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**DYNAMIC COLUMN (ELUTION) AND  
ADSORPTION STUDIES ON SOIL AND  
WATER SAMPLES FROM THE  
CIMARRON CORPORATION SITE IN  
CRESCENT, OKLAHOMA**

June 20, 2006

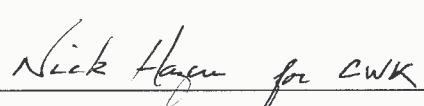
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Hazen Project 10364

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## **INTRODUCTION**

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On January 25, 2006, ENSR International authorized Hazen Research, Inc. to perform dynamic column (elution) studies on soils and adsorption studies on waters collected from the Cimarron Corporation site in Crescent, Oklahoma. This work is in response to Hazen's Proposal 2006-018, Revision 1 to ENSR dated January 25, 2006.

The purpose of the studies was to evaluate uranium leachability from aquifer material under a dynamic pumping environment and to evaluate the adsorption capacity of underlain soil at the soil storage facility. Therefore, both leaching and sorption tests were performed. Data obtained from these tests will be used in remedial system design and will also allow for the calculation of uranium soil–water distribution coefficients ( $K_d$ ). Additionally, ENSR wished to evaluate the feasibility and effectiveness of commercially available sorbent media in removing uranium from groundwater in order to obtain data that will be used for designing a full-scale treatment system. Therefore, equilibrium batch tests and dynamic column tests were performed.

Client representative Dr. Xiaoyun (James) Cao was present twice to supervise the equilibrium batch tests and the start of the dynamic column tests. He also witnessed the start of the elution tests.

## TECHNICAL APPROACH AND SUMMARY OF RESULTS

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Five soil samples, identified as SC-01 (7.3 kg), SC-02 (14.8 kg), SC-03 (7.2 kg), SC-04 (1.6 kg), and SC-05 (6.5 kg) were received from the Cimarron site on January 30, 2006. In addition, three core samples, labeled SC-01BD, SC-03BD, and SC-04BD, were received for determination of bulk densities. Subsequently, about 2 kg of additional SC-04 material was received from the Cimarron site on February 21, 2006, to have sufficient material to fill a column.

The soil samples were prepared by drying at 60°C under a blanket of N<sub>2</sub> to near-complete dryness. The dried samples were broken up to pass a 6-mesh screen, homogenized, and sampled for residual moisture and for analysis. Residual moistures amounted to only a few tenths of a percent. Analytical samples were sent to General Engineering Laboratories, LLC (GEL) in Charleston, SC. Results were reported directly to ENSR.

Four water samples, identified as TMW-09 (55 gal), TMW-02 (30 gal), 02W04 (55 gal), and 02W33 (10 gal) were received on February 17, 2006. Samples were contained in 5-gal plastic bottles packaged in coolers. Upon receipt, the samples were combined into their respective composites. Water composites were checked for pH every 1–3 days and adjusted to their original values, if necessary, by bubbling in CO<sub>2</sub>. About 10 gal of synthetic rainwater was prepared according to EPA Method 1312 for West of the Mississippi Extraction Fluid.

The elution studies were performed in 3-in.-diameter by 12-in.-long columns. The construction of the columns followed the guidelines in ASTM D4874, except that the columns were constructed of clear polyvinyl chloride (PVC) rather than glass and the end pieces were made of solid PVC rather than stainless steel (SS). PVC is inert and considered an acceptable replacement for glass or SS when pressure requirements are met. The existing end pieces were modified to accept the 3-in.-diameter columns, which were able to withstand an internal pressure greater than 100 psi. A 100-µm porous SS plate was used as the distributor at the inlet end of each column. The discharge ends of each column were filled with sand wrapped in filter media to occupy the space between the packed soil and the end piece. Water was introduced to each column using a N<sub>2</sub>-pressurized SS container. The water feed rate was regulated by adjusting the pressure in the container and measuring the rate of discharged effluent. Figure 1 shows Elution Column 1 in operation. Photos of the other columns in operation are shown in Appendix B (Figures B8 and B9); Figure B10 (Appendix B) shows the bulk-water storage containers.



**Figure 1. Elution Column 1 in Operation**

The six elution columns were prepared and operated for about 16 days with the following specifications:

- Column 1: SC-01 soil and TMW-02 water operated upflow at 2 pore volumes (PV)/day.
- Column 2: SC-02 soil and TMW-02 water operated upflow at 2 PV/day.
- Column 3: SC-02 soil and 02W33 water operated upflow at 2 PV/day.
- Column 4: SC-03 soil and TMW-02 water operated upflow at 1 PV/day.
- Column 5: SC-04 soil and TMW-02 water operated upflow at 1 PV/day.
- Column 6:  $\frac{1}{3}$  SC-02 soil,  $\frac{2}{3}$  SC-05 soil, and prepared synthetic rainwater operated downflow at 1 PV/day.

Discharge samples were collected at the intervals specified by the client. One portion of each sample was acidified with HNO<sub>3</sub> to pH 2 for eventual uranium analysis, another portion was stored at 4°C for eventual analysis of anions, and the remainder was added to a composite for discharge from each column. Samples for analysis were shipped to GEL.

Each column was loaded by placing layers of dry soil into the column and wetting with water. A vibrator placed on the outside of the column was used to aid in displacing air. After the end piece was bolted into place, the column was saturated with the water designated for that particular column

and allowed to stand for 12 hr. Column operations were started on February 23, 2006. Flow rates were checked and adjusted at 12-hr intervals throughout the duration of the testing. Table 1 shows column packing and flow rate data.

**Table 1. Column Packing and Initial Flow Rate Data**

Column No.	Soil Designation	Dry Density, g/cm <sup>3</sup>	Packed Weight, g	Calculated Voids <sup>a</sup> V <sub>p</sub> , cm <sup>3</sup>	Packed Dry Density, <sup>b</sup> g/cm <sup>3</sup>	Flow Rate Targets, <sup>c</sup> mL/hr
1	SC-01	2.67	2,256	545	1.6	45
2	SC-02	2.68	2,427	484	1.7	40
3	SC-02	2.68	2,450	476	1.8	40
4	SC-03	3.06	1,949	753	1.4	31
5	SC-04	2.82	2,225	601	1.6	25
6	<sup>2/3</sup> SC-05, <sup>1/3</sup> SC-01	2.70	2,230	564	1.6	24

<sup>a</sup>Void = column volume - (packed weight ÷ dry density), where the column volume is 1,399 cm<sup>3</sup>.

<sup>b</sup>Packed density = packed weight ÷ empty column volume.

<sup>c</sup>The flow rate targets were based on 1 PV every 12 hr for Columns 1, 2, and 3, and 1 PV every 24 hr for Columns 4, 5, and 6.

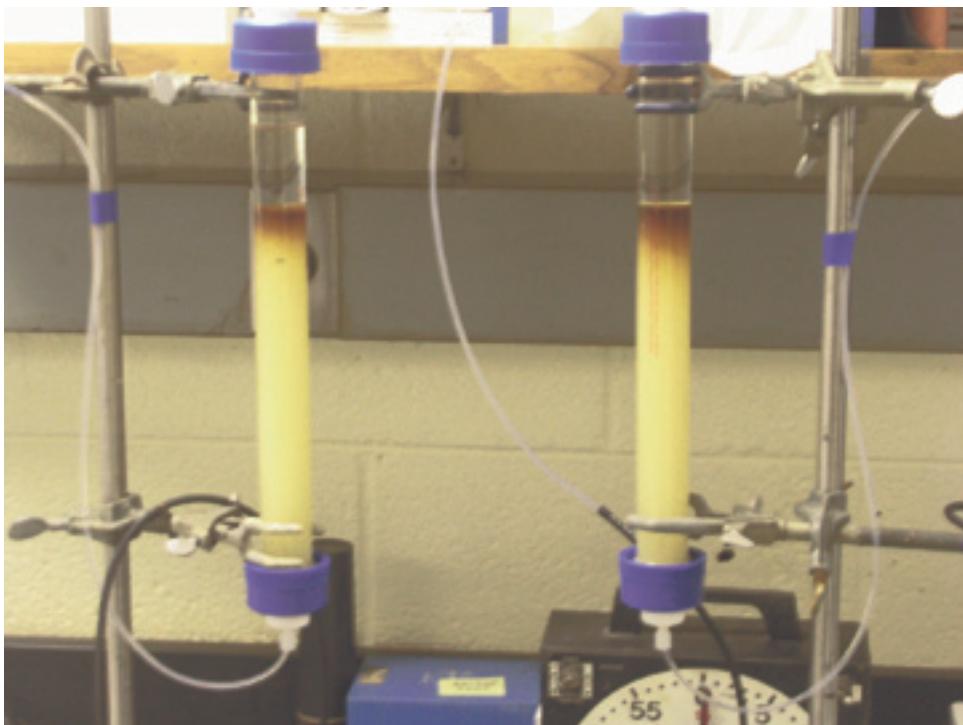
The flow through Column 4, which contained clayey soil, essentially stopped after one day of operation; therefore, after 4 days, and at the direction of the client, the column was dismantled, emptied, and loosely repacked with 1,757 g of soil. Flow rates again decreased to nearly zero after about 2 days.

At the direction of the client, the flows to Columns 2 and 3 were shut off after eight days, then restarted four days later at one-third the original flow rate, that is, 1 PV every 36 hr instead of every 12 hr.

Due to Hazen's oversight, the temperature, conductivity, pH, and oxidation-reduction potential (ORP) measurements were not taken for the column discharge sample during the first 5 days of operation. To gather this missing data, the client performed a series of tests in which soil and water representative of the contents of each column were placed into 500-mL bottles. The pH of each bottle was monitored and periodically adjusted with HCl to levels expected to be present within each column. The client did the pH measurements and HCl adjustments, and Hazen took the final measurements.

Three sorbents were received on February 6, 2006: Rohm and Haas Amberjet<sup>TM</sup> 4400 CL Resin (dry), Supelco Dowex<sup>®</sup> 21K Anion Exchange Resin (dry), and ARCTECH Humasorb-CS<sup>®</sup> (under water). Bottle tests using varying amounts of the three sorbents with constant amounts of 02W04 or TMW-09 waters containing about 1 and 4 ppm uranium, respectively, were performed to test the adsorptive

capacity of each sorbent. Uranium analyses were performed in Hazen's analytical laboratory. Both resins demonstrated the ability to remove uranium to less than detectable limits, but the Humasorb removed very little uranium. The Dowex resin was selected by the client for use in the breakthrough tests. Two breakthrough tests were performed using 02W04 water at two flow rates. The client-designated samples were acidified with HNO<sub>3</sub> and shipped to GEL for uranium analysis. Figure 2 shows the two breakthrough columns. The columns were not operated concurrently.



**Figure 2. Breakthrough Columns after Completion of Testing**

## **EXPERIMENTAL DATA**

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The receipt and handling of test, analytical, and residual samples are covered in Hazen's Standard Operating Procedures in Appendix A. Photographs taken during the test program are in Appendix B. Appendix C contains the logbook entries for the duration of the project. Pages 8–15 of the logbook in Appendix C show the Statement of Work for the dynamic column (elution) studies, and Pages 16–19 show the procedure for performing the adsorption isotherm–adsorption breakthrough curve column testing. Appendix D contains copies of all Chains of Custody forms. Appendix E contains a water-check log, Appendix F contains soil elution column effluent measurements and column-test logs, and Appendix G contains breakthrough column data.

## **SAMPLES AND SAMPLE PREPARATION**

The following describes the samples that were received and the preparation of the samples prior to starting the studies.

### **Soils**

#### **Receipt**

The initial soil samples were received on January 30, 2006, in good condition. Table 2 shows the client identifications and the numbers that Hazen assigned for future reference.

**Table 2. Soil and Core Identifications**

HRI	Client Identification	Approximate Weight, kg
51177-1	SC-01, 10' – 15'	7.3
51177-2	SC-01BD, 12' – 12.5'	0.5
51177-3	SC-02A, 15' – 18'	14.8
51177-4	SC-03, 17' – 19.5'	7.2
51177-5	SC-03BD, 14' – 14.5'	0.9
51177-6	SC-04, 16' – 17'	1.6
51177-7	SC-04BD, 12.5' – 15'	0.9
51177-8	SC-05, 0 – 0.5'	6.5

Subsequently, about 2 kg of additional SC-04 soil were received on February 21, 2006, in order to have sufficient material to fill the column. Digital photographs were taken of the as-received soils and are included in Appendix B.

### **Sample Preparation**

A sample preparation flowsheet is shown in Figure 3. Bulk densities were determined on the samples with the BD designation, which consisted of 3-in.- to 4-in.-long and 3-in.-diameter cores that were sealed in plastic to retain the original moisture. Each as-received sample was weighed, and its physical dimensions were measured. Sample SC-01BD did not retain its original shape; therefore, it was not possible to measure its dimensions. The dimensions of the other two samples were estimated. The samples were dried and reweighed. Table 3 shows the results of the bulk density determinations.

Soils Received  
on 1/30/06

1. SC-01
2. SC-01BD
3. SC-02A
4. SC-03
5. SC-03BD
6. SC-04
7. SC-04BD
8. SC-05

Determine the Bulk Density  
of Each of the  
Following Samples

SC-01BD  
SC-03BD  
SC-04BD

Prepare Each of the  
Following Samples  
for Column Testing

SC-01  
SC-02  
SC-03  
SC-04  
SC-05



Procedure:

1. Unseal each sample.
2. Measure its weight.
3. Measure its dimensions.
4. Dry at 110°C.
5. Reweigh and determine BD.

Procedure:

1. Unseal each sample.
2. Measure its weight.
3. Dry at 60°C, if necessary.  
(Under a N<sub>2</sub> cover, record initial and final weights; minimize exposure to air.)
4. Thoroughly homogenize the sample.
5. Take an aliquot for residual moisture determination.
6. Take an aliquot for uranium analysis.
7. Seal dried solids under N<sub>2</sub> until used.
8. SC-01 will be used for Column Test No. 1.
9. SC-02 will be equally split and used for Column Tests No. 2 and 3.
10. SC-03 will be used for Column Test No. 4.
11. SC-04 will be used for Column Test No. 5.
12. SC-05 and either SC-01, -02, -03, or -04 will be used for Column Test No. 6.

Note: Sample SC-04 was to be used as Column Test 5. As of 2/2/06, the sample will be prepared and tested in the same manner as the samples for Column Tests 1, 2, 3, and 4. The size of Column 5 will be the same size as the rest of the columns.

Figure 3. Soil Sample Preparation Flowsheet

**Table 3. Bulk Densities Determined by Measurements**

Measurement	Sample		
	SC-01BD	SC-03BD	SC-04BD
Diameter, cm	-- <sup>a</sup>	7.62	6.99
Height, cm	-- <sup>a</sup>	9.84	10.16
Vol, cm <sup>3</sup>	--	449	390
Wet Wt., g	458.3	917.1	852.6
Dry Wt., g	381.2	711.3	775.3
Moisture, %	16.8	22.4	9.1
Solids, %	83.2	77.6	90.9
Wet Density, g/cm <sup>3</sup>	--	2.0	2.2
Dry Density, g/cm <sup>3</sup>	--	1.6	2.0

<sup>a</sup>No shape

Because no measurements were available for Sample SC-01BD, a second method of bulk density determination was used for all three samples. The dried samples were coated with paraffin to seal the pores and weighed in both air and water to determine the dry bulk density. Table 4 shows these results.

**Table 4. Bulk Densities Determined by Paraffin Coating**

Weights	Sample		
	SC-01BD	SC-03BD	SC-04BD
In Air, g	367.47	567.13	774.29
In Water, g	112.78	169.95	357.59
Bulk Density, g/cm <sup>3</sup>	1.44	1.43	1.86

Determining the bulk density by paraffin coating of porous samples is a common method used by Hazen, and the results are considered more accurate than the dimensional measurements.

The soil samples for column testing were very wet; therefore, they were dried almost completely at 60°C under a blanket of N<sub>2</sub>. The very hard, dried samples were crushed in a jaw crusher, screened to minus 6 mesh, and thoroughly blended. Table 5 shows the initial moisture contents and the weights of the samples before and after drying. Residual moistures were very low at a few tenths of a percent.

**Table 5. Weights and Moisture Contents of Soils**

Samples for Column Testing			
Soil No.	Wet Wt., g	Dry Wt., g	Initial Moisture, %
SC-01	7,254	6,106	15.8
SC-02	14,804	13,036	11.9
SC-03	7,241	5,466	24.5
SC-04	1,622	1,413	12.9
SC-05	6,534	6,176	5.5

Because the amount of SC-04 soil was not sufficient to fill a column, two additional core pieces, labeled SC-04 16' – 17' and SC-04 17' – 17.5', were supplied. These samples were dried and prepared in the same way as the original soils. All the SC-04 16' – 17' (13.6% moisture, 854 g dry) and about one-half the SC-04 17'-17.5' (9.1% moisture, 906 g dry) were added to the original SC-04 sample for a total sample weight of about 2,700 g. A 50-g split of each soil was taken for uranium analysis.

In order to accurately calculate PVs in loaded columns, the actual particle density of each dried soil was determined using a stereopycnometer. These results are shown in Table 6.

**Table 6. Densities of Soils as Determined Using a Stereopycnometer**

Measurement	Sample				
	SC-01BD	SC-03BD	SC-04BD	SC-02	SC-05
Density, g/cm <sup>3</sup>	2.67	3.06	2.82	2.68	2.72

### Waters

Water samples were received on February 17, 2006. Table 7 shows the client sample identifications and the numbers that Hazen assigned for future reference.

The waters were received in 5-gal plastic containers packaged in coolers. Upon inspecting the as-received coolers, one container of TMW-02 was found to have leaked about half of its contents due to a break in the plastic. The remaining material was high in solids; therefore, the contents of this container were not used. The remaining waters were combined into appropriately sized drums or tanks. Each combined water sample was analyzed for uranium, major cations (preserved with HNO<sub>3</sub>), and anions (preserved at 4°C).

**Table 7. Water Identifications**

HRI	Client Identification	Approximate Volume, gal
51187-1	TMW-09, 11 containers	55
51187-2	TMW-02, 6 containers	30
51187-3	02W04, 11 containers	55
51187-4	02W33, 2 containers	10

One column test required using synthetic rainwater prepared according to EPA Method 1312 for Leaching Fluid West of the Mississippi (Extraction Fluid #2 was made by adding a 60:40 wt% mixture of H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> to DI water until the pH was 5.00 ± 0.05). Ten gal of solution were prepared. Table 8 shows the measurements taken after the waters were combined or prepared.

**Table 8. Initial Water Measurements**

Measurement	Sample				
	02W04	TMW-02	TMW-09	02W33	Synthetic Rainwater
Temp, °C	12	16	12	12	13
Conductivity, mS/cm	1.4	0.5	1.0	2.0	0.0
pH	7.08	7.46	7.06	7.25	5.01
Eh, <sup>a</sup> mV	629	577	580	579	604
Adjusted pH <sup>b</sup>	6.76	No adjustment	6.70	6.70	No adjustment

<sup>a</sup>The Eh was determined using a platinum-silver, AgCl-saturated, KCl-filled combination electrode and adding 199 to the reading.

<sup>b</sup>The pH was adjusted with CO<sub>2</sub> bubbling.

Water measurements were taken frequently during the course of the studies, and the pH was adjusted when necessary by bubbling CO<sub>2</sub> into the water. A water-check log is contained in Appendix E.

### Sorbents

Three 1-kg samples of sorbents were received on February 6, 2006, in good condition. These were Rohm and Haas Amberjet™ 4400 CL Resin (dry), Supelco Dowex® 21K Anion Exchange Resin (dry), and ARCTECH Humasorb-CS® (under water). The sorbents were used for the adsorption studies.

## DYNAMIC COLUMN (ELUTION) STUDIES

### Setup of Columns

The elution studies were performed in 3-in.-diameter by 12-in.-long columns. The construction of the columns followed guidelines found in ASTM D4874, except that the columns were constructed of clear PVC rather than glass and the end pieces were made of solid PVC rather than SS. The existing end pieces were modified to accept the 3-in.-diameter columns, which were able to withstand an internal pressure greater than 100 psi. A 100- $\mu\text{m}$  porous SS plate was used as the distributor at the inlet end of each column. The discharge ends of each column were filled with sand wrapped in filter media that occupied the space between the packed soil and the end piece. Water was introduced to each column using a N<sub>2</sub>-pressurized SS container. The water feed rate was controlled by adjusting the pressure in the container and measuring the rate of the discharged effluent. Photos of the columns in operation are shown in Appendix B (Figures B8 and B9); Figure B10 (Appendix B) shows the bulk-water storage containers.

### Operations

Figure 4 shows the general parameters for the column soils, feed waters, and flow rates. Each column was packed with the appropriate soil by placing dry soil into the column in stages, wetting the soil with DI water for Column 1 and the column's designated water for the remaining columns, and vibrating the column to eliminate or minimize air spaces. The end cap was then bolted into place, and the column was attached to the frame. The soil in each column was thoroughly wetted with feed water, then allowed to stand for at least 12 hr to equilibrate. All columns were started on February 23, 2006. Flow rates were checked and reset during the day starting at 0800, with the last check made at 2000 each day.

Solids: SC-01 Fine, Silty Sand	Solids: SC-02 Coarse Sand	Solids: SC-02 Coarse Sand	Solids: SC-03 Clay	Solids: SC-04 Sand- stone B Core	Solids: $\frac{1}{3}$ SC-01 and $\frac{2}{3}$ SC-05 Top Soil
Column 1	Column 2	Column 3	Column 4	Column 5	Column 6
Solution: TMW-02 Sandstone B Water	Solution: TMW-02 Sandstone B Water	Solution: 02W33 Sandstone C Water	Solution: TMW-02 Sandstone B Water	Solution: TMW-02 Sandstone B Water	Solution: Synthetic Rainwater
Feed: Upflow at 2 PV/day, 16 Days Minimum	Feed: Upflow at 2 PV/day, 16 Days Minimum	Feed: Upflow at 2 PV/Day, 16 Days Minimum	Feed: Upflow at 1 PV/day, 16 Days Minimum	Feed: Upflow at 1 PV/day, 16 Days Minimum	Feed: Downflow at 1 PV/day, 16 Days Minimum
Est. Soln. Required: 22 L	Est. Soln. Required: 22 L	Est. Soln. Required: 22 L	Est. Soln. Required: 11 L	Est. Soln. Required: 11 L	Est. Soln. Required: 11 L

Figure 4. Elution Columns Operating Parameters

The flow through Column 4, which contained clayey soil, virtually stopped very shortly after starting. The column was stopped and loosely repacked after 4 days. Upon startup, the flow was established for about a day, then diminished to a very low flow rate again.

The tables in Appendix F are logs of daily measurements for each of the columns. Due to an oversight, the effluent measurements for temperature, pH, ORP, and conductivity were not taken for the first 5 days, but were taken at each sampling period thereafter.

Effluent samples discharged into separate plastic buckets for each column. At the end of each PV period, when sufficient sample was available (100 cm<sup>3</sup> minimum), about 100 cm<sup>3</sup> of sample were withdrawn from the bucket into a separate syringe for each column, filtered through a 0.45-µm Millipore apparatus, and acidified with HNO<sub>3</sub> to pH 2 for uranium analysis. An additional 100 cm<sup>3</sup> (if available) of unacidified sample were collected and stored at 4°C for possible future analysis. Any remaining sample was added to a composite for each column. Although the buckets were emptied at the end of each sampling period, they were not cleaned prior to collecting the next sample. If 100 cm<sup>3</sup> were not available at the end of a sampling period, the sampling was bypassed until a minimum of 100 cm<sup>3</sup> were available. All samples were labeled with the column number, soil number, and the PV number that was expected at the time of sampling (for example, Sample C1-SC01-01 was labeled Column 1 containing soil SC01 and sampled at PV1). Selected samples, designated by the client, were sent to GEL for uranium analysis by inductively coupled plasma–mass spectrometry. Samples of the four waters were also sent for uranium analysis. Additional water samples (unacidified) were sent for anion analysis as specified by the client.

### Bottle Tests for Soil pH Measurements

To compensate for the oversight of not taking water measurements during the first 5 days of column operation, the client directed that 100 g dried soil plus 200 g water be placed into plastic bottles. The client adjusted the pH with dilute HCl to approximately the same pH as the original waters. The samples were intermittently shaken and allowed to settle before the pH of each was measured. Initially, the client took the pH measurements and made adjustments with HCl. Hazen took the final pH measurements, which are reported in Table 9.

**Table 9. Final Soil Bottle Test pH Measurements**

Bottle Test No.	Date and Time		
	3/14/2006 1648	3/15/2006 0630	3/17/2006 0915
	pH		
SC-01-TMW-02	7.93	7.22	7.04
SC-02-TMW-02	7.88	7.21	7.07
SC-02-02W33	7.38	7.1	7.03
SC-03-TMW-02	7.88	7.48	7.08
SC-04-TMW-09	7.42	7.51	7.43

## ADSORPTION STUDIES

The three sorbents were evaluated to determine their suitabilities for removing uranium from contaminated groundwater following the procedure shown on Pages 16 and 17 in Appendix C. The only exception to this procedure was the testing of three sorbents rather than two. The client was present during testing and directed the work.

### Bottle Tests

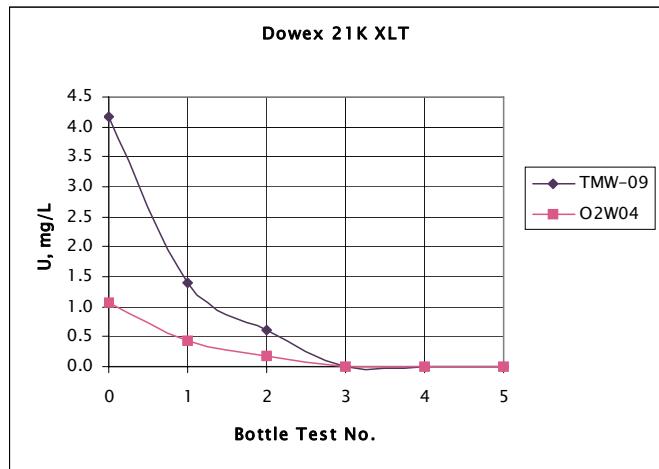
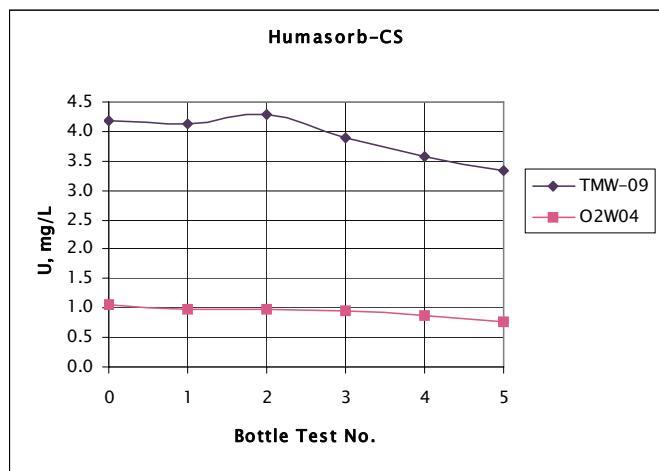
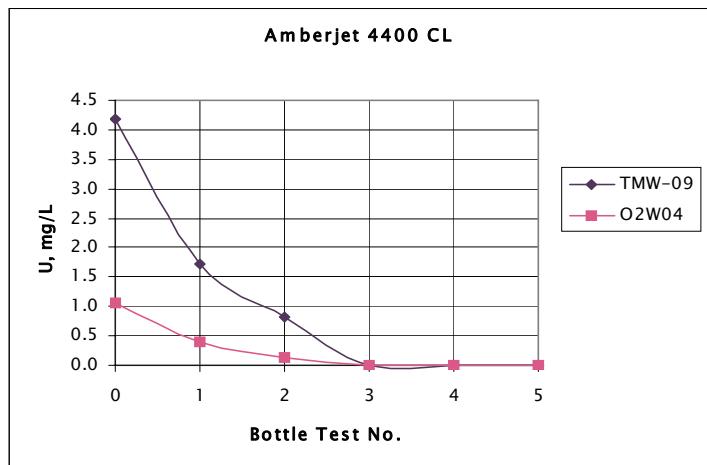
As-received Amberjet and Dowex resins were weighed and placed into 500-mL bottles. DI water was added to condition the resins. The excess water was drained, and test waters were added to a total volume of 400 cm<sup>3</sup>. The Humasorb was already wet. Samples were shaken for 30 min, then allowed to stand for about 3 hr. Waters were filtered with 0.45-µm filter paper, acidified with HNO<sub>3</sub> to pH 2, and sent to Hazen's laboratory for fluorometric uranium analysis. Data and analyses are shown in Table 10 and in Figures 5, 6, and 7. The data show that the two resins appeared to remove uranium to less-than-detectable levels, while the Humasorb removed very little uranium.

**Table 10. Bottle Test Results**

Book No.	Resin	Test Solution	Bottle Tare (with Lid), g	Resin Wt., g	Wt. DI H <sub>2</sub> O Added, g	Bottle Wt. + Resin Wt. (calc), g	Bottle Wt. after DI Removal, g	Wt. DI H <sub>2</sub> O Remaining in Bottle, g	Total Volume of Sample, mL	pH	Eh, <sup>a</sup> mV	Conductivity, mS/cm	U Analysis, <sup>b</sup> mg/L
2988-52-1	Amberjet	TMW-09	38.61	0.5136	19.94	39.12	40.93	1.81	400	6.90	589	0.963	1.72
2988-52-2	Amberjet	TMW-09	38.41	1.0050	20.58	39.42	41.72	2.31	400	6.87	590	1.000	0.83
2988-52-3	Amberjet	TMW-09	38.75	4.9860	20.98	43.74	48.94	5.20	400	6.56	589	1.093	0.0028
2988-52-4	Amberjet	TMW-09	38.37	10.0523	27.07	48.42	57.50	9.08	400	6.40	588	1.127	<0.0007
2988-52-5	Amberjet	TMW-09	38.62	20.0647	29.60	58.68	73.89	15.21	400	6.16	589	1.145	0.0017
2988-52-6	Amberjet	O2W04	38.86	0.5177	19.85	39.38	41.89	2.51	400	6.94	593	1.238	0.41
2988-52-7	Amberjet	O2W04	38.50	1.0040	20.00	39.50	43.55	4.05	400	6.87	594	1.282	0.14
2988-52-8	Amberjet	O2W04	39.53	5.0468	29.40	44.58	49.50	4.92	400	6.68	592	1.404	<0.0007
2988-52-9	Amberjet	O2W04	38.20	10.0106	29.84	48.21	55.92	7.71	400	6.55	593	1.452	<0.0007
2988-52-10	Amberjet	O2W04	38.24	20.0437	29.98	58.28	74.81	16.53	400	6.32	592	1.476	<0.0007
2988-52-11	Dowex	TMW-09	37.97	0.4976	20.30	38.47	42.41	3.94	400	6.82	589	0.955	1.40
2988-52-12	Dowex	TMW-09	38.44	0.9986	20.18	39.44	42.73	3.29	400	6.74	587	0.990	0.60
2988-52-13	Dowex	TMW-09	38.07	5.0547	20.07	43.12	49.90	6.78	400	6.51	587	1.081	0.0018
2988-52-14	Dowex	TMW-09	38.11	10.0061	29.96	48.12	54.71	6.59	400	6.36	586	1.123	<0.0007
2988-52-15	Dowex	TMW-09	38.00	20.0800	30.06	58.08	69.74	11.66	400	6.20	589	1.143	0.0009
2988-52-16	Dowex	O2W04	38.46	0.5004	20.12	38.96	41.36	2.40	400	6.84	588	1.227	0.42
2988-52-17	Dowex	O2W04	38.72	1.0097	20.03	39.73	42.65	2.92	400	6.82	588	1.274	0.18
2988-52-18	Dowex	O2W04	37.95	5.0245	20.07	42.97	47.22	4.25	400	6.63	589	1.392	<0.0007
2988-52-19	Dowex	O2W04	37.55	9.9903	29.92	47.54	54.88	7.34	400	6.52	589	1.441	<0.0007
2988-52-20	Dowex	O2W04	38.00	20.0415	30.15	58.04	69.28	11.24	400	6.38	588	1.433	0.0015
2988-52-21	Humasorb	TMW-09	38.96	2.00	--	--	--	--	400	6.96	588	1.019	4.14
2988-52-22	Humasorb	TMW-09	38.13	5.02	--	--	--	--	400	6.93	589	1.213	4.30
2988-52-23	Humasorb	TMW-09	37.94	9.94	--	--	--	--	400	6.93	590	1.487	3.89
2988-52-24	Humasorb	TMW-09	37.79	20.03	--	--	--	--	400	7.02	593	2.132	3.57
2988-52-25	Humasorb	TMW-09	38.25	39.86	--	--	--	--	400	7.20	595	3.270	3.33
2988-52-26	Humasorb	O2W04	37.62	2.01	--	--	--	--	400	6.93	592	1.290	0.97
2988-52-27	Humasorb	O2W04	38.04	5.01	--	--	--	--	400	7.01	593	1.484	0.98
2988-52-28	Humasorb	O2W04	37.96	10.03	--	--	--	--	400	7.08	595	1.793	0.95
2988-52-29	Humasorb	O2W04	38.34	19.94	--	--	--	--	400	7.10	596	2.386	0.87
2988-52-30	Humasorb	O2W04	38.32	40.17	--	--	--	--	400	7.31	596	3.470	0.78
2988-52-31	Blank	--	--	--	--	--	--	--	--	--	--	--	<0.0007
2988-52-32	Water Head	TMW-09	--	--	--	--	--	--	--	6.67	585	0.899	4.18
2988-52-33	Water Head	O2W04	--	--	--	--	--	--	--	6.66	587	1.169	1.07

<sup>a</sup>Eh determined using a platinum-silver, AgCl-saturated, KCl-filled combination electrode and adding 199 to the reading.

<sup>b</sup>Performed in Hazen's Analytical Laboratory by fluorometry.



**Figures 5, 6, and 7. Plots of Bottle Test Results**

### **Breakthrough Tests**

Using only the Dowex resin, two column breakthrough tests were performed following the procedure supplied by the client and shown on Pages 17–19 in Appendix C. Tests were performed in 2.5-cm-diameter by 30-cm-long chromatographic columns (VWR No. KT420830-3010) with 147-mL capacities and 20- $\mu\text{m}$  polyethylene bed supports. The resin was preconditioned by soaking in DI water for a minimum of 4 hr. Preconditioned resin was placed into each column to about 70% of its capacity (22.0 cm height), thus requiring about 75 g of as-received resin.

Table G1 in Appendix G shows data for the 10 min/PV test (Column 1), and Table G2 shows data for the 5 min/PV test (Column 2). Hazen's procedures consisted of measuring pH, conductivity, and temperature. The client selected samples that were sent to GEL for uranium analysis. Photographs of the columns are shown in Figures B11 and B12 in Appendix B.

Because of the sampling schedules, both columns could not be properly operated simultaneously; therefore, they were operated individually with around-the-clock attention. Column 1 was started, but after a few hours of operation, bubbles were noticed within the resin. The flow to the column was stopped, and a vacuum was applied to the top of the column to displace the bubbles, but without success. The client was present during this attempt. The column was then emptied and readied for an eventual restart. Column 2 testing occurred on March 15–17, 2006. Column 1 was then operated on March 20–24, 2006.

Both columns operated without incident during their durations. We noticed that Column 1 (lower flow rate) began to form a few small bubbles near the top of the resin about halfway through the test, and by the end of the test, several small bubbles were noticed in the top half of the column. Some of these bubbles are shown in the photograph in Figure B12 in Appendix B. No bubbles were observed in Column 2 (higher flow rate).

## **SUMMARY**

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No conclusions are presented for the test program. Hazen performed testing as directed by the client in both the scope of work and in verbal and e-mail communications, but analytical results from GEL were not made available to us.

**APPENDIX A**

**Standard Operating Procedures**

**Hazen Research, Inc.**  
**Standard Operating Procedures**

SOP No. 1      Sample receipt, drying, homogenization, sampling, and safety and health

SOP No. 2      Sample handling, disposition, data record keeping, and quality assurance

## **STANDARD OPERATING PROCEDURE NO. 1**

### **1.0 SAMPLE RECEIPT, INSPECTION, AND DOCUMENTATION**

- 1.1 Visually inspect the physical condition of the shipping package. Document any damage to the package or any material leakage. Sign the Chain of Custody document showing receipt of the sample if one is provided.
- 1.2 Determine the gross weight of the sample and record on the Hazen in-house inventory form. Log the sample in to show its receipt and generate a Hazen identification number.
- 1.3 Safely open the packaged soil and observe its physical condition.
- 1.4 Review the summary sheet listing the analytical tests required for soil characterization and obtain required sample jars for all tests

The following SOP applies to the initial characterization (bulk density determination, drying, homogenizing, and sampling) of soils.

### **2.0 BULK DENSITY, DRYING, HOMOGENIZATION AND SAMPLING**

For each bulk density sample (marked as BD)

- 2.1.1 Unseal each sample.
- 2.1.2 Measure its weight.
- 2.1.3 Measure its dimensions.
- 2.1.4 Dry at 110°C.
- 2.1.5 Reweigh and determine BD.

For each soil sample

- 2.2.1 Unseal each sample.
- 2.2.2 Measure its weight.
- 2.2.3 Drying at 60°C may be necessary (Dry under an N<sub>2</sub> cover, record initial and final weights, minimize exposure to air).

- 2.2.4 Thoroughly homogenize the sample.
- 2.2.5 Take an aliquot for residual moisture determination.
- 2.2.6 Take an aliquot for uranium analysis.
- 2.2.7 Seal dried solids under N<sub>2</sub> until used.
- 2.2.8 SC-01 will be used for Column Test No. 1.
- 2.2.9 SC-02 will be equally split and used for Column Test Nos. 2 and 3.
- 2.2.10 SC-03 will be used for Column Test No. 4.
- 2.2.11 SC-04 will be used for Column Test No. 5.
- 2.2.12 SC-05 and either SC-01, -02, -03, or -04 will be used for Column Test No. 6.

### **3.0 EQUIPMENT DECONTAMINATION**

- 3.1 Ensure that equipment surfaces are clean. Wash with hot detergent water, triple rinse with cold water prior to using.
- 3.2 Decontaminate equipment both before and after use.

### **4.0 SAFETY AND HEALTH**

- 4.1 Standard safety procedures will be followed for all project operations under the guidance of the Hazen Safety Plan and Chemical Hygiene Plan.
- 4.2 Project-specific safety requirements, including level of protection for operating personnel, monitoring of surrounding environment, and any other special requirements, will be addressed by the Hazen Safety Officer.
- 4.3 Hazen clientele or visiting personnel witnessing project operations are required to adhere to both the standard Hazen safety policies and those policies developed for the specific project.

## **STANDARD OPERATING PROCEDURE NO. 2**

The following SOP applies to sample handling, tracking, and disposition, data record keeping, and quality assurance.

### **1.0 SAMPLE HANDLING AND DISPOSITION**

- 1.1 Label all samples and residual test products with the time, date, and project number. Store unacidified liquid analytical samples in a refrigerator specified for samples at (4°C) until they are utilized. A common method used at Hazen for analytical sample tracking is to identify the sample by using the logbook number, page number, and sequence number on the page (for instance, 2988-45-1). This number is entered into the logbook along with a sample description. The number and description will follow the sample and analytical results throughout the project and will be included in reports.
- 1.2 Send the analytical samples to the specified analytical laboratory for required analyses. Samples will be packaged in a cooler and shipped by overnight delivery.
- 1.3 All unused soil, residual test products, cleanout material, and disposable safety equipment will be stored until the completion of the program report.
- 1.4 At the completion of program report and acceptance by client, dispose of the products from 1.3 in accordance with contractual arrangements.

### **2.0 DATA RECORDKEEPING**

- 2.1 A dedicated logbook will be maintained for the bench-scale testing. The logbook shall have, as a minimum, the following information:
  - 2.1.1 Organization to whom the book is assigned
  - 2.1.2 Book number
  - 2.1.3 Project number
  - 2.1.4 Start date
  - 2.1.5 End date

- 2.2 Each page will show the following information as a minimum:
- 2.2.1 Date of test
  - 2.2.2 Test information, including all observations, notes, calibrations, and sample collection data and numbers.
  - 2.2.3 Equipment used for testing
- 2.3 The entries into the logbook will be in ink, and corrections are to be single-lined out, initialed, and dated.

Following are the QA/QC procedures to be used during the bench-scale studies.

### **3.2 EQUIPMENT SPECIFICATIONS**

- 3.2.1 Measurement of pH
  - 3.2.1.1 Measurement of pH will be accomplished using a standard laboratory pH meter with a range of 0–14 and an accuracy of  $\pm 0.1$  pH. The procedure for measuring pH will follow Hazen's standard procedure, which is in accordance with ASTM Method D-1293, "pH of Water and Waste Water".
  - 3.2.1.2 The pH electrode will either be an epoxy body, permanent gel-filled combination electrode or a conventional AgCl-filled, glass combination electrode. Either electrode will be capable of measuring 0.1 pH units at a temperature range of 0–100°C.
  - 3.2.1.3 Buffer solutions will be capable of providing  $\pm 0.1$  pH accuracy at 25°C, as verified against NBS-certified buffers.
- 3.2.2 Measurement of Temperature

Temperatures will be measured using thermometers that conform to the test designations specified by the American Society of Testing Materials.
- 3.2.3 Measurement of Conductivity.

Conductivities will be measured using a conductivity meter accurate to 0.1 mS/cm.

**3.2.4 Measurement of oxidation-reduction potential (ORP).**

ORPs will be measured using a meter with a platinum–silver, AgCl-saturated, KCl-filled combination electrode. Eh will be determined by adding 199 to the reading.

**3.2.5 Glassware**

All glassware used during testing (beakers, graduated cylinders, etc.) will be either KIMAX or PYREX brand glass designed for general laboratory applications.

**3.2.6 Demineralized Water**

Water used for rinsing of pH electrodes, glassware, etc., will be treated by passing tap water through a mixed anionic–cationic resin bed in a demineralizer provided by U.S. Filter.

**4.0 CALIBRATION**

**4.1 Thermometers**

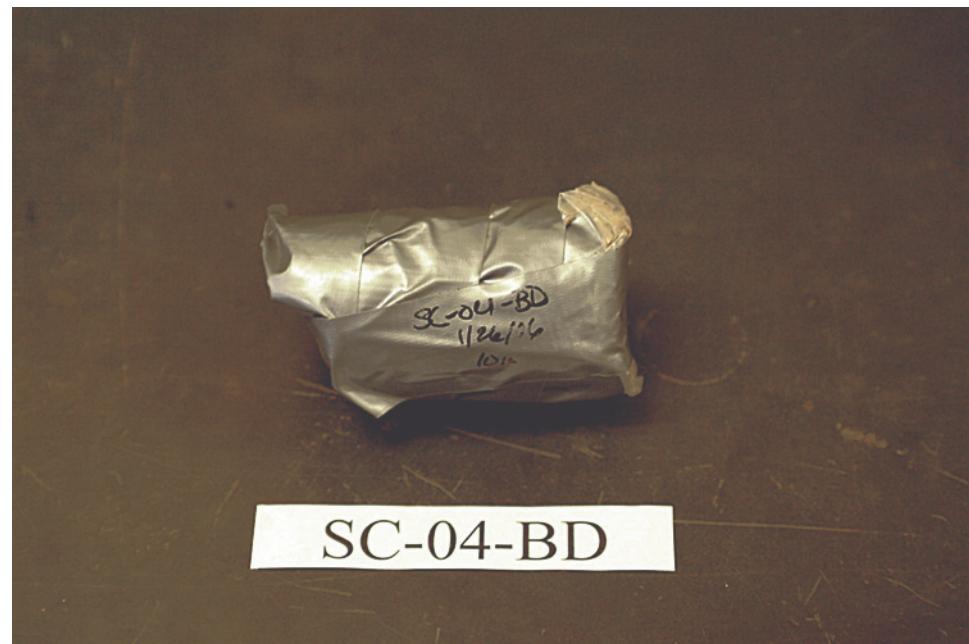
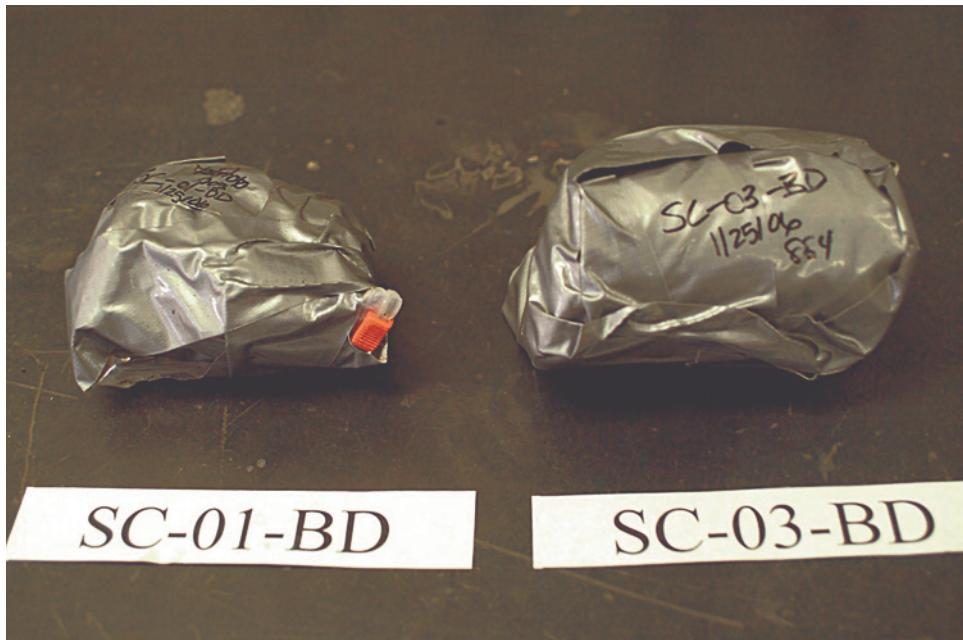
Temperatures will be measured using glass, general purpose, immersion thermometers that conform to the test designations specified by the American Society of Testing Materials.

**4.2 Balances**

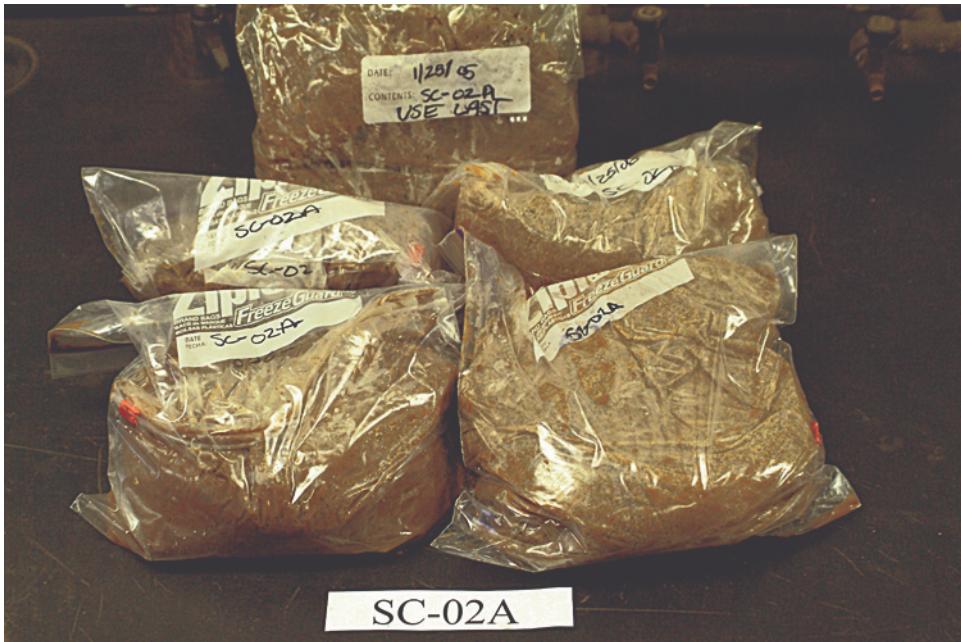
Balances are annually serviced by QA Balance Services, Inc., 7812 S. Newbern Cir., Aurora, Colorado.

## **APPENDIX B**

### **Photographs**



**Figures B1 and B2. As-received Samples for Bulk Density Determinations**



**Figures B3 and B4. As-received Soils SC-01 and SC-02A**



**Figures B5 and B6. As-received Soils SC-03 and SC-04**



**Figure B7. As-received Soil SC-05**



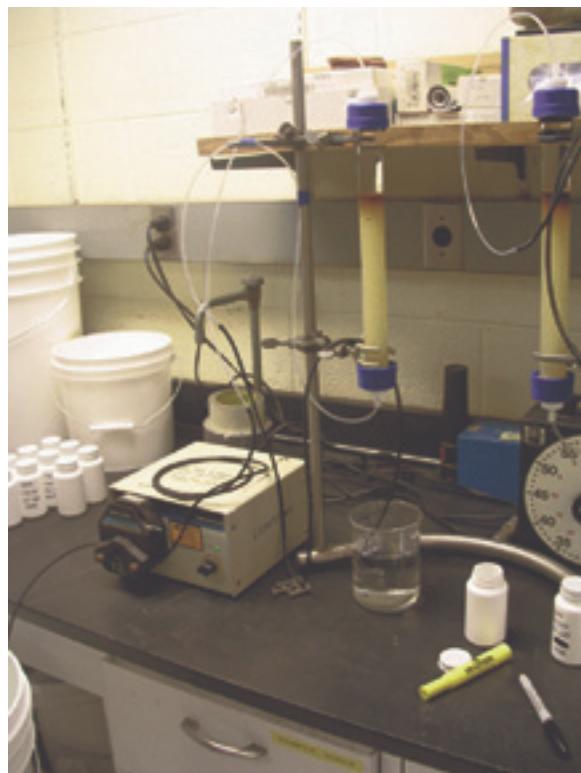
**Figure B8.** All Six Columns in Operation



**Figure B9.** Columns 1, 2, and 3 in Operation

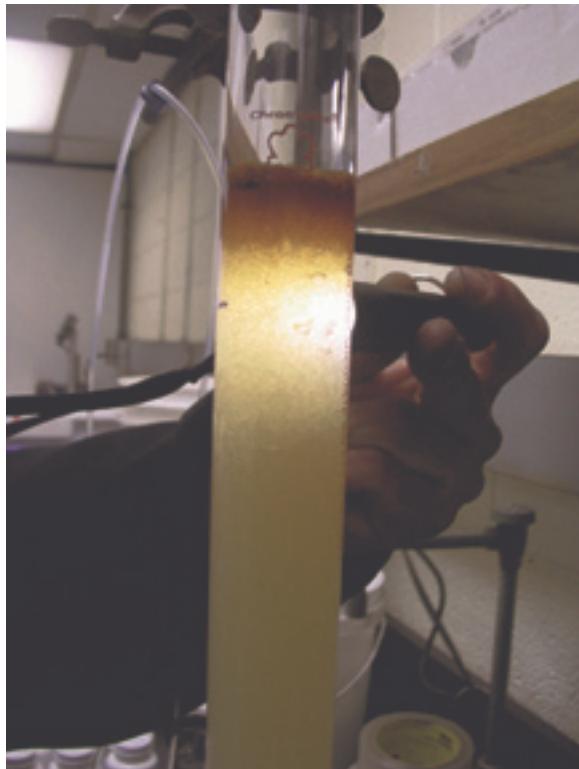


**Figure B10. Water Storage**



**Figure B11. Operating Breakthrough Column 1**

Column 1 (lower flow rate, 5 min/PV) on the left is shown during operation. The 5-gal bucket just visible at the lower left of the photo held the feed water. The adjustable-rate peristaltic pump fed water into the bottom of the column. Water discharged from the top of the column and was collected in the beaker located at the lower center of the photo. The columns were operated one at a time. The test with Column 2 (higher flow rate, 10 min/PV) on the right had already been completed.



**Figure B12. Completed Breakthrough Column 1 Showing Bubbles**

The first attempt at operating Column 1 (lower flow rate at 5 min/PV) was aborted because of bubbles forming in the midst of the resin. Attempts at clearing the bubbles by applying a vacuum to the column were not successful; therefore, the test was restarted. Some small bubbles, as seen in this photo, formed during this run, late in the run, and were located mostly near the top of the resin. The resins at the tops of both columns darkened during the course of the runs.