

CHARACTERIZATION AND ANION EXCHANGE REMOVAL OF  
URANIUM FROM HANFORD GROUND WATER

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ABSTRACT

In February 1985, uranium concentrations increased abruptly to 0.1 kgU/m<sup>3</sup> in ground waters underlying a retired liquid waste disposal facility in the United States Department of Energy-Richland Operations Hanford Site. Characterization tests showed the uranium was present as an anionic carbonate complex not sorbable by Hanford sediments. The uranium was mobilized by flow from a perched zone of water caused by recent nearby cooling water disposal above an impermeable sediment layer. In a unique demonstration of the concept of "as low as reasonably achievable," efforts were immediately undertaken to minimize the spread of the plume and to reduce the amount of uranium in the ground water. An anion exchange-based uranium removal process flowsheet was rapidly developed and implemented. Operational for six months, the process has treated over 30,000 m<sup>3</sup> of ground water and collected 94% of the uranium while producing a treated effluent that meets criteria for discharge to the soil column.

CHARACTERIZATION

Unconfined Hanford Site ground waters, lying in thick sedimentary beds of sand and gravel 60-70 m below ground surface, are routinely monitored for total alpha, total beta, tritium, nitrate, total uranium and gamma-emitter concentrations. Monitoring of the ground waters in the Separations Areas is the responsibility of Rockwell Hanford Operations (Rockwell). The uranium concentrations increased abruptly from earlier levels of about 10<sup>-4</sup> kgU/m<sup>3</sup> to about 10<sup>-1</sup> kgU/m<sup>3</sup> in two routine ground water samples collected in January 1985. Re-sampling and analyses of the ground water confirmed the initial analyses of approximately 3.1 million Bq<sup>238</sup>U/m<sup>3</sup> (85,000 pCi/L).

The ground water samples were taken from wells 299-W19-3 (W19-3) and 299-W19-11 (W19-11), which lie near the retired 216-U-1 and 216-U-2 (U-1,2) cribs (Fig. 1). The U-1,2 cribs, subsurface low- and intermediate-level liquid radioactive waste disposal facilities, had been operated from 1952 to 1967 and had received over 70 sediment column volumes of

aqueous waste containing an estimated 4,000 kg of uranium. Late in their service life, the U-1,2 cribs also received a small quantity of acidic decontamination waste containing fission and activation products. Operating nearby the U-1,2 cribs was the newly activated 216-U-16 (U-16) crib. The U-16 crib had received between 0.018 and 0.043 m<sup>3</sup>/s of cooling water during its service commencing in July 1984.

Immediately upon confirmation of the high uranium concentrations, water flow to the U-16 crib was stopped and existing wells near the U-1,2 cribs were grouted to preclude direct pathways between the contaminated sediments and the ground water. Existing wells included not only W19-3 and W19-11, but also an abandoned unused reverse well, 299-W19-9 (W19-9).

Ground Water Characterization

The compositions of natural and contaminated Hanford ground waters are presented in Table I.

TABLE I

Compositions of Hanford Ground Waters

Component	Concentration (mol/m <sup>3</sup> )			
	Natural	W19-2	W19-3	W19-11
Ca	0.5	1.9	10.3	11.8
Mg	0.3	1.0	5.4	6.2
Na	0.4	1.0	5.3	15.8
Si	0.5	0.8	0.8	0.9
HCO <sub>3</sub>	2.0	2.3	6.2	14.4
Cl	0.1	0.7	1.6	1.6
SO <sub>4</sub>	0.1	0.4	2.0	3.4
NO <sub>3</sub>	<0.0006	3.1	23.9	27.3
TOC* (kg/m <sup>3</sup> )	--	--	0.005	0.005
U (kg/m <sup>3</sup> )	<0.0005	<0.0005	0.013	0.087
pH	8.2	8.1	7.4	7.1

\*Total organic carbon.

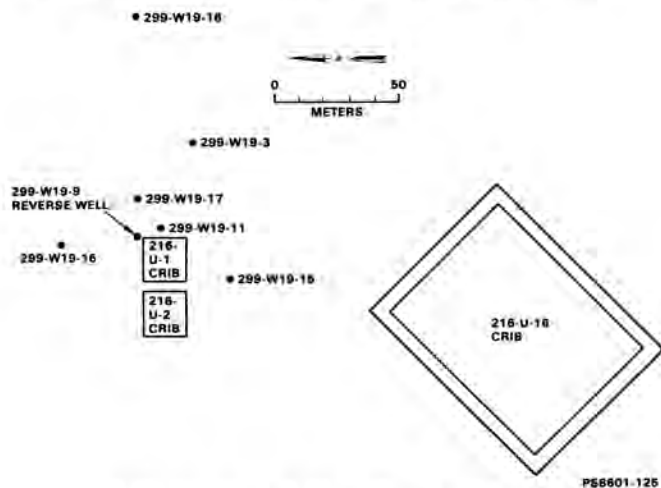


Fig. 1. Layout of Crib and Well Sites.

A Hanford-affected, but uranium-uncontaminated, ground water is represented by the W19-2 sample in Table I. The ionic strengths of the uranium-contaminated ground waters taken from the W19-3 and W19-11 wells in February 1985 were about 20 times higher and the pH values about one unit lower than found in the natural Hanford ground water. While the nitrate salts were the major contributors to the ionic strength in the W19-3 and W19-11 samples, bicarbonate concentrations also were significantly enhanced compared to the natural and W19-2 Hanford ground waters.

The concentration of uranium in the contaminated ground water was not diminished by passing through 0.003- $\mu$ m, pore-size ultrafilters; thus, the uranium was present as a dissolved species. Spectrophotometric analyses of both the W19-3 and W19-11 samples and comparison with published spectra<sup>1</sup> indicated that the uranium was presented principally as the  $UO_2(CO_3)_3^{4-}$  species. Thermodynamic calculations suggested that, at the ground water compositions given in Table I, a fraction of the uranium also may have been present as  $UO_2(CO_3)_2^{2-}$ .<sup>2</sup> The isotopic composition of the uranium in the W19-11 ground water was 99.322%  $^{238}U$  and 0.665%  $^{235}U$ .

#### Crib Sediment Characterization

The W19-11 well was drilled to a depth of 76 m in March and April 1983. Sediment samples taken during the well's construction were logged and archived, thus providing a profile of the contaminated sediment in the U-1,2 cribs between their retirement and the activation of the U-16 crib.

The uranium concentrations in the W19-11 sediments are presented in Fig. 2 as a function of depth. Though over 70 sediment column volumes of uranium-bearing waste solutions were disposed to the U-1,2 cribs, most uranium remained in a sharply defined band within approximately 8 m of the 6-m discharge depth. Uranium in the sediment peaked at approximately 36,000 mgU/kg sediment at 10.8 m. A uranium concentration peak of approximately 100 mgU/kg also was found at about 50 m. Uranium concentrations from that point dropped rapidly with

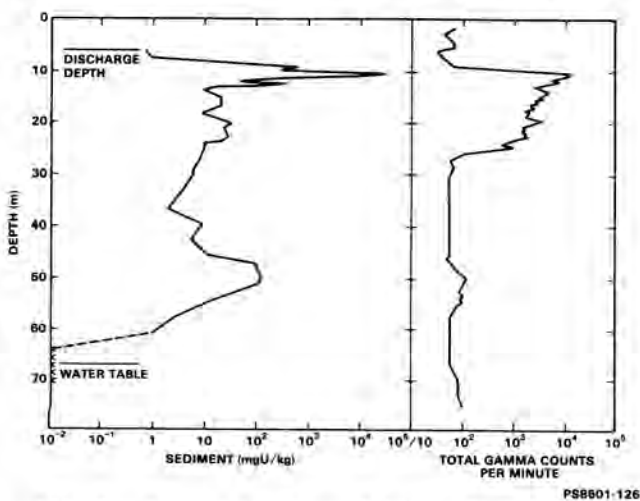


Fig. 2. Uranium and Gamma Activity Distribution in Well W19-11.

less than 0.01 mgU/kg found in samples taken near water table depth.

A down-hole gamma logging of W19-11 was performed in March 1985 (Fig. 2). This measurement showed a broadened profile compared to the uranium distribution. Like the uranium distribution, enhanced activity was found at about 50 m. In the gamma measurements, however, water table activity levels were higher than the activity in the sediment layers above.

Detailed chemical and physical characterization was performed to determine what mobilized the uranium that originally had remained high in the sediment column. Tests were run on the sediment sample taken from the 10.8-m depth with the following results: the sediment was acidic, a 17 wt% suspension of the sediment in synthetic ground water was pH 4.2, and the sediment contained 0.3 wt% phosphate. The sediment also contained approximately  $4 \times 10^3$  Bq  $^{60}Co$ ,  $3 \times 10^5$  Bq  $^{137}Cs$ , and  $3 \times 10^7$  Bq  $^{89,90}Sr$  per kg. The acid and radionuclides arose principally from decontamination wastes disposed in the early 1960s. The phosphate and uranium originated from wastes disposed during uranium recovery operations in the 1950s. Hanford sediments normally contain negligible phosphate.

Quartz, illite, and feldspar normally found in Hanford sediment as well as meta-autunite were identified in the 10.8-m depth sediment. Meta-autunite,  $Ca(UO_2)_2(PO_4)_2 \cdot 6H_2O$ , is a secondary mineral formed in nature from oxidation of primary ore materials such as uraninite ( $UO_2$ ) in the presence of phosphate, low concentrations of carbonate and intermediate pH.<sup>2</sup>

The isotopic composition of the uranium in the 10.8-m sample was 99.304%  $^{238}U$  and 0.674%  $^{235}U$ . The isotopic composition in a sediment sample taken from the 51-m depth of W19-11 was 99.330%  $^{238}U$  and 0.658%  $^{235}U$ .

#### Uranium Sorption and Desorption Tests with Hanford Sediments

Tests were conducted to determine if Hanford sediments sorb the dissolved uranium in the ground water. Sorption would retard uranium migration with respect to ground water movement and limit release to the accessible environment. Uranium from the contaminated ground water sorbed negligibly on uncontaminated Hanford sediments.

Uranium-free synthetic Hanford ground water similar in composition to the W19-3 and W19-11 samples was contacted with uranium-contaminated sediments collected during drilling of the W19-11 well. The 10.8-m depth sediment from W19-11 desorbed only 0.5 to 2.5% of its uranium on contact with the ground water; 60 to 75% of the uranium desorbed from sediments taken from the 47-, 51-, and 55-m depths of W19-11.

#### Drilling Tests and Sediment Stratigraphy

The sediment layers underlying the vicinity of the U-1,2 and U-16 cribs are extensive, uniform, show little dip and, in general, are unconsolidated and porous.<sup>4</sup> They consist (Fig. 3) of mixed sand, silt, and gravel layers extending to the underlying basalt at about 150-m depth. The water table lies at about 67 m while a thin noncontinuous caliche layer lies at 51 m. The caliche is a cemented,  $CaCO_3$ -rich crust, which is virtually impervious to water.

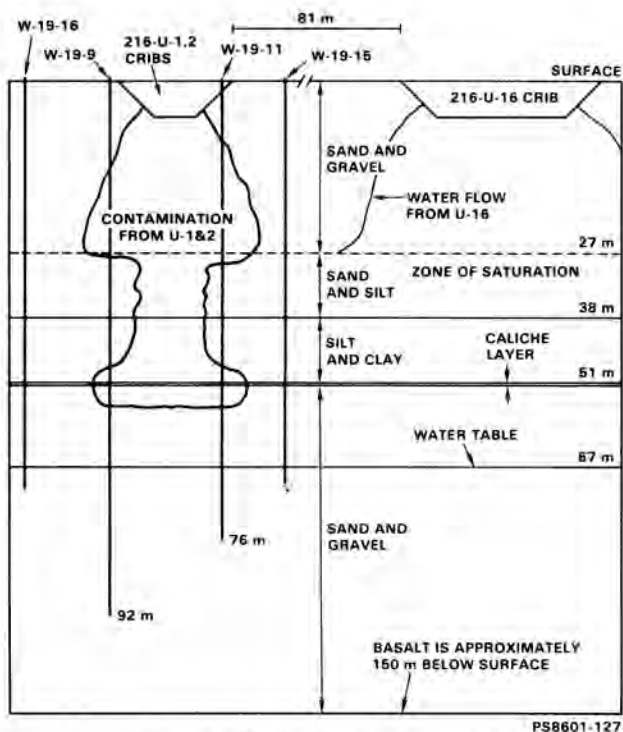


Fig. 3. Sediment Stratigraphy.

A study of exploratory wells W19-15, W19-16, W19-17, and W19-18 (Fig. 1) showed a zone of water perched on the caliche layer 16 m above the water table. The perched water extended 23, 21, and 8 m above the caliche layer for W19-15, -16 and -17, respectively. The W19-18 well, which hydrologically is located farthest down-gradient from the U-16 and U-1,2 cribs, showed no perched water. The perched waters were contaminated by uranium. The W19-16 waters, lying downstream of the U-1,2 cribs from the U-16 crib, contained as much as 0.05 kgU/m<sup>3</sup>. The W19-15 waters reached about 0.003 kgU/m<sup>3</sup>.

#### Mechanism of Uranium Transport

Data from the laboratory and field investigations were combined to deduce the likely mechanism for the presence of uranium in the ground water. Because of their large uranium inventory and their proximity to the contaminated ground water, the U-1,2 cribs were identified as the source of the uranium. The agreement in isotopic compositions of the uranium in the crib sediments and in the ground water corroborated this identification. Uranium in U-1,2 is distributed mostly near the original discharge level and owed its immobility during disposal of uranium recovery operations wastes to formation of low solubility phosphate phases such as meta-autunite. Later disposal of acidic decontamination wastes to U-1,2 partially dissolved the uranium-bearing phosphate precipitates. The low volume of the decontamination wastes, however, precluded significant transport of the dissolved uranium to the ground water. A zone of increased uranium concentration was deposited near the caliche layer at about 50-m depth. The acidic uranium solution encountered sufficient carbonate in passing through the sediment to convert the uranium to poorly sorbed anionic carbonate complexes.

In July 1984, the nearby U-16 crib began receiving cooling water discharges. The semi-

permeable caliche layer at 51-m depth partially intercepted drainage of the disposed solution and, instead, transported the liquid laterally at a perched level to contaminated sediments underlying U-1,2. The resulting uranium-contaminated water reached the ground water by draining through windows or well perforations in the caliche layer. The relatively low pH and high nitrate ion concentration of the contaminated ground water confirmed the involvement of the decontamination wastes, while spectrophotometric and sorption tests verified both the existence and mobility of the carbonate complexes.

Ground water flows north from the U-1,2 site about 11 km before emerging at springs near the Columbia River. Calculations performed by Battelle Pacific Northwest Laboratory, assuming negligible uranium sorption, showed dilution and dispersion would reduce <sup>238</sup>U concentrations to well below the 2.22x10<sup>4</sup> Bq/m<sup>3</sup> (600 pCi/L) guidelines<sup>4</sup> in the 200 to 600 years required for the plume to reach the Columbia River.

#### DEVELOPMENT OF AN ION EXCHANGE URANIUM REMOVAL TECHNIQUE

To minimize the time needed to institute a process for removal of uranium from the ground water, investigation was limited to proven processes for uranium removal from aqueous streams using available or easily procured equipment. Review of technical literature showed anion exchange to be the most desirable approach for removal of the UO<sub>2</sub>(CO<sub>3</sub>)<sub>3</sub><sup>4-</sup> from the ground water.

#### Batch Contact Screening Tests

Three batches of actual ground water (Table II) were used during testing. Batch contacts of Feed 1 were made with five strong-base anion exchange resins: Dowex MSA-1, Dowex SBR-C, Dowex 1-X2 (Dow Chemical Co.) and Amberlite IRA 900 and IRA 904 (Rohm and Haas Co.).

TABLE II

Compositions of Ground Water Feeds Used for Ion Exchange Tests

Component	Concentration (mol/m <sup>3</sup> )		
	Feed 1	Feed 2	Feed 3
Ca	20.3	7.2	3.7
Mg	10.3	3.7	1.7
Na	--	13.1	10.8
HCO <sub>3</sub>	11.7	12.0	9.0
SO <sub>4</sub>	3.4	3.0	--
NO <sub>3</sub>	22.8	18.3	--
U (kg/m <sup>3</sup> )	0.101	0.0881	0.0246

Comparison of uranium removal efficiency was made by means of Freundlich plots (Fig. 4). Assuming that the Freundlich equilibrium relationship holds over the range of concentrations measured, extrapolation of the plots to feed composition allows estimation of the capacity of the resin to sorb uranium at column exhaustion. The projected capacities were 300 CV (column volumes) for Amberlite IRA 900 and 9 CV for IRA 904, while Dowex 1-X2 gave 53 CV, MSA-1 gave 30 CV and SBR-C, 17 CV. Since Dowex 1-X2 was no longer commercially available, Amberlite IRA 900 and Dowex MSA-1 were selected for further testing.

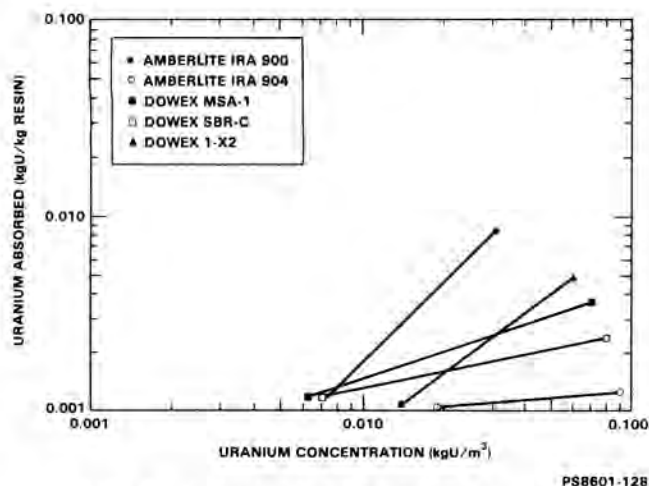


Fig. 4. Freundlich Plots for Resin Batch Column Tests

Column tests were conducted to optimize the performance of the two candidate resins. The tests showed flow rate had little effect on loading while prewashing the resin with  $(\text{NH}_4)_2\text{CO}_3$  solution improved capacity slightly.

A marked improvement in resin capacity was found when the feed was treated with  $(\text{NH}_4)_2\text{CO}_3$  (Feed 2, Table II). The treated feed, clarified of the precipitated  $\text{CaCO}_3$  and  $\text{MgCO}_3$ , was passed through a column of Dowex MSA-1. At 450 CV throughput, breakthrough was less than 0.02%. The performance of the Dowex MSA-1 exceeded preliminary engineering goals and thus was selected for scaleup testing.

#### Development of Operating Parameters for Scaleup to Production

Laboratory experiments were designed to define the chemical solutions and the operating parameters for an ion exchange system using Dowex MSA-1 resin. Experiments were aimed towards defining conditions to minimize regeneration volumes and to ensure compatibility with chemicals used in other Hanford Site waste streams.

A 1.5-cm diameter column was used for all experimentation. In most runs, the column was loaded with approximately 500 CV of feed to simulate the partial uranium loading expected in a production-scale column. In one experiment, 750 CV of feed were passed through the resin bed. The final effluent uranium concentration was 20% of the feed corresponding to a loading of 0.068 kg U per kg of resin, comparable to the loadings reported for uranium mining operations.<sup>5</sup>

Column tests were conducted comparing  $\text{Na}_2\text{CO}_3$  and  $(\text{NH}_4)_2\text{CO}_3$  feed pretreatment. Sodium carbonate was more suitable for disposal of the uranium-depleted effluent to a crib, was easier to prepare using available equipment, and formed a faster-settling precipitate less susceptible to postprecipitation with no loss of column-loading efficiency.

Both 2 kmol/m<sup>3</sup>  $(\text{NH}_4)_2\text{CO}_3$  and 2 kmol/m<sup>3</sup>  $\text{NaNO}_3$  successfully eluted sorbed uranium. Ammonium carbonate solutions are used as uranium eluants in the mining industry. However,  $\text{NaNO}_3$  is more suitable for disposal with the uranium into Hanford underground waste storage tanks. With a  $\text{NaNO}_3$  eluant, downflow regeneration is essentially complete after 6 CV; upflow (counterflow) regeneration is complete in less than 4 CV.

Seven complete load and regeneration cycles were completed on the laboratory ion exchange column with no loss of uranium-removal efficiency and no fouling. No change in loading efficiency was noted when the feed flow rate was doubled.

For disposal of the uranium-bearing regeneration solution into the waste tanks, the pH of the solution had to be adjusted to greater than 12. At high pH, uranium precipitates from solution as  $\text{Na}_2\text{U}_2\text{O}_7$ . Laboratory studies characterizing the nature of the slurry produced from pH adjustment of the regenerant stream included determination of total percent solids in the slurry, settling times, abrasiveness of the solids, and viscosity (pumpability) of the slurry.

Difficulties were encountered in plant operation due to carbonate pretreatment of the feed. Analysis of the ground water feed when plant-scale ion exchange operations started showed that the uranium concentration was much lower (Feed 3, Table III) with little change in the carbonate concentration. A single batch-contact test indicated that sufficient capacity might be achievable without pretreatment. An additional laboratory column run was made using Feed 3. Though over 400 CV of untreated feed were pumped through the column, effluent uranium concentration remained below 0.5% of the feed concentration. Column efficiency appeared to be somewhat less than for pretreated feed, but acceptable column lifetime in the plant-scale equipment was expected.

#### PROCESS FLOW AND EQUIPMENT

Bench-scale test results were used to design the plant-scale anion exchange process to treat the uranium-contaminated ground water (Fig. 5). Ground water is pumped 800 m overland from the W19-9 well to a surge tank located outside the ion exchange facility building. The 180-m<sup>3</sup> surge tank provides a 12-hour residence time within high and low operating limits. To comply with environmental discharge standards, the tank breathes through a high efficiency particle air (HEPA) filter to trap uranium entrained in droplets. The surge tank is set in a concrete basin to collect possible overflow and has a sump pump to return water to the tank when the tank level drops. A pressure sensor installed on the feed line alarms should low line pressure occur, indicating either a well pump failure or a line failure between the well and the surge tank.

#### Feed Pumps and Filters

Two 0.075-m<sup>3</sup>/min pumps are used in parallel to deliver about 0.13 m<sup>3</sup> of ground water per minute to the ion exchange column from the surge tank. Low-level sensors and flowmeters are used to control pump

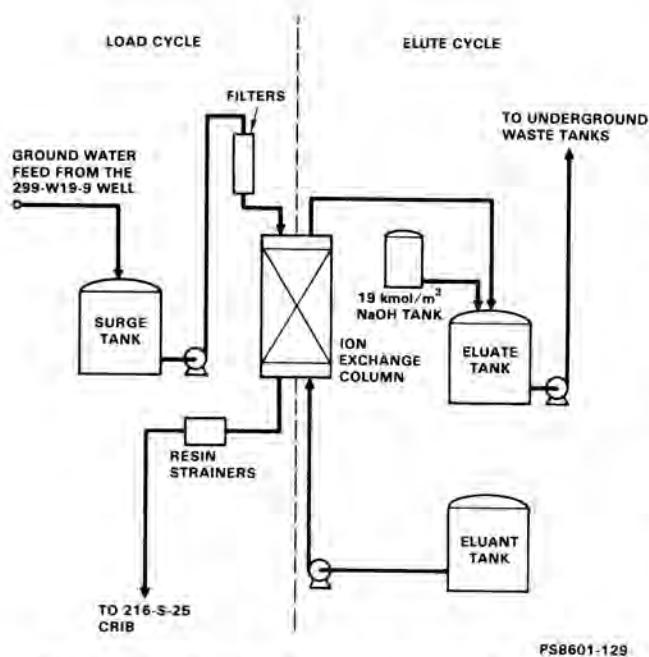


Fig. 5. Uranium Ion Exchange Process Equipment.

operation and flow rates. The ground water is filtered through a bank of fifty 25- $\mu\text{m}$  filter cartridges prior to entering the ion exchange column.

#### Ion Exchange Column and Associated Equipment

The filtered ground water enters the top of the ion exchange column. The column contains about 2.8  $\text{m}^3$  of Dowex MSA-1 resin supported by a 0.15-mm screen. Pressure gauges installed on the inlet and outlet of the column monitor pressure drop across the resin bed. The decontaminated ground water passes through 0.07-mm strainers to collect resin which might escape through the bottom screen. A back pressure of 20 kPa is maintained on the column to prevent gas buildup within the column. The decontaminated ground water is sampled every 8 hours, analyzed for uranium, and discharged to the 216-S-25 (S-25) crib. A flowmeter is located downstream to monitor ion exchange column flow.

#### Eluant and Eluate Tanks

The ion exchange resin is regenerated with 11.4  $\text{m}^3$  of 2  $\text{kmol}/\text{m}^3$   $\text{NaNO}_3$  solution. The eluant is held in the steam-jacketed tank. To elute the uranium and regenerate the resin, the  $\text{NaNO}_3$  is pumped upflow at about 0.23  $\text{m}^3/\text{min}$ . Analyses of the eluate taken during regeneration are used with feed and outlet uranium concentrations at loading to verify the total uranium balance. The eluate is collected in a 67- $\text{m}^3$  tank and is adjusted to pH ~12 by addition of 19  $\text{kmol}/\text{m}^3$   $\text{NaOH}$ .

#### Process Performance

Pretreatment of the ground water with carbonate was deemed necessary to achieve high-resin loading. This treatment was abandoned because of operating problems. In the carbonate pretreatment, a 2  $\text{kmol}/\text{m}^3$   $\text{Na}_2\text{CO}_3$  solution was injected into the feed line of the surge tank. Precipitation of the  $\text{CaCO}_3$  and  $\text{MgCO}_3$  occurred inside the surge tank. A 12-hour residence

time allowed for settling of solids. However, after a few weeks of operation, the feed pump plugged. The 5- $\mu\text{m}$  filters upstream of the ion exchange columns also began plugging every four hours. Larger (25  $\mu\text{m}$ ) filters were installed, but the ion exchange column then began fouling as high differential pressures developed across the resin bed. The filter changeout required disassembly and removal of the spent filters for disposal. The frequency of changeout required full-time support and the incurred costs from this and pump replacements were unacceptable.

Accordingly, the carbonate pretreatment was discontinued. The cycle length and uranium removal efficiency attained without pretreatment are acceptable and are attributed to lower feed uranium concentrations than used in the laboratory tests.

Feed and effluent uranium concentrations are analyzed daily to monitor breakthrough behavior. Since the breakthrough is gradual (5-7 days), the need for regeneration can be anticipated. A uranium removal efficiency of 96% was achieved early in the first few cycles. An operating set point of 80% removal efficiency is used as a basis for halting flow to the column and regenerating the resin. The resulting average removal efficiency for the first few cycles was 93%. Allowing partial breakthrough reduced the overall efficiency but also reduced the volume of waste generated through resin regeneration and subsequent water flushing. The removal efficiency increased to above 99% after eight cycles. The overall removal rate through eight cycles is 94%. The average amount of uranium recovered per cycle was 86 kg, while uranium concentration in the eluate varied between 2 and 30  $\text{kgU}/\text{m}^3$ . The uranium loading on the resin was approximately 0.035  $\text{kgU}/\text{kg}$  resin.

The cycle length is the period of operation between resin regenerations. The feed concentration decrease from 0.058 to 0.016  $\text{kgU}/\text{m}^3$  in the first eight cycles directly affected the cycle length (Fig. 6). As the feed concentration decreased, the cycle length increased from 10 days to 38 days. The longer cycle length is beneficial in that it reduces the volume of waste generated per volume of ground water treated and increases the overall removal efficiency of the column.

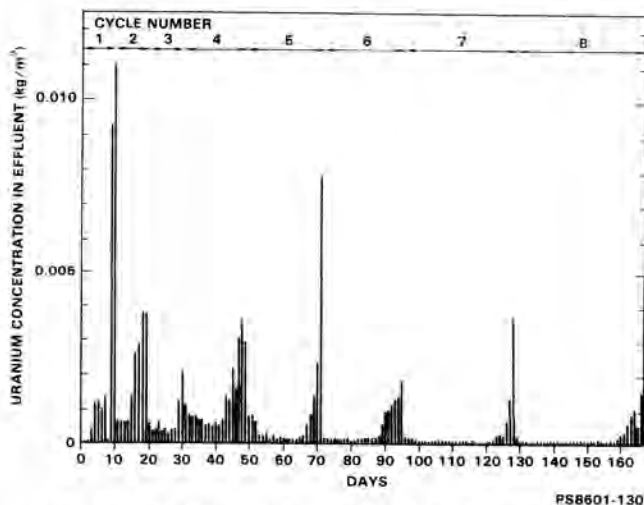


Fig. 6. Uranium Concentration in Column Effluent and Column Cycle Length.

The limit on  $^{238}\text{U}$  discharged into a controlled area is set by Rockwell internal guidelines and is  $22,200 \text{ Bq/m}^3$  (600 pCi/L) for liquid effluents. The  $^{238}\text{U}$  discharged to the S-25 crib varied between  $2,400$  and  $61,400 \text{ Bq/m}^3$  ( $0.0002$  and  $0.005 \text{ kgU/m}^3$ ) and averaged  $21,000 \text{ Bq/m}^3$ . Other uranium isotopes,  $^{234}\text{U}$  and  $^{235}\text{U}$ , also were present but were well below discharge limits.

The volume of waste generated during resin regeneration is about  $16 \text{ m}^3$  per cycle. This includes 4 CV of  $\text{NaNO}_3$  eluant and 2 CV subsequent water flushing. Resin loss during the regeneration is minimal. The volume of waste generated per volume of ground water treated decreased as the cycle length increased. The first cycle lasted 10 days and waste volume was 1.4% of the feed volume. These figures changed to 0.4% in the 38-day eighth cycle.

The waste eluate is neutralized with  $19 \text{ kmol/m}^3$  NaOH solution before pumping to an underground waste tank. The volume of NaOH required to neutralize the solution was 5.7% of the waste volume.

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