
91	22												
	44												
92	22												
	44												
93	22												
	44												
94	22												
	44												
95	22												
	44												
96	22												
	44												
97	22												
	44												
98	22												
	44												
99	22												
	44												
100	22								12.42				
	44												
101	22												
	44												
102	22												
	44												
103	22												
	44												
104	22												

22-210

MPERT01-13-19

5

Monticello EPA Col 4 R1M3=U

		Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Collect Time (min)	Source	PV(1=14.4)	7	Ca (mg/L)	U (ug/L)	10	11	12	13
drain	1	44		3.6	14	R1M3=U								
4-105	2	22			7									
	3	44			14									
106	4	22			7									
	5	44												
107	6	22												
	7	44												
108	8	22												
	9	44												
109	10	22												
	11	44												
110	12	22								9.28				
	13	44												
111	14	22												
	15	44												
112	16	22												
	17	44												
113	18	22												
	19	44												
114	20	22												
	21	44												
115	22	22												
	23	44												
116	24	22												
	25	44												
117	26	22												
	27	44												
118	28	22												
	29	44												
119	30	22												
	31	44												

22-210

Zom

(6)

Monticello EPA Col 4 RIM3=U MPERT01-13-20

		Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Collect Time (min)	Source	PV (1=14.4)	7	Ca (mg/L)	U (µg/L)	10	11	12	13
4-120	1	22		3.6	7	RIM3=U				8.15				
	2	29.5			14									
4-121	3	7.5												
	4	11.5												
122	5	4												
	6	7												
123	7	3												
	8	5												
124	9	2												
	10	4												
125	11	2												
	12	4												
126	13	2												
	14	2.6												
127	15	0.6												
	16	1.2												
128	17	0.6												
	18	1.2												
129	19	0.6												
	20	1.2												
130	21	0.6												
	22	1.2												
131	23	0.6												
	24	1.2												
132	25	0.6												
	26	1.2												
133	27	0.6												
	28	1.2												
134	29	0.6												
	30	1.2												
135	31	0.6												

22-210

MPERT01-13-21

(7)

Monticello EPA Col 4 RIM3EU

		Vol (ml)	Cum Vol (ml)	Set Rate (ml/min)	Collect Time	Source	PV (1=14.4)	7	Ca (mg/L)	U (ug/L)	10	11	12	13
drain	1	1.2			14	RIM3EU								
4-136	2	0.6			7									
	3	1.2			14									
137	4	0.6			7									
	5	1.2												
138	6	0.6												
	7	1.2												
139	8	0.6												
	9	1.2												
140	10	0.6												
	11	1.1												
141	12	0.5												
	13	1												
142	14	0.5												
	15	0.8												
143	16	0.3												
	17	0.6												
144	18	0.3												
	19	0.6												
145	20	0.3												
	21	0.6												
146	22	0.3												
	23	0.6												
147	24	0.3												
	25	0.6												
148	26	0.3												
	27	0.6												
149	28	0.3												
	29	0.6												
150	30	0.3												
	31	0.6												

22-210

MPERT01-13-22

8

Montecello EPA Column 4 RIM3

		Vol (mL)	Cum Vol (mL)	Set Rate (mL/min)	Set Time (min)	Source	AV (1=14.4)		Ca (mg/L)	U (ug/L)	Comments		12	13
4-151	1	23		3.6	7	RIM3=U				2.35				
	2	46			14									
152	3	23			7									
	4	46			14									
153	5	23			7									
	6	46			14									
154	7	23			7									
	8	46			14									
155	9	23			7									
	10	46			14									
156	11	23			7									
	12	46			14									
157	13	23			7									
	14	46			14									
158	15	23			7									
	16	46			14									
159	17	23			7									
	18	46			14									
160	19	23			7					5.07				
	20	46			14									
161	21	23			7									
	22	46			14									
162	23	23			7									
	24	46			14									
163	25	23			7									
	26	46			14									
164	27	23			7									
	28	46			14									
165	29	23			7					4.12				
off	30				off									
	31													

Procedure for Column Tests 3 and 4 for EPA Region VIII
PRB Rejuvenation Project
Revision 1

Column tests to be conducted week of June 14, 2004

Sarah, Following is a brief outline of the tests. Please contact me if you have any questions or if you need help (Brandon should be available this week). The purpose of these tests is to determine if EDTA treatment improves U-uptake rates. From the previous column (Column 2) we determined that disodium EDTA was capable of removing calcite from ZVI.

Use OMNI 22 mL columns. Collect in fraction collector.

Use sample(s) PE 11-5, 11-6 and/or 11-7 (if you use more than one sample, be sure to blend them) for the tests. This is ZVI sampled from the August 2003 coring of the Monticello PRB. It was selected because it has some of the highest Ca concentrations (i.e. it should contain the most calcite). It has no rad component and because it is from the ZVI zone, it will not require magnetic separation.

Fill 2 columns (Columns 3 and 4) with the ZVI. Determine mass of ZVI in each. Make a solution (at least 120 mL) of 0.1 ~~mL~~ M disodium EDTA (pH should be about 5). Flow this solution through column 3 at 3 mL/min. It will take about 40 min to complete. Collect samples every 10 mL (total of 12 samples). Preserve in acid (about 1-2% HNO3) and analyze these for Ca. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

Make up a solution (about 1 liter should be sufficient) of synthetic R1M3 (no U). Run 50 mL of this solution through each column at 1.2 mL/min (residence time about 10 min). This should take about 42 min. Collect samples every 10 mL, preserve, and analyze for Ca and U. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

Spike R1M3 with 1 mg/L U. Run 400 mL (about 33 pore volumes) of this solution through each column at 2.4 mL/min (residence time about 5 min). This should take about 3 hr. Collect samples every 20 mL (total of 20 samples), preserve, and analyze for U. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

Continue to run 400 mL (about 33 pore volumes) of the U-spiked solution through each column at 1.2 mL/min (residence time about 10 min) or another specified rate dependent on the results. This should take about 6 hr. Collect samples every 20 mL (total of 20 samples), preserve, and analyze for U. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

	A	B	C	D	E	F	G	H	I	J	K
1	R1-M3, Samples 1/14/2003; for Rejuvenation Column Study Initiated- 4/28/03										
2		mL per									
3		L of	Na	K	Ca	Mg	SO4	Cl	C	U	V
4		Soln	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
5	Liquid Components										
6	V2O5, V=996mg/L, 2%HNO3	0.00									0.00
7	V2O5, V=1000mg/L, 5%HNO3	0.00									0.00
8	V2O5, V=1000mg/L, 1.4%HNO3	0.00									0.00
9	V2O5, V=1000mg/L, 2%HNO3	0.00									0.00
10	Na2MoO4.2H2O, Mo=925mg/L	0.00	0.00								
11	Na2MoO4.2H2O, Mo=1000 mg/L	0.00	0.00								
12	(NH4)2MoO4, 1000 mg/L Mo	0.00									
13	U3O8, U=10000mg/L, 3.5%HNO3	0.00								0.00	
14	U3O8, U=10000mg/L, 5.2%HNO3	0.00								0.00	
15	U Std, U=1000mg/L, 2% HNO3	0.00								0.00	
16	Ra Std, Ra=68.7pCi/mL, 2% HNO3	0.00									
17	As Std, As=999mg/L, 5%HNO3	0.00									
18	As Std, As=1000mg/L, 1.4%HNO3	0.00									
19	Se =999 mg/L 5% HNO3	0.00									
20	Cd Std = 1000mg/L, ~2% HNO3	0.00									
21											
22	100g/L K2CO3	0.00		0.00					0.00		
23	50g/L NaHCO3	11.00	150.60						78.57		
24	100g/L Na2SO4.10H2O	0.00	0.00				0.00				
25	100g/L Na2SO4.(0.89 H2O)	0.00	0.00				0.00				
26	500g/L (NH4)2SO4	0.00					0.00				
27	1.5g/L CaSO4.2H2O	515.00			179.93		430.96				
28	1.2 g/L CaSO4	0.00			0.00		0.00				
29	200g/L MgSO4.7H2O	2.70				53.28	210.48				
30	100g/L K2SO4	0.25		11.22			13.78				
31	conc H2SO4(36N)	0.00					0.00				
32	927.2 g/L CaCl2.2H2O	0.00			0.00			0.00			
33	92.72 g/L CaCl2.2H2O	1.70			42.95			76.09			
34	300g/L NaCl	0.00	0.00					0.00			
35	100g/L MgCl2.6H2O	0.00				0.00		0.00			
36	100g/L NH4Cl	0.00						0.00			
37	100g/L NaNO3	0.00	0.00								
38	2% HNO3	0.00									
39	mL 1N NaOH needed to neutralize	0.00	0.00								
40	66.67 g/L NaN3	0.00	0.00								
41											
42											
43											
44	Totals (mg/L)	530.65	150.60	11.22	222.88	53.28	655.21	76.09	78.57	0.00	0.00
45	ACTUAL (mg/L)		111.00	11.30	213.00	52.80	677.00	76.80	76.00	0.57	0.35
46	Totals (mol/L)		6.55E-03	2.87E-04	5.56E-03	2.19E-03	6.83E-03	2.15E-03	6.55E-03	0.00E+00	0.00E+00
47											
48	ACTUAL pH	6.85									
49	Measured pH(no acid or base)	?									
50	Measured pH (________ added)										
51	ACTUAL Alk (mg/L CaCO3)	317.00									
52	Measure Alk (no acid or base)	?									

WORK SUBMITTAL TO ENVIRONMENTAL SCIENCES LABORATORY

Submittal Date 6/14/2004 Date Required 6/18/2004

Submitted By Stan Morrison Signature Stan Morrison by SM

Formal ESL Report ? Yes ✓ No ___

Data Report Only? Yes ___ No ___

Electronic Data Deliverable? Yes ___ No ✓

Project: EPA-PRB-Phase II Charge No. 113401105

Analysis Type (check one): Kd ___ Leaching ___

Expedited Site Characterization ___ Other (Specify) ✓

Column

Sample Numbers PE 11-5, Columns 3 & 4

Analytes Ca, Fe, U, pH, ORP, Cond, alkalinity

Solution Composition

See attached recipe (1) 0.1M EDTA disodium salt dihydride (2) RIM3 (3) RIM3 spiked 1mg/L U

Comments (attach procedure if needed)

6/14/2004 See attached procedure.

See attached recipe for synthetic RIM3

Procedure for Column Tests 3 and 4 for EPA Region VIII
PRB Rejuvenation Project
Revision 1

Column tests to be conducted week of June 14, 2004

Sarah, Following is a brief outline of the tests. Please contact me if you have any questions or if you need help (Brandon should be available this week). The purpose of these tests is to determine if EDTA treatment improves U-uptake rates. From the previous column (Column 2) we determined that disodium EDTA was capable of removing calcite from ZVI.

Use OMNI 22 mL columns. Collect in fraction collector.

Use sample(s) PE 11-5, 11-6 and/or 11-7 (if you use more than one sample, be sure to blend them) for the tests. This is ZVI sampled from the August 2003 coring of the Monticello PRB. It was selected because it has some of the highest Ca concentrations (i.e. it should contain the most calcite). It has no rad component and because it is from the ZVI zone, it will not require magnetic separation.

Fill 2 columns (Columns 3 and 4) with the ZVI. Determine mass of ZVI in each. Make a solution (at least 120 mL) of 0.1 ~~mL~~ M disodium EDTA (pH should be about 5). Flow this solution through column 3 at 3 mL/min. It will take about 40 min to complete. Collect samples every 10 mL (total of 12 samples). Preserve in acid (about 1-2% HNO3) and analyze these for Ca. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

Make up a solution (about 1 liter should be sufficient) of synthetic R1M3 (no U). Run 50 mL of this solution through each column at 1.2 mL/min (residence time about 10 min). This should take about 42 min. Collect samples every 10 mL, preserve, and analyze for Ca and U. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

Spike R1M3 with 1 mg/L U. Run 400 mL (about 33 pore volumes) of this solution through each column at 2.4 mL/min (residence time about 5 min). This should take about 3 hr. Collect samples every 20 mL (total of 20 samples), preserve, and analyze for U. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

Continue to run 400 mL (about 33 pore volumes) of the U-spiked solution through each column at 1.2 mL/min (residence time about 10 min) or another specified rate dependent on the results. This should take about 6 hr. Collect samples every 20 mL (total of 20 samples), preserve, and analyze for U. Analyze source tank for pH, ORP, conductivity, alkalinity, U and Ca.

	A	B	C	D	E	F	G	H	I	J	K
1	R1-M3. Samples 1/14/2003; for Rejuvenation Column Study initiated- 4/28/03										
2		mL per									
3		L of	Na	K	Ca	Mg	SO4	Cl	C	U	V
4		Soln	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
5	Liquid Components										
6	V2O5, V=998mg/L, 2%HNO3	0.00									0.00
7	V2O5, V=1000mg/L, 5%HNO3	0.00									0.00
8	V2O5, V=1000mg/L, 1.4%HNO3	0.00									0.00
9	V2O5, V=1000mg/L, 2%HNO3	0.00									0.00
10	Na2MoO4.2H2O, Mo=825mg/L	0.00	0.00								
11	Na2MoO4.2H2O, Mo=1000 mg/L,	0.00	0.00								
12	(NH4)2MoO4, 1006 mg/L Mo	0.00									
13	U3O8, U=10000mg/L, 3.5%HNO3	0.00								0.00	
14	U3O8, U=10000mg/L, 5.2%HNO3	0.00								0.00	
15	U Std, U=1000mg/L, 2% HNO3	0.00								0.00	
16	Ra Std, Ra=68.7pCi/mL, 2% HNO3	0.00									
17	As Std, As=999mg/L, 5%HNO3	0.00									
18	As Std, As=1000mg/L, 1.4%HNO3	0.00									
19	Se =999 mg/L, 5% HNO3	0.00									
20	Cd Std = 1000mg/L, ~2% HNO3	0.00									
21											
22	100g/L K2CO3	0.00		0.00					0.00		
23	50g/L NaHCO3	11.00	150.60						78.57		
24	100g/L Na2SO4.10H2O	0.00	0.00				0.00				
25	100g/L Na2SO4.(0.89 H2O)	0.00	0.00				0.00				
26	500g/L (NH4)2SO4	0.00					0.00				
27	1.5g/L CaSO4.2H2O	515.00			179.93		430.96				
28	1.2 g/L CaSO4	0.00			0.00		0.00				
29	200g/L MgSO4.7H2O	2.70				53.28	210.48				
30	100g/L K2SO4	0.25		11.22			18.78				
31	conc H2SO4(36N)	0.00					0.00				
32	927.2 g/L CaCl2.2H2O	0.00			0.00			0.00			
33	92.72 g/L CaCl2.2H2O	1.70			42.95			76.09			
34	300g/L NaCl	0.00	0.00					0.00			
35	100g/L MgCl2.6H2O	0.00				0.00		0.00			
36	100g/L NH4Cl	0.00						0.00			
37	100g/L NaNO3	0.00	0.00								
38	2% HNO3	0.00									
39	mL 1N NaOH needed to neutralize	0.00	0.00								
40	66.67 g/L NaN3	0.00	0.00								
41											
42											
43											
44	Totals (mg/L)	530.85	150.60	11.22	222.88	53.28	655.21	76.09	78.57	0.00	0.00
45	ACTUAL (mg/L)		111.00	11.30	213.00	52.80	677.00	76.80	76.00	0.57	0.35
46	Totals (mol/L)		6.55E-03	2.87E-04	5.58E-03	2.19E-03	6.83E-03	2.15E-03	6.55E-03	0.00E+00	0.00E+00
47											
48	ACTUAL pH	6.65									
49	Measured pH (no acid or base)	?									
50	Measured pH (______ added)										
51	ACTUAL Alk (mg/L CaCO3)	317.00									
52	Measure Alk (no acid or base)	?									

WORK SUBMITTAL TO ENVIRONMENTAL SCIENCES LABORATORY

Submittal Date 5/13/2004 Date Required ____
Submitted By Stan Morrison Signature Stan Morrison by Sm
Formal ESL Report ? Yes ✓ No ___
Data Report Only? Yes ___ No ___
Electronic Data Deliverable? Yes ___ No ___
Project: EPA PRB - PHASE II Charge No. 11340110S
Analysis Type (check one): Kd ___ Leaching ___
Expedited Site Characterization ___ Other (Specify) ✓

Rejuvenation column test (Col #2)

Sample Numbers Zn/gravel mix

Analytes Cu, Fe

Solution Composition
RIM3 - see attached recipe

Comments (attach procedure if needed)

Use Qubit Vernier data logger

Set. frac collector to 35 minute collects
205 minute drains

	A	B	C	D	E	F	G	H	I	J	K
1	R1-M3, Samples 1/14/2003; for Rejuvenation Column Study Initiated- 4/28/03										
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3		L of	Na	K	Ca	Mg	SO4	Cl	C	U	V
4		Soln	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
5	Liquid Components										
6	V2O5, V=996mg/L, 2%HNO3	0.00									0.00
7	V2O5, V=1000mg/L, 5%HNO3	0.00									0.00
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10	Na2MoO4.2H2O, Mo=925mg/L	0.00	0.00								
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12	(NH4)2MoO4, 1006 mg/L Mo	0.00									
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20	Cd Std = 1000mg/L, ~2% HNO3	0.00									
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23	50g/L NaHCO3	11.00	150.60						78.57		
24	100g/L Na2SO4.10H2O	0.00	0.00				0.00				
25	100g/L Na2SO4.(0.89 H2O)	0.00	0.00				0.00				
26	500g/L (NH4)2SO4	0.00					0.00				
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33	92.72 g/L CaCl2.2H2O	1.70			42.95			76.09			
34	300g/L NaCl	0.00	0.00					0.00			
35	100g/L MgCl2.6H2O	0.00				0.00		0.00			
36	100g/L NH4Cl	0.00						0.00			
37	100g/L NaNO3	0.00	0.00								
38	2% HNO3	0.00									
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