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TECHNOLOGY TRANSFER: ION EXCHANGE RESINS FOR TECHNETIUM-99 REMOVAL FROM X-705 RAFFINATES

By

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SUMMARY

An ion exchange process will be used at Portsmouth to remove technetium-99 from uranium recovery waste solutions (raffinates). Subsequent treatment will then remove nitrates from the raffinates by a biodenitrification process prior to discharge to receiving streams to meet environmental standards for liquid wastes. Ion exchange process parameters affecting safe and efficient raffinate treatment have been examined in the laboratory, and results are described in this report.

Amberlite IRA-401 resin, in the nitrate form, was evaluated for the technetium-99 acidic ion exchange removal process as a follow up to previously reported work. Additional studies on pretreatment and usage of Amberlite IRA-401 indicated that conversions of the resin from the as-received chloride form to the hydroxyl form, and finally to the nitrated form, could be easily monitored by means of the color changes which occur. Changes in pH over the operational range (0.1 - 1.0) were found to have no significant effect upon the technetium removal capability of the resin. Therefore, pH adjustment of the raffinate is not necessary to optimize technetium removal by the nitrated resin. Uranium is also retained on the nitrate form Amberlite resin at a loading capacity of 1 mgU/g-resin. Subsequent washing of the resin with distilled water successfully removed approximately half of the uranium from the resin without removing technetium. The information on uranium loading and water leaching from the resin is required for assessing nuclear criticality concerns during disposal/storage of the spent resin.

Comparison studies of Amberlite IRA-401 and Dowex 1-X8 ion exchange resins in the chloride and hydroxyl forms operating in an alkaline condition were investigated to replace the nitrated resins because the latter are potentially more chemically unstable and hazardous based on literature reports. Results showed that both the hydroxyl and chloride form resins were equally effective for removing technetium from neutralized (pH 8.0) and filtered raffinate. However, the chloride form resin reacted with the alkaline raffinate resulting in large amounts of off-gassing which would cause channelling in the resin bed. The hydroxyl form resin should, therefore, be used. The technetium loading capacity for the Dowex 1-X8 resin in hydroxyl form was found to be 23 mgTc/g-resin, while the loading capacity of the Amberlite IRA-401 in hydroxyl form was found to be 20 mgTc/g-resin. In comparison, the previously determined nitrated Amberlite resin loading capacity for untreated acidic raffinate was only 1.4 mgTc/g-resin.

Although very low concentrations of uranium (<1 ppm) remain after raffinate neutralization, some uranium will be retained on the hydroxyl form resins, and loading information for criticality assessment must be established. Data and calculations are presented upon which to base nuclearly safe conditions for disposal of the spent resins.

It is recommended that hydroxyl form resin be used in an alkaline ion exchange process for the removal of technetium from uranium recovery waste solutions generated at Portsmouth.

INTRODUCTION

The internal surfaces of cascade equipment removed from service may be contaminated with technetium-99 (a weak beta emitter) as well as uranium. For worker protection and to allow recovery of uranium, such equipment is wet-decontaminated before upgrading, maintenance, or disposal. Uranium is recovered from the resultant decontamination solutions by solvent extraction; the technetium-99 remains in the concentrated acidic waste solutions or "raffinates" from the solvent extraction process. Discharge of these waste solutions is controlled to meet DOE guidelines for radioactive effluents and waste solutions are stored when discharge limits are reached. Based upon laboratory studies of a number of possible approaches, an ion-exchange process was selected for removal of technetium from the raffinates to allow unrestricted waste solution discharge.¹

The process selected was based upon the removal of technetium directly from the highly acidic raffinates using the nitrated form of Amberlite IRA-401 ion exchange resin. As design and engineering of the proposed facility proceeded, additional laboratory studies were made to address concerns of a safety, nuclear criticality, and operational nature associated with the specific ion exchange resin to be used. The purpose of this report is to document and transfer the technology identified by those additional studies.

Section I of the report describes testing of the nitrated resin on the acidic raffinate. This included resin conversion, technetium removal at pH levels of 0.0 to 0.9, and uranium loading capacity with subsequent water leachability.

The processing of raffinates to meet environmental restrictions also incorporates a biodenitrification process to control nitrate level. The acidic raffinates contain dissolved metals some of which are toxic to the biodenitrification bacteria and/or exceed discharge limits. These metals are removed by treating the raffinates with sodium hydroxide to form the insoluble metal hydroxides which are then filtered off. Section II of this report deals with the testing of hydroxyl and chloride form ion exchange resins for technetium removal from the alkaline solution resulting from treatment of the acidic raffinate to remove metals.

A flow diagram depicting the acidic or alkaline ion exchange processes for technetium removal in relation to the other operations involved is shown in Figure 1.

Figure 1. Flow Diagram for Alternative Acidic or Alkaline Ion Exchange Processes of Tc-99 Removal From Uranium Recovery Raffinate Solutions.

I. LABORATORY STUDIES OF NITRATE FORM ION-EXCHANGE RESIN

Amberlite IRA-401 ion-exchange resin, in the nitrate form, was originally proposed for use in the X-705 Decontamination Building Ion-Exchange Facility for technetium removal from raffinate. Amberlite IRA-401 resin is a quaternary ammonium polystyrene resin manufactured by Rohm and Haas Company, Philadelphia, Pennsylvania, available only in the chloride form. Initial work involved converting the resin from the chloride form via an intermediate hydroxyl form, to a final nitrate form. Conversion to the hydroxyl form intermediate is necessary to achieve as large a capacity for pertechnetate loading as possible.² Resin conversion was investigated in-situ using the pilot plant ion exchange columns, and as a batch treatment in the laboratory, to determine the most practical method. The technetium removal capability of the nitrated resin was then determined at various raffinate pH levels. The degree of uranium retention on the resin was also established by studies which included attempts to remove the uranium from the resin by distilled water washes.

Resin Conversion From Chloride Form to Hydroxyl Form to Nitrate Form

In-place conversion of the Amberlite IRA-401 ion-exchange resin from the chloride form to nitrate form was performed in a full-size (5' L x 4" ID) glass column. The glass column was filled to 33½ inches bed depth with the resin which was then washed with six liters of distilled water. The resin column bed compacted by 1½ inches (3%) after washing. This was followed by three liter additions of one molar sodium hydroxide (NaOH) to convert the chloride (Cl⁻) form resin to the hydroxyl (OH⁻) form. A color change of the resin (yellow to light brown) was useful in determining when this treatment was complete. A total of 16 liters of 1 molar NaOH was required. The resin was then washed with eight liters of distilled water to remove excess hydroxyl ions. This resulted in resin column bed expansion of 2¾ inches (7%). The hydroxyl form resin was converted to the nitrate form by slowly adding four-liter increments of 0.75 Normal nitric acid (HNO₃). Another color change (light brown to gold) indicated when this treatment was complete. A total of nine liters of HNO₃ was needed. The nitrate form resin was again washed with distilled water (eight liters total). This operation compacted the resin bed by 4¾ inches (14%). Final resin bed level was 30 inches (85% of original dry resin loading).

A batch treatment of the resin was then evaluated in the laboratory to compare with results of the glass column treatment. The same solutions, 1M NaOH and 0.75 N HNO₃, were used in this conversion procedure, including washings with distilled water. Initially, five liters of resin were washed with three liters of distilled water for a total volume (resin and water) of six liters. Four liters of 1M NaOH were added to achieve the light brown color throughout the

resin. The resin was rewashed and decanted to six liters total volume. Two liters of 0.75N HNO_3 were required to obtain the gold color. No hazardous or exothermic reactions occurred when slowly adding the nitric acid to the basic resin.

Effect of pH Adjustment on Technetium-99 Removal Efficiency

The capability of the nitrated Amberlite IRA-401 resin for technetium removal was examined using actual raffinate solutions. The raffinate was neutralized with 25% (by weight) NaOH solution, through a nominal pH range of 0.0 - 1.0. Samples (50 ml) were taken at each one-tenth pH increment and processed through an 80-gram nitrated resin column at slow drip rates. Influent and effluent samples were taken and analyzed for uranium and technetium removal capability of the resin at each pH increment, and the amount of uranium retention on the resin (Table I).

Results indicated that no significant difference in technetium removal by the resin was seen at each pH increment over the range investigated. However, the resin also retained some uranium from the raffinate. This uranium loading onto the resin could present possible criticality problems affecting resin disposal.

TABLE I
Tc-99 & URANIUM REMOVAL VERSUS pH FOR NITRATED AMBERLITE
IRA-401 RESIN

pH	Influent		Effluent		% Removal	
	Tc, $\mu\text{g/ml}$	U, mg/l	Tc, $\mu\text{g/ml}$	U, mg/l	Tc	U
≤ 0.0 (as-received)	6.08	411	0.030	100	99.5	75.7
0.1	4.95	299	0.019	40	99.6	86.6
0.2	4.86	307	0.022	31	99.5	89.9
0.3	4.91	304	0.015	32	99.7	89.5
0.4	4.77	298	0.023	35	99.5	88.3
0.5	4.70	285	0.024	39	99.5	86.3
0.6	4.50	284	0.025	34	99.4	88.0
0.7	4.54	288	0.036	47	99.2	83.7
0.8	4.66	287	0.027	35	99.4	87.8
0.9	4.58	279	0.043	56	99.1	79.9
1.0	4.80	282	0.061	117	98.7	58.5

Uranium Retention on Amberlite IRA-401 Resin

A. Batch Raffinate Treatment Method

Tests were done to determine the amount of uranium taken up from raffinates by the resin and to determine if distilled water washes of the resin removed any uranium.

The tests were done by continuously mixing 250 grams of nitrated Amberlite IRA-401 with three liters of acidic raffinate for 24 hours. The resin was filtered and washed three times with one liter of demineralized water (pH 5.0). The resin was then eluted with one liter of 3N nitric acid (HNO_3) for 72 hours to strip the resin of technetium. Samples were taken of the as-received raffinate, after the 24 hour mixing, after each wash, and of the final elution. This procedure was repeated in triplicate, and all the samples were analyzed for uranium and technetium content (Table II).

TABLE II
URANIUM LOADING ON NITRATED AMBERLITE IRA-401
RESIN (BATCH RESIN METHOD)

Sample	Batch "A"		Batch "B"		Batch "C"	
	U mg/l	Tc μ g/ml	U mg/l	Tc μ g/ml	U mg/l	Tc μ g/ml
Original Raffinate	382	3.56	382	3.56	382	3.56
Raffinate After Resin Treatment	340	.923	301	.922	313	.916
1st DI H ₂ O Wash of Resin	94	.063	116	.092	148	.108
2nd DI H ₂ O Wash of Resin	29	.018	26	.025	17	.041
3rd DI H ₂ O Wash of Resin	3.8	.019	9.1	.026	1.8	.039
HNO ₃ Elution of Resin	35	1.20	22	1.22	22	1.33

The results indicated that technetium is loaded onto the resin and held through each water rinse. The final elution of the resin with 3N HNO₃ did not effectively strip the technetium off the resin, indicating that it is tightly bound to the resin.

Analytical results for uranium indicated that uranium is initially loaded onto the resin, but can be partially removed with subsequent washings with demineralized water. After the final elution with 3N HNO₃, an average of 23.4% of uranium initially present still remained on the resin.

B. Column Ion Exchange Raffinate Treatment Method

Uranium loading determinations were repeated using ion exchange columns as opposed to the previous batch tests. Two columns were packed with 125 grams of nitrated Amberlite resin each. One and a half liters of acidic raffinate were slowly passed through each column. The effluent was collected and analyzed for uranium and technetium content. The resin was washed three times with distilled water to evaluate possible technetium or uranium removal. The volume of each wash was 500 ml, and the color of successive washes diminished from yellow to clear. Finally, the resin was eluted with 500 ml of 3N HNO₃ to remove the remaining technetium and uranium. Results of these tests are shown in

Table III. Overall, the batch test and column test produced similar results. Uranium concentrations on the resin were reduced approximately 50% by the distilled water washings. After removing residual raffinate in the resin by the first wash, subsequent washings removed very little of the technetium. The strong resin-technetium attraction was again shown by low recovery in the acid elution. Calculations of the uranium content on the resin indicated an initial loading capacity of approximately 1.0 mg U/g-resin.

TABLE III
URANIUM LOADING ON NITRATED AMBERLITE IRA-401
RESIN (ION-EXCHANGE COLUMN METHOD)

Sample	Column "A"		Column "B"	
	U mg/l	Tc μ g/ml	U mg/l	Tc μ g/ml
Original Raffinate	332	6.1	332	6.1
Raffinate After Resin Treatment	225	0.77	255	0.73
1st Dist. H ₂ O Wash	105	0.20	107	0.21
2nd Dist. H ₂ O Wash	19	0.0016	22	0.0019
3rd Dist. H ₂ O Wash	5.2	0.005	5.3	0.007
HNO ₃ Elution	26	2.2	20	2.2

II. LABORATORY STUDIES OF HYDROXYL AND CHLORIDE FORM ION EXCHANGE RESINS

A laboratory study was done to examine the feasibility of using a hydroxyl (OH⁻) form or chloride (Cl⁻) form ion-exchange resin for technetium removal from the alkaline solution resulting after neutralizing the acidic raffinate to pH 8.0. Comparison studies of Amberlite IRA-401 and Dowex 1-X8 resins in the chloride and hydroxyl forms were conducted to determine relative capabilities for removing technetium. Dowex 1-X8 is a laboratory grade, anionic resin manufactured by DOW Chemical Company, Midland, Michigan. It is also a quaternary ammonium polystyrene resin, available in either the chloride or hydroxyl form. After determining the preferred form of resin, the technetium and uranium loading capacities were calculated from the data.

Resin Comparison - Amberlite IRA-401 and Dowex 1-X8

"C" loop raffinate and X-701B Holding Pond effluent were the sources of technetium-containing solutions used for the resin comparison. Four liters of "C" loop raffinate were neutralized to pH 8.0 with 25% (by weight) sodium hydroxide (NaOH) solution, and then filtered. A sample of this neutralized raffinate solution was analyzed for technetium content. Aliquots of 250 ml of the neutralized raffinate solution were then passed through the ion exchange columns. The columns were filled with 100 grams of each resin in the hydroxyl form and 100 grams of each resin in the chloride form. Duplicate samples were run with each resin. The last 100 ml of each 250 ml aliquot were collected and analyzed for technetium content. Results are in Table IV.

TABLE IV
RESIN COMPARISON - "C" LOOP RAFFINATE

Sample	$\mu\text{g Tc/ml}$	D/m/ml*
"C" Loop Raffinate pH 8.0	7.35	278,000
Dowex 1-X8 Hydroxyl Form	0.01 0.01	255 210
Amberlite IRA-401 Hydroxyl Form	0.01 0.01	370 510
Dowex 1-X8 Chloride Form	0.25 0.19	9500 6800
Amberlite IRA-401 Chloride Form	0.01 0.01	280 210

*Disintegrations/minute/ml

The same procedure was used with the effluent from the X-701B Holding Pond. As-received, the solution had a pH 9.0, so no pretreatment was necessary. An initial sample was analyzed for technetium content. Aliquots of 250 ml were passed through each of the ion exchange columns with the last 100 ml of each aliquot collected and analyzed for technetium. The columns were prepared with each resin as before, and duplicate samples run. Results are shown in Table V.

TABLE V
RESIN COMPARISON - X-701B HOLDING POND

Sample	$\mu\text{g Tc}/100 \text{ ml}$	D/m/ml *
X-701B Holding Pond pH 9.0	2.1	798
Dowex 1-X8 Hydroxyl Form	<0.03 ----- <0.03	<10 ----- <10
Amberlite IRA-401 Hydroxyl Form	<0.03 ----- <0.03	<10 ----- <10
Dowex 1-X8 Chloride Form	0.28 ----- 0.24	96 ----- 81
Amberlite IRA-401 Chloride Form	<0.03 ----- <0.03	<10 ----- <10

*Disintegrations/minute/ml

In comparing the test results for the "C" loop raffinate and X-701B Holding Pond effluent, technetium can be removed from either of these solutions using Amberlite IRA-401 resin or Dowex 1-X8 resin in either the chloride or hydroxyl form. However, the efficiency of technetium removal decreased with the Dowex 1-X8 in the chloride form. Reaction of the basic solutions with the resin resulted in large amounts of visible evolution of gas. Gas pockets migrated to the top of the column, disturbing the resin bed, as the neutralized raffinate was added. Observations indicated channelling through the resin occurs which decreases the efficiency of the chloride resins if used for technetium removal. Therefore, the hydroxyl resin is preferred. The hydroxyl forms of Dowex 1-X8 and Amberlite IRA-401 resins were found to be equally efficient for technetium removal.

Technetium Loading on Hydroxyl Form Resins

Tests were made to determine the technetium loading on each of the hydroxyl resins. Actual raffinate solutions were pretreated prior to use. Seven and one-half liters of "C" loop raffinate were neutralized with 25% (by weight) NaOH solution, to pH 8.0. The solution was filtered to remove the resulting heavy floc, and the filtrate spiked with approximately 1800 ml of ammonium pertechnetate solution.

The resulting pH of the solution was 0.27, so 25% NaOH was again used to raise the pH to 8.0. A sample analyzed for technetium content indicated 280 μg Tc/ml.

Ten grams of Amberlite IRA-401 resin and ten grams of Dowex 1-X8 resin (both in hydroxyl form) were placed into two ion exchange columns. 250 ml aliquots of the spiked raffinate solution were passed at a slow drip rate through each of the resins, and 10 ml samples collected and analyzed for technetium content. Dowex 1-X8 resin required 1.5 liters of the spiked raffinate solution for complete breakthrough. Results of technetium removal of the Dowex resin, based on technetium analysis of the 10 ml column effluent samples, is shown in Figure 2. Amberlite IRA-401 resin required 1.25 liters for complete breakthrough. Figure 3 shows the plot of technetium removal by the Amberlite resin.

Loading capacities were then calculated for the Dowex and Amberlite resins. Dowex 1-X8 resin was found to have a loading capacity of 23 mg Tc/g-resin, while Amberlite IRA-401 resin yielded 20 mg Tc/g-resin. This contrasts with the much lower uptake capacity of 1.4 mg Tc/g-resin found for nitrated resin in earlier work.

Figure 2. Technetium Removal From Neutralized Raffinate (pH 8.0), Using Dowex 1-X8 Resin (10 grams) in Hydroxyl Form.

Figure 3. Technetium Removal From Neutralized Raffinate (pH 8.0), Using Amberlite IRA-401 Resin (10 grams) in Hydroxyl Form.

Technetium and Uranium Removal by Distilled Water Washes

As with the nitrated resins, the hydroxyl form resins were checked for technetium and uranium removal by washing with distilled water. Each resin was washed with four 250 ml increments of distilled water. The individual washings were collected and analyzed for technetium and uranium content. Table VI lists the analytical results for each resin. Results of technetium removal were similar for the Dowex and Amberlite resins. The first wash rinsed the residual raffinate from the resin, accounting for the relatively larger technetium concentration found, but the subsequent washings removed very little technetium from the resin. Neutralization and filtration of the raffinate had removed most of the uranium. Consequently, the low uranium concentration, in the neutralized and filtered raffinate, resulted in small amounts of uranium removed by the resin. Subsequent distilled water washings removed no appreciable amount of uranium from the resin.

TABLE VI
TECHNETIUM AND URANIUM ANALYSIS OF DISTILLED H₂O WASHINGS

	Dowex 1-X8		Amberlite IRA-401	
	Tc $\mu\text{g/ml}$	U mg/l	Tc $\mu\text{g/ml}$	U mg/l
1st 250 ml wash	37.9	0.02	28.6	0.03
2nd 250 ml wash	0.26	<0.01	0.46	<0.01
3rd 250 ml wash	0.016	<0.01	0.047	<0.01
4th 250 ml wash	0.018	<0.01	0.009	<0.01

Uranium Loading on Hydroxyl Form Resins

The 10 ml samples from the technetium-loading study of the hydroxyl form resin were placed into composite samples of ten each. The resulting 100 ml samples were then analyzed for uranium content to determine the uranium loading on the Dowex 1-X8 and Amberlite IRA-401 resins (both in hydroxyl form). The results showed uranium removal was not consistent at the reduced uranium concentration found in neutralized, filtered raffinate.

Although uranium loadings could not be determined analytically, theoretical calculations can approximate the uranium loading capacities using known technetium loading data. Assuming a concentration of 10 μg Tc/ml of process raffinate, it would require 17, 135 liters of raffinate to saturate one 7.45 kg resin column⁵ at a 23 mg Tc/g-resin

loading capacity. This assumes continuous complete removal of technetium. Ideally, the resin should remove 100% of the technetium until it is saturated. Laboratory results show that the removal decreases until the resin is saturated and breakthrough occurs. The average removal of technetium over this period is 55%. Therefore, 31,155 liters of raffinate would be needed to saturate the resin column with technetium based on the above assumptions. The estimated maximum amount of raffinate being generated is 54,000 liters per month.

Precipitation and filtration had reduced the uranium concentration from 400 to <1 mg/l. If 100% of the uranium is removed from the treated raffinate solution while processing through the resin column, the theoretical total loading of uranium on the resin would be 17.1 grams. Adjusting this to the 55% efficiency would result in a total of 31.2 grams of uranium loaded onto the resin of each column. Uranium at this concentration should produce no criticality problems for resin disposal.

CONCLUSIONS

The following conclusions were reached from the results of the laboratory tests:

- Amberlite IRA-401 and Dowex 1-X8 resins in the hydroxyl and chloride forms effectively remove technetium from neutralized (pH 8.0) and filtered raffinate. However, the chloride form resins reacted with the alkaline raffinate evolving large amounts of gas which can interfere with column operation, thus making the hydroxyl resins preferable for actual process use.
- To provide data for engineering estimates of resin replacement rates, technetium loading capacities were determined for each resin in the hydroxyl form. Dowex 1-X8 was found to have a loading capacity of 23 mg Tc/g-resin, while Amberlite IRA-401 has a loading capacity of 20 mg Tc/g-resin. This is a substantial increase over the 1.4 mg Tc/g-resin previously reported for nitrated resin.
- Uranium is partially precipitated and removed with other metals by filtration after pH adjustment of the acidic raffinate to 8 - 8.5, thus alleviating subsequent potential criticality problems with resin disposal.
- Nitrated Amberlite IRA-401 resin effectively removed technetium from acidic raffinate in pH ranges of 0.0 to 0.9, which indicates that partial neutralization of raffinate is not needed to optimize technetium removal by the nitrated resin.
- Uranium was retained on the nitrated resin at a loading capacity of 1.0 mg U/g-resin, but subsequent distilled water washings removed approximately 50% of the uranium.
- Technetium is not significantly removed from the resin during water washing regardless of which resin process is used.
- As Amberlite IRA-401 resin is converted from the as-received chloride form to the hydroxyl form and to the nitrate form, corresponding color changes of yellow to light brown to gold are observed. These color changes could be useful for process control purposes.

RECOMMENDATIONS

Based upon the studies which have been made, it is recommended that a hydroxyl form ion exchange resin be used for technetium removal in the X-705 Ion Exchange Facility. Hydroxyl form resins are compatible with a process scheme in which technetium removal is accomplished after neutralization of the acidic raffinate and sludge removal of precipitated metal hydroxides by filtration. This metals removal process is proposed for the influent to the biodenitrification facility. Hydroxyl form resin avoids the potential safety problem associated with the chemical instability of the nitrate form resin and, furthermore, has substantially greater technetium removal capacity than nitrated resin. Removal of uranium from the raffinate by alkaline precipitation also minimizes the loading of uranium on the resin to alleviate possible criticality problems with resin storage. Since Amberlite resin is no longer commercially available, when current supplies are exhausted, Dowex 1-X8 ion exchange resin in the hydroxyl form should be procured.

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