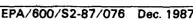
Research and Development





Project Summary

Removal of Uranium from Drinking Water by Ion Exchange and Chemical Clarification

Steven W. Hanson, Donald B. Wilson, Naren N. Gunaji, and Steven W. Hathaway

A pilot demonstration was conducted of ion exchange and chemical clarification equipment for removing uranium from drinking water. Four commercial-type ion exchange columns and a prefiltering and regeneration solution system were constructed along with a pilot-scale chemical clarification unit. These units were assembled and installed in a van trailer for location at a well site containing uranium-contaminated water. Uranium concentrations in the well varied during the study period from 190 to 400 µg/L.

The four ion exchange columns each contained 2 ft³ of resin. Three different ion exchange resins were used. The 1-gal/min chemical clarification unit consisted of a rapid-mix tank and precoat rotary vacuum filter. The unit was operated continuously for 3 months at pH values of 6 to 10 and ferric chloride concentrations of 15 to 40 mg/L. Greater than 99% removal of uranium was achieved when operating at 30 mg/L ferric chloride and pH 10. The diatomaceous earth precoat filter achieved complete solid-liquid separation.

In addition to the pilot study, the report analyzes several currently operating water treatment systems whose feed supplies contain uranium. Cost analysis data for capital equipment is also included in the report along with a discussion of ultimate disposal methods for uranium-containing water treatment wastes.

This Project Summary was developed by EPA's Water Engineering

Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Background

Most drinking water supplies come from groundwater. The quality of these supplies depends on the mineral and biological content, which vary widely from source to source. Nearly all supplies contain some small quantity of uranium. In most drinking water supplies, the average uranium concentration is 3 µg/L (equivalent to 2.0 pCi/L uranium), but a large number of community supplies have uranium concentrations in the 10to 50-µg/L uranium range. Though no federal regulations currently exist setting a maximum contaminant level (MCL) for uranium in drinking water, the U.S. Environmental Protection Agency (EPA) is evaluating a limit of 15 μ g/L (approximately 10 pCi/L). That evaluation involves the assessment of available control (removal) technology for uranium and the economic impact of such an MCL.

Aqueous solutions of uranium salts have an acid reaction as a result of hydrolysis, which increases in the order $U^{3+} < UO_2^{2+} < U^{4+}$. The uranyl, UO^{2+} , and U^{4+} solutions are well studied. The main hydrolyzed species of UO_2^{2+} at 25°C are UO_2OH^+ , $(UO_2)_2(OH)_2^{2+}$, and $(UO_2)_3(OH)_5^{1+}$; but the system is a complex one, and the

species present depend on the medium. In addition to these hydrolytic products, uranium ions can undergo complexing reactions with all ions other than C104.

Figure 1 shows the pH dependency of the uranyl ion in aqueous solution in the presence of carbonate, a constituent in most drinking water supplies.

Previous work at EPA's Oak Ridge National Laboratory at Los Alamos National Laboratory had shown that ion exchange and chemical clarification were effective treatment methods of removing uranium species from aqueous solutions.

Scope of Work

This report presents the results of a demonstration of pilot-plant ion exchange and chemical clarification equipment for the removal of uranium from drinking water. The test units were housed in a 40-ft van trailer (Figure 2) and located at a uranium-contaminated groundwater well on the New Mexico State University campus. The well had not been used for a long period and was returned to service for this project. The uranium concentration in the water varied over the project, begining with 190 μ g/L, rising to 350 to 400 μ g/L during the project, and falling back to 200 μ g/L at the end. These levels greatly exceeded the tentative study level of 15 μ g/L(~10 pCi/L).

Four ion exchange columns were constructed, each containing 2 ft³ of resin. Three different ion exchange resins were used: DOWEX SBRP. DOWEX 21 K, and IONAC A641.* Three columns were operated in the conventional down-flow mode, and the fourth column was operated with upward flow of the feed water. Pretreatment consisted of particulate filtering only. Regeneration was by chloride ion (10% NACL). Four bed-volumes of chloride solution were used at one-third the normal flow rate followed by two bed-volumes of rinse (product water). Resin capacity was represented by 12,000 to 20,000 bed volumes. Four cycles of operation were completed for each column, processing a total of approximately 4 million gal of feed containing an average uranium concentration of 300 μ g/L.

The chemical clarification unit consisted of a Joy Manufacturing Co. Model 0-12 Laboratory Sub-A Flotation Cell as a rapid-mix vessel and a continuous

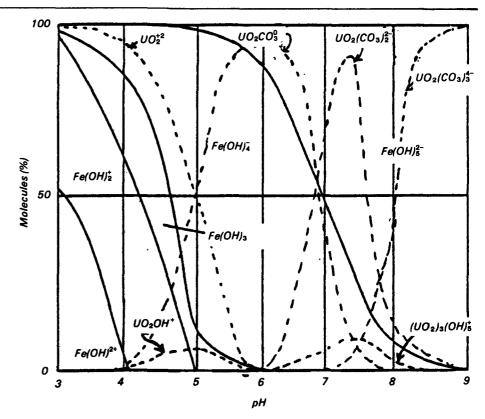


Figure 1. Uranium species in aqueous solution at various pH levels.

- 1. Water Supply
- 2. Feed Pump
- 3. Pre-Filter
- 4. Chemical Clarification System
- 5. Four Unit Ion Exchange Columns
- 6. Ion Exchange Control System
- 7. Product Pump
- 8. Product Tank
- 9. Product Discharge
- 10. Regenreation Solution
- 11. Regeneration
- 12. Waste Discharge
- 13. Pilot-Scale Up Flow Unit

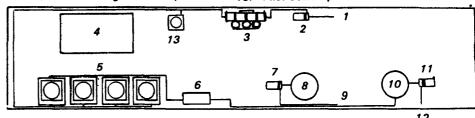


Figure 2. Organization of van system.

precoat rotary vacuum filter. The system flow rate was 1 gal/min. This system was operated continuously over a period of 3 months using pH values of 6 to 10.0 and ferric chloride concentrations of 15 to 40 mg/L. Greater than 99% removal of uranium was achieved when operating

at 30 mg/L ferric chloride and pH 10.0. The diatomaceous earth precoat filter achieved complete solid-liquid separation.

The full report also analyzes several currently operating water treatment systems whose feed supplies contain

^{*}Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

uranium. In addition, cost analysis data for capital equipment is included along with a discussion of ultimate disposal methods for uranium-containing water treatment wastes.

Demonstration Test Program

Table 1 outlines the quality of the well water used in this project. The major constituent of interest was the uranium. At the start of the project, the uranium concentration was 190 µg/L. During the project, the uranium concentration rose to 450 μ g/L but returned to around 200 μg/Lat the end of the project period. Both the ion exchange units and the chemical clarification units were operated on a continuous basis. Initially, sampling for uranium was performed daily. Later, it was done weekly until breakthrough was anticipated, and then daily samples were taken. The chemical clarification effluent was sampled on a daily basis until steady state was reached (after a change in operating conditions); then it was sampled every other day.

Ion Exchange

Water treatment using ion exchange technology is a well established operation. Table 2 summarizes the operation of the four ion exchange units. Though the feed to each unit was adjusted individually and a uniform flow through all columns was attempted, it was not achieved. Each column effluent was sampled individually, but when one column reached breakthrough, all four were regenerated. Thus throughout the four cycles, some units had not been processed to exhaustion.

All four systems operated well once the problems of mechanical equipment had been corrected.

Several physical and chemical characteristics of the resins need to be considered in the selection of ion exchange as a water treatment technology. Physical bead breakage may occur in some applications. For downflow systems, this breakage results in increased pressure drop and reduced efficiency. The three downflow units exhibited no resin breakage. In an upflow unit, breakage could result in entrainment of broken resin in the effluent and subsequent downstream problems in addition to the loss of exchange capacity through loss of resin. The upflow unit exhibited no resin breakage. These determinations were based on operating pressure drop and visual inspection of the resin at the end of the project.

Thermal stability of the resins is a second consideration in selecting ion exchange. Weak-base resins are usable up to 212 °F, followed by Type I strong bases to 122°F (the resins of this demonstration). Temperatures in the van housing the units ranged from a winter low of 35°F to a summer high of 115°F. The average inlet (well temperature) was 65°F. No noticeable change in operation occurred as a result of thermal effects.

Although chemical degradation is not normally a problem in most water

treatment applications, strong oxidizing agents can rapidly degrade the polymer matrix and should be avoided. Slower degradation with oxygen may be catalytically induced, so ionic iron, manganese, and copper should be minimized. In the present operations, the raw water had high iron and manganese contents (4 mg/L and 1.1 mg/L, respectively). Resin degradation was not observed.

The capacity of the ion exchange resin is the measure of ionic attraction per volume, and it is expressed in a number of ways. Total capacity is the theoretical measure of the total number of exchange

Table 1. Chemical Composition of Well Water* Used in Demonstration (mg/L, except as otherwise noted)

Component	Concentration (mg/L)	Component	Concentration (mg/L)
Ū	0.30±0.10*	Cd²⁺	<0.005
Na ⁺	17.9	Cr ³⁺	<0.01
K ⁺	18.4	Pb ²⁺	<0.005
Ca ²⁺	375.2	Hg²⁺	0.0004
Mg²⁺	69.0	SE ^{2~}	0.005
CI ⁻	555.2	Ag ⁻	<0.05
CO3 ²⁻	o	NO ₃ -	0.01
HCO₃¯	96.4	F	0.54
SO4 ²⁻	400.0	Fe ³⁺	1.69
7DS	2152	MN²⁺	0.95
AS*+	0.005	Hardness (as CaCO₃)	1220
BA ²⁺	0.12	Alkalinity	79
		ρΗ	7.62
		Electrical conductivity mmhos/cm	2.36

Notes.

Table 2. Operation Summary for the Demonstration Ion Exchange Units*

Resin Type	Flow Condition	Total Gallons Processed	Number of Cycles	Average Bed-Volumes Treated/Cycle	Maximum Removal Efficiency (%)
DOWEX 21K	Down	797,540	4	12920	99
DOWEX SBR-P	Down	890,600	4	14925	99
IONAC A-641	Down	853,870	4	16040	99
IONAC A-641	Up	903,450	4	15370	99

^{*}Inlet water concentration ranged between 200 and 400 µg/L uranyl complex (as uranium).

^{*}Uranium concentration in the well water varied and ranged from 200 to 450 µg/L; pH of well water was 7.62; electrical conductivity of well water was 2.36 mmhos/cm.

sites available and is normally calculated in three different ways: Dry weight capacity (meq/dry g), wet volume capacity (meq/wet g) and wet volume capacity (meq/mL). Table 3 gives the average capacities for the resins used in this operation.

Regardless of how it is expressed, operating capacity is the most realistic performance measure for ion exchange resins, and in water treatment, it is usually expressed as kilograins/ft³ of resin. Here the resin is measured in a column for actual operation under prescribed conditions.

A final consideration in the selection of an ion exchange resin is the cost of regeneration. Small differences in efficiency will be magnified over the life of the system. Perhaps of equal importance are the potential environmental problems encountered in disposing of waste regenerant.

This demonstration showed that chloride ion (NaCl) successfully regenerated all resins used.

Chemical Clarification

. The flotation cell was used as the rapid mixer and adsorption vessel. Entrained air only was used—that is, no forced-air mixing was used, and no surfactants were used to enhance bubble stability of flocculation. This vessel had a 1-min residence time at a 1-gpm feed rate. The effluent was channeled by gravity flow to the filter vessel, where a continuous rotary vacuum filter separated the ferric hydroxide precipitate.

The filter membrane was 0.45-micron polymer mesh, which allowed precipitate to bleed through into the filtrate when operated without precoating. A 0.25-in. precoat of diatomaceous earth was used and gave a complete solid-liquid separation as measured by our analytical procedures. Additional diatomaceous

earth (12% by weight) was added to the filter feed and mixed by a mechanical agitator with the feed slurry. A stationary knife removed the cake buildup. Visual inspection of residual precoat showed no binding by the ferric hydroxide precipitate. Sampling of the precoat material after a run for uranium showed that all uranium removed was adsorbed on the ferric hydroxide and not on the diatomaceous earth.

Table 4 summarizes the effect of pH on removal of uranium from the well water at a constant ferric chloride addition of 30 mg/L.

Table 5 shows how varying the ferric chloride concentration affects uranium removal efficiencies at pH 6 and 10.

Experimental data for the effect of the ferric chloride dosage and the equilibration pH of the solution show that pH is a major controlling factor in the removal of uranium from drinking water by means of the coagulation/filtration process. Proper choice of pH is a requirement for effective chemical coagulant treatment. The results of the pH dependence for uranium removal with iron salt can be interpreted by the stability and charge characteristics of uranyl species and metal hydroxide precipitates at the adjusted pH of the solution. No data are shown for similar experiments examining the pH dependencies for other dosages of ferric chloride. However, similar results can be expected from the physiochemical properties of the metal hydroxide formed during the coagulation process at a given pH and the dominant uranyl species in the solution. The role of ferric hydroxide as a coagulant formed from ferric chloride in aqueous solution is well known. The stability, solubility, and reactivity (adsorption) of the hydroxide are pH-dependent. The pH dependence of the distribution of uranyl species in natural water is also known. Dominant uranyl species and charge characteristics of iron hydroxide floc at pH 4, 6, 8, and 10 are shown in Figure 1. At a low pH (say pH < 5), ferric hydroxide had a positive charge. Fe(OH)3-w and the uranyl carbonate complex also dissociated to a positively charged uranyl ion (UO2+, UO2OH*). Thus at pH 4, less adsorption can occur because of the strong repulsion between ferric hydroxide and uranyl ion or ion-complex. As the pH was increased to 6, the dominant uranyl complex would be UO₂CO₃, with a small positive charge of uranyl ions, and the mixed charge of negative and neutral hydrolyzed ferric iron would be overall negative. Statistically, the possibility exists for adsorption, since at least there is no repulsion between the two charged particles, UO₂CO₃ and Fe(OH)4. At pH 8, the same phenomenon was observed as at pH 4, except that both interacting species would have negative charges. Thus a lower removal efficiency was obtained in both conditions (pH 4 and 8). When the pH exceeds 9.5, the UO₂(CO₃)4 species are known to be stable; but (UO₂)₃(OH)[†]₅ would be the dominant species in carbonate-depleted water. The carbonate in water could be depleted by CaCO₃ precipitation during the coagulant treatment process. The reaction is shown as follows:

3 UO₂(CO₃)₄⁴ + 15 Ca²⁺ + 2 FeCl₃ + 15 OH⁻ → (UO₂)₃(OH)₅⁺ + 12 CaCO₃(s) + 2 Fe(OH)₅²⁻ (s) + CaCl₂

Since the charge of the ferric hydroxide is pH-dependent, it should adsorb on its active surface sites the stable uranyl complex through electrostatic attraction. Minimum uranium removal was therefore observed when the charge of the uranyl species was the same as the charge of the flocs, and maximum removal occurred when the charges were opposite or neutral.

Monitoring Program

As part of this study, an analysis was conducted of several currently operating conventional water treatment facilities with uranium in their feed supplies. A detailed search was conducted to locate such facilities. For this search, a minimum uranium level of 15 μ g/L was arbitrarily selected, and conventional water treatment facilities were defined as any type of treatment facility more complex than sand separation and chlorination.

The wide variety of state and U.S. Government agencies responsible for the

Table 3. Ion Exchange Resin Capacity

Resin	Uranium Capacity* (kilograins/ft³)	Uranium Wet Volume Capacity+ (meq/mL)	Total Wet Volume Capacity≠ (meq/L)
DOWEX 21K	2.95	0.03	1.20
DOWEX SBRP	<i>3.15</i>	0.032	1.20
IONAC A641	3.67	0.0 38	1.16
IONAC A641 (upflow)	3.54	0.037	1.16

^{*}As UO2(CO3)2-

⁺Average over three cycles of operation (i.e., cycles 1,2, and 4). ≠Manufacturer's value: 1.2 meq/mL is 26 kilograins/ft³ as CaCO₃

Table 4. Summary of Uranium Removal Using 30 mg/L of Ferric Chloride Solution on Samples with Various pH levels

Adjusted pH	Uranium Remaining in the Tested Water (μg/L)	Uranium Removed (%) 31.3 44.2	
4.0	309	31.3	
4 .9	251	44.2	
<i>5.3</i>	180	60.0	
5.8	<i>86</i>	80.9	
8.2	<i>369</i>	18.0	
9.4	6	98.7	
10.0	<2	99.0	

Table 5. Effect of Ferric Chloride Concentration on Uranium Removal Efficiency in Solutions of pH 6 and 10

FECI ₃	Dosage(mg/L)	Uranium Remaining in the Tested Water (µg/L)	Uranium Removed (%)
pH 6	30	86	80.9
	60	<i>60</i>	86.6
	90	59	86.9
pH 10	<i>30</i>	<2	98+
•	<i>60</i>	<2	<i>98</i> +
	90	<2	98+

radiological monitoring of water supplies made the search very time-consuming. A total of 34 municipal systems and an additional 21 municipal wells were located in a 6-state area. Of these 55 possible study sites, only four provide treatment above and beyond sand separation and chlorination. Three of these cities are located in Colorado—Arvada, Denver, and North Table Mountain—and they all draw their water from the uranium-contaminated Ralston Reservoir. The fourth city is a small city in South Dakota (Harrisburg).

The City of Arvada treats their water using a microfloc system. The system employs alum and Separan (a polyelectrolyte) to create the microfloc. The water is then passed through mixed-media filters. A 125-cc water sample is collected daily from the raw water and treated water to form monthly composite samples. For the past couple of years, these monthly samples have been tested for uranium. The raw water from Ralston Reservoir contains a range of less than 1 μ g/L to 36 μ g/L, with an average of 14.7 \pm 9.6 (South Dakota) μ g/L of uranium. The Arvada facility removed 18% to 90% of this incoming uranium with a mean efficiency of $60\% \pm 15\%$. The meticulous nature of these records and the time span of the monitoring clearly indicate that conventional water

treatment facilities can greatly reduce the uranium content of natural waters.

The Moffat Treatment Facility (Denver) also draws water from Ralston Reservoir and has been keeping uranium records for about 2 years. Unfortunately, Denver's monthly samples are grab samples, and correlation between raw and treated waters are less meaningful. Using their records, uranium removal efficiency varies widely: $78\% \pm 190\%$. North Table Mountain declined to participate.

Harrisburg, South Dakota, treats about 100,000 gal/day using aeration, KMnO₄ greensand filters, chlorination, and fluoridation. No previous data exist on the uranium removal efficiency of this facility. Duplicate sampling over two different periods of operation showed no uranium removal by the current treatment system.

Ultimate Waste Disposal

As previously described, uranium-contaminated drinking water is a common problem, particularly in the western United States. If regulations are accepted and enforced, many communities will be required to remove the uranium from their drinking water supplies. Removal of this radioactive element produces a new problem of radioactive waste dis-

posal. The physical form of this waste and the longevity of uranium cause difficulties in formulating a waste disposal plan.

Three disposal alternatives were considered in this study: Dilution and release, reuse or resale, and burial. The choice of method must be based on environmental acceptability. Each participating community will have unique drinking water and waste characteristics, so it is impossible to prescribe a single solution to the problem. Each community must consider its situation and choose the optimum plan. The full report analyzes two cases: (1) a community using 150,000 gal water/day with 30 μ g/L uranium, and (2) a community using 150,000 gal water/day with 200 μ g/L uranium. Table 6 summarizes the quantity and form of uranium to be disposed of for each case. The first alternative of dilution and release is by far the most economical; however, there are usually restrictions on the quantity of uranium that can be returned to surface waters (e.g., in New Mexico, such flows must be less than 5 mg/L). Table 6 shows that for both Cases 1 and 2, the ion exchange regeneration solutions could be discharged to surface flows. However, the value given for Case 2 is an average value, and during operation of this project, we observed solutions exceeding 25 mg/L at the start of regeneration. This discharge would be the usual disposal solution for ion exchange systems.

Total environmental consideration suggests that there be alternative means of disposing of the uranium, and the filter cake resulting from chemical clarification is the first step in such disposal. In Case 1, the cake would contain 0.057 nanocuries of uranium/g, and in Case 2, 0.38 nanocuries of uranium/g. Both cases are well below the 100-nanocurie/g definition of waste requiring special packaging and disposal in a permanent repository. The filter cake could be drummed and sent to an approved site for shallow surface burial. The alternative is to send it to a uranium mill for processing as part of the mill feed.

Design Analaysis

Though complete cost analysis has been covered by several recent publications, two specific designs are summarized in Tables 7 an 8. They include capital costs for equipment only, since operating costs are site dependent.

Table 6. Ultimate Disposal Values for Uranium in a Community Using 150,000 gal water/day

Uranium Source	Amount and Form of Uranium to be Disposed of
Case 1 (30-µg/L uranium content):	
lon exchange treatment regenerant solution Chemical clarification filter cake Total uranium	0.3 mg/L, 1500 gal/day 17.03 kg/day 1.7 g/day
Case 2 (200-µg/L uranium content):	
lon exchange treatment regenerant solution Chemical clarification filter cake Total uranium	2.0 mg/L, 1500 gal/day 17.04 kg/day 11.35 g/day

Table 7. Ion Exchange System Design*

ltem .	Design Data
Average influent concentration of UO ₂ (CO ₃) ₂ ⁻²	84 μg/L or ≅ 50 μg/L as uranium
Feed needed (1-day basis) based on average capacity of 3.3 kilograins/ft ³	600 gal x .084 ppm x .0547 min
•	$\frac{grains/gal}{ppm} \times 24 \text{ hr } \times 60 \frac{min}{hr} =$
	3960 grains
Resin needed for 1 day of operation	$\frac{3.96 \text{ kilograins}}{3.3 \text{ kilograins/}tt^3} = 1.20 \text{ ft}^3$
Size of tank based on 10 gpm/ft² flow rate	$\frac{600 \text{ gpm}}{10 \text{ gpm/ft}^2} = 60 \text{ ft}^2$
Diameter of tank 9 ft	Area = 63.6 ft²
Bed depth 3 ft	Resin quantity = 63.6 ft^2 x 3 ft = 191 ft^3
Use 100% freeboard for B/W and/or expansion	
tank size	9 ft dia. x 5 ft side shell
Approximate running time between regeneration	
Time of operation	191 ft ³ 1.20 ft ³ /day
Salt needed for regeneration	$15 lb/ft^3 x 191 ft^3 = 2.86 lb$
Estimated capital equipment cost*	\$125,000

^{*}Cost analysis for chemical composition is given in Table 1.

The cost curves for the two unit operations considered were updated to 1985 costs using the Civil Engineering plant cost index data published in *Chemical Engineering*. The index used was 375.2 (July 1985) based on 1957-59 as 100. These cost curves were scaled for the specific processing of uranium.

Conclusions

Four commercial-type ion exchange columns and prefiltering and regeneration solution systems were tested to remove uranium concentrations in the water supply well in which concentrations varied from 190 to 400 μ g/L during the study period.

Greater than 99% removal of uranium was achieved when operating at 30 mg/L ferric chloride and pH 10. The diatomaceous earth precoat filter achieved complete solid-liquid separation. No thermal degradation of resins were observed in the temperature range of 35°F to 115°F. Chemical degradation also was not experienced in this study. Minimum uranium removal was observed when the charge of the uranyl species was the same as the charge of the flocs, and maximum removal was experienced when the charges were opposite or neutral.

Review and analysis of records of currently operating water treatment systems whose feed supplies contain uranium indicated that conventional water treatment facilities can greatly reduce the uranium contents of natural waters.

Ultimate waste disposal plans for radioactive waste from drinking water supplies are difficult to formulate because of the longevity of uranium and the physical form of the waste. Methods considered in this study are dilution and release, reuse or resale, and burial. If the uranium content of the waste is below that required by the permanent repository guidelines to have special packaging and disposal, then the waste can be drummed and sent to an approved site for shallow surface burial or to a uranium mill for processing as part of the mill feed.

The full report was submitted in fulfillment of Cooperative Agreement CR 810453 by New Mexico State University, under the sponsorship of the U.S. Environmental Protection Agency.

Table 8. Chemical Clarification System Design

ltem .	Design Data	
Average influent concentration	0.5 mg/L as U	
Feed rate	60 gpm	
Joy Manufacturing flotation cell	16 ft ³	
Residence time	2 min	
Ferric chloride addition	25 mg/L, 35.7 lb/day	
pH adjustment (assuming feed at pH 7 to 10)	2 L conc. (37%) HCI/day*	
Continuous rotary vacuum (precoat) filter	6 ft² total surface area	
Filter-aid addition 2x weight of ferric chloride (diatomaceous earth)	70 lb/day	
Total filter production/day (dry weight and wet weight, 8% moisture)	106 lb/dry weight, 115 lb/wet weight	
Total estimated capital equipment costs	\$44,000	

^{*}NaOH could also be used if pH needs to be increased.

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Richard P. Lauch is the EPA Project Officer (see below).

The complete report, entitled "Removal of Uranium from Drinking Water by Ion Exchange and Chemical Clarification," (Order No. PB 88-102 900/AS; Cost: \$13.95, subject to change) will be available only from:

National Technical Information Service

5285 Port Royal Road

Springfield, VA 22161

Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

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