

Title:

**THE REMOVAL OF URANIUM FROM ACIDIC MEDIA
USING ION EXCHANGE AND/OR EXTRACTION
CHROMATOGRAPHY**

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The Removal of Uranium from Acidic Media Using Ion-Exchange and/or
Extraction
Chromatography

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Abstract

The separation and purification of uranium from either nitric acid or hydrochloric acid media can be accomplished by using either solvent extraction or ion-exchange. Over the past two years at Los Alamos, emerging programs are focused on recapturing the expertise required to do limited, small-quantity processing of enriched uranium. During this period of time, we have been investigating ion-exchange as a method for extraction/purification of uranium from acidic media. In addition, waste stream polishing is associated with this effort in order to achieve more complete removal of the uranium prior to recycle of the acid. Extraction chromatography has been demonstrated to further polish the uranium from both nitric and hydrochloric acid media thus allowing for a more complete recovery of the actinide material and creation of less waste during the processing steps.

We will discuss contact experiments for evaluation of eleven different resins for uranium removal from nitric acid, the evaluation (again by contact experiments) of uranium elution from the ion-exchange media by various chemicals, and small-scale experiments for the removal of ^{235}U from nitric acid. Larger-scale (100-gram) flow experiments, using depleted uranium, were done after the small-scale evaluation of the selected resin. A discussion is also included concerning the shortcomings of the contact experiments and how the flow experiments were modified in order to overcome these problems. We will also discuss the application of ion-exchange for some specific process needs at Los Alamos.

Contact experiments were used to evaluate some commercially available solvent-impregnated resins in addition to evaluation of several resins of this type synthesized in our laboratory. These experiments involved both nitric and hydrochloric acid media. The data from these experiments will be used to determine the column size and amount of resin required to polish the waste stream produced by the ion-exchange process described above. Small-scale flow experiments will be described and data given on the mass balances achieved using this two step process.

Future work for evaluation of new ion-exchange and solvent-impregnated resins as well as the search for new eluants will be discussed. Other future experiments for both processes, including large-scale experiments, will be discussed in detail.

Introduction

The extraction of uranium from ores is typically accomplished by leaching the ores with sulfuric acid (H_2SO_4) and the crude uranyl sulfate is extracted either by ion-exchange or solvent extraction methods. After extraction, the uranium is precipitated either as the peroxide or by using ammonium hydroxide to neutralize the H_2SO_4 solution thus forming ammonium diuranate. This impure uranium material is then redissolved in an acid media, typically nitric acid (HNO_3), and precipitated as either uranium peroxide or as ammonium diuranate. These solid compounds can then be thermally decomposed to form a variety of uranium oxides, however U_3O_8 is frequently produced. If the uranium is to be enriched, then the U_3O_8 is either redissolved in HNO_3 , reprecipitated, calcined to uranium trioxide (UO_3), which is reduced using hydrogen to uranium dioxide, (UO_2); or the U_3O_8 can be directly reduced using hydrogen (H_2) at $750\text{ }^\circ\text{C}$ to form UO_2 . (1) The UO_2 can then be hydrofluorinated using anhydrous hydrogen fluoride (HF) to form uranium tetrafluoride (UF_4), which is easily converted to uranium hexafluoride (UF_6), using either chlorine trifluoride (ClF_3) or fluorine (F_2). The gaseous UF_6 is then enriched either by gaseous diffusion or by gas centrifuges. The enriched UF_6 is reduced back to UF_4 using hydrogen and can be further treated by either pyrohydrolysis to obtain either UO_2 , UO_3 , or U_3O_8 . Also, Ca metal can fully reduce the UF_4 to uranium metal with calcium fluoride (CaF_2) as a by-product.

Processing plants in the nuclear complex have used a variety of methods for purification of either enriched or depleted uranium arising from the purification of uranium oxides and the production of uranium metal. For example the Y-12 plant at Oak Ridge, Tennessee, uses solvent extraction as the primary method for extraction and purification of the uranium. A generalized processing flow sheet is shown as Figure 1.

Nuclear Fuels Services (NFS), of Erwin, Tennessee, has processed uranium fuel for nuclear submarines, also using solvent extraction for recovery and purification of the uranium. The major difference between these two plants is that the Y-12 plant uses two extractants, tributyl phosphate (TBP) and dibutyl carbitol (DBC), to effect the separation and purification. This has been combined into a single step by NFS, using TBP. The separation and purification of uranium from irradiated fuel arising from the production reactors at Hanford, was accomplished using multiple TBP extractions. The recovered uranium was then thermally denitrated, converted to UO_3 and eventually fabricated into new fuel elements. This processing was quite similar to the steps outlined in Figure 1.

At Los Alamos, prior to 1984, highly enriched uranium, HEU, was processed at DP-site (TA-21). This processing included solvent extraction and the reduction of UF_4 to metal. It is this processing technology which is currently being established through a Uranium Technology Development Program, which began 2-3 years ago. The goal of this program is the construction of a glovebox processing line whose acronym is ULISSES, which stands for Uranium Line for Special Separation Science. Not only is the capability of uranium processing being recaptured, but new technologies/processes are under development. Included in these various technologies is the use of ion-exchange for the separation/purification of uranium. Part of the experimentation for this program will be more fully described in following paragraphs.

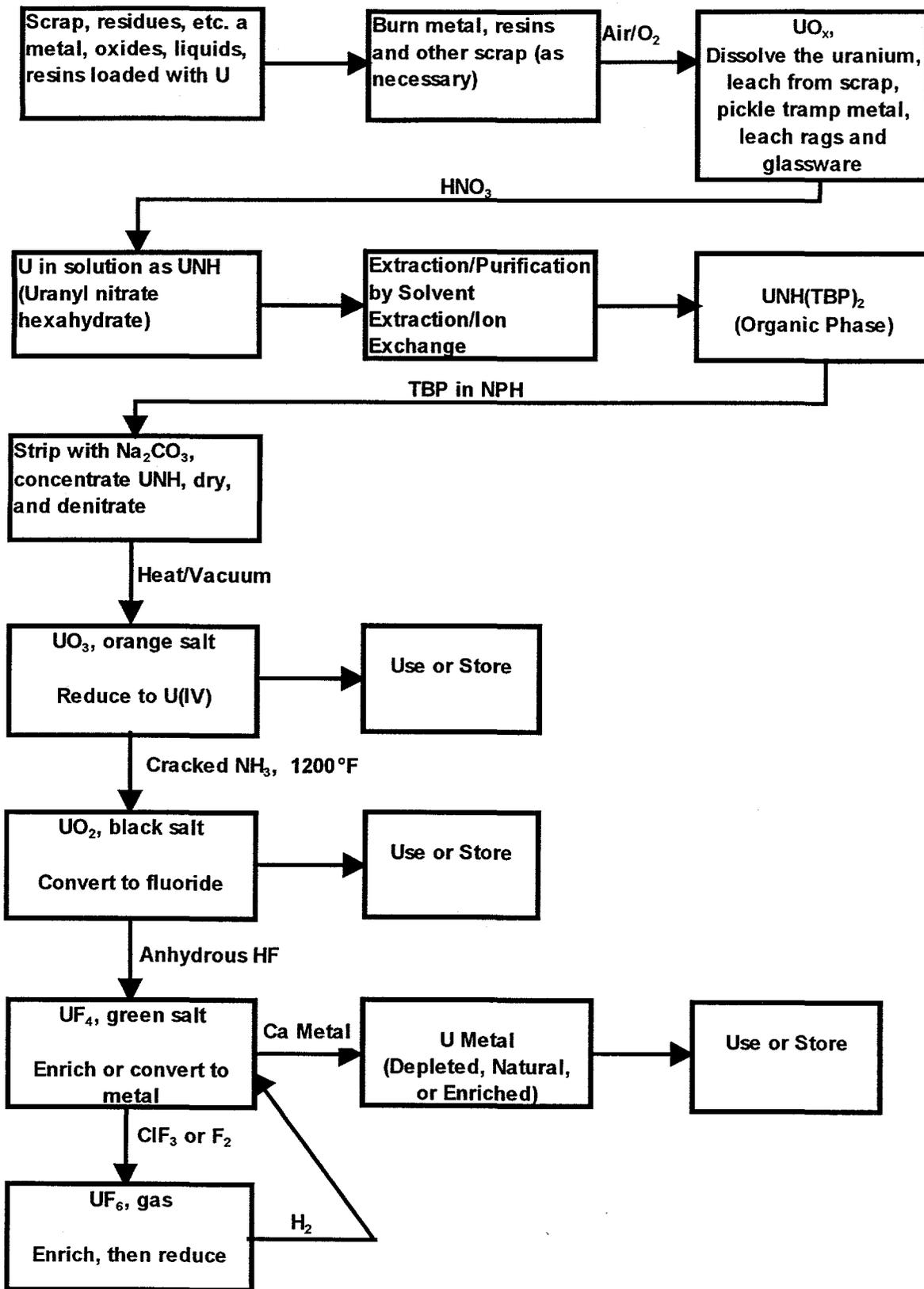


Figure 1 - Typical Uranium Processing Flow Sheet

Ion-Exchange Contact Studies, HNO₃ Media

The contact studies were done with a variety of resins to determine which one(s) would be best suited for uranium extraction from nitric acid media. The resins studied are listed in Table I. Included in the table are the manufacturer, the resin backbone or matrix material and the functional group.

Table I - Resins Used in This Study

Resin	Manufacturer	Matrix Material	Functional Group
Chelex 100	BioRad Laboratories, Richmond, CA	Styrene and Divinylbenzene Copolymer	Imino diacetic acid
Diphonex	Eichrom Industries, Darien, Illinois	Styrene and Divinylbenzene Copolymer	Sulfonic acid and gem-diphosphinic acid
Dowex 50x2	Dow Chemical Company, through BioRad Laboratories	Styrene and Divinylbenzene Copolymer	Sulfonic Acid
Duolite C-467	Rohm & Haas, Philadelphia, Pennsylvania	Styrene and Divinylbenzene Copolymer	Amino phosphonate
IRC-718	Rohm & Haas, Philadelphia, Pennsylvania	Macroreticular	Imino diacetic acid
Lewatit M500	Miles Inc., Philadelphia, Pennsylvania	Cross-linked polystyrene	Quaternary Amine
TRU-Spec	Eichrom Industries, Darien, Illinois	Amberchrom CG-71	13% CMPO with 27% TBP
UTEVA-Spec	Eichrom Industries, Darien, Illinois	Amberchrom CG-71	40% Diamyl amyl phosphonate (DAAP)
XAD-4; TBP	Los Alamos	Amberlite XAD-4	40% TBP
XAD-4; CMPO	Los Alamos	Amberlite XAD-4	40% CMPO
XAD-7; CMPO	Los Alamos	Amberlite XAD-7	40% CMPO

Experimental Conditions

The contact experiments were accomplished using 20 mL, capped polyethylene columns (Bio-Rad Laboratories) with a frit at the bottom. Each column was sealed at the bottom with a small plastic valve. A 0.5 gram sample of the resin was placed in the column and 5 mL of the acid was also added. The acid concentration was varied from 1-10M, while later experiments were done with 0.001-1.0 M acid. The uranium sample (0.1-0.5 mL), as uranyl nitrate in 5M HNO₃, was added to the tube and the sample was rotated on a Lab-Quake™ shaker

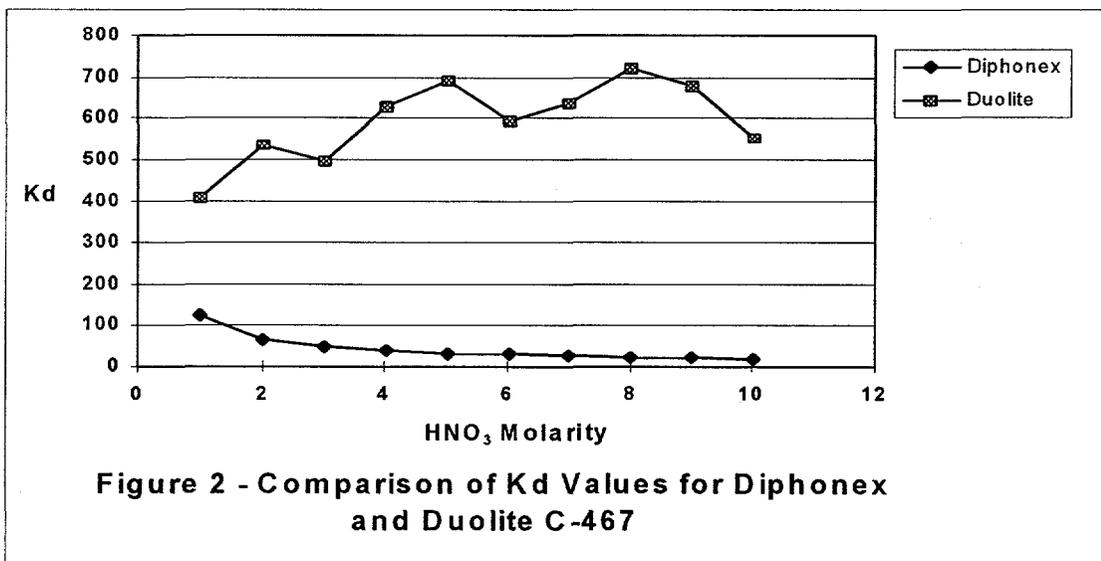
(Labindustries, Berkeley, California) at ~20 rpm for one (1) hour. At that time, the liquid was drained from the column into a scintillation vial, mixed, and then sampled twice for counting in a liquid scintillation counter (LSC). The LSC used was a Packard 2200 Liquid Scintillation Counter (Packard Instrument Co., Meridan, Connecticut). Each LSC sample (0.1 mL) was mixed with 6 mL water and 14 mL of Ultima Gold-XR™ (Packard Instrument Co.) scintillation cocktail. Samples were counted for 10 minutes each. Background samples consisted of 6 mL of water and 14 mL of Ultima Gold-XR™. The average background data (in counts per minute or CPM) was subtracted from each sample count, the CPM of the two samples for each acid molarity were averaged and then the Kd calculated and plotted as a function of acid molarity using an Excel™ (Microsoft Corporation, Redmond, Washington) spreadsheet. All experiments was repeated at least once.

The Kd value is defined in equation 1.

$$Kd = \frac{\text{(cpm (corrected) remaining in the solution after 1 hour)/(gram of resin)}}{\text{(1) (cpm(corrected) contained on the resin after 1 hour)/(mL of solution)}}$$

Results

The Excel plots indicate the variations of the Kd values as a function of acid molarity. These plots of Kd versus acid molarity are one method for comparison of the uranium removal efficiency for the various resins and also give some indication of "ideal" acid molarity for loading and elution of each resin. The initial resultss for the various resins listed above (Table I) are shown in Figures 2 and 3.



From these two figures, one observes the high Kds obtained for both Duolite C-467 and Diphonex, although the loading conditions (acid molarity) are quite different! It is also apparent that there may not be an "ideal" condition of simply changing the acid molarity for elution of the uranium from the Duolite C-467.

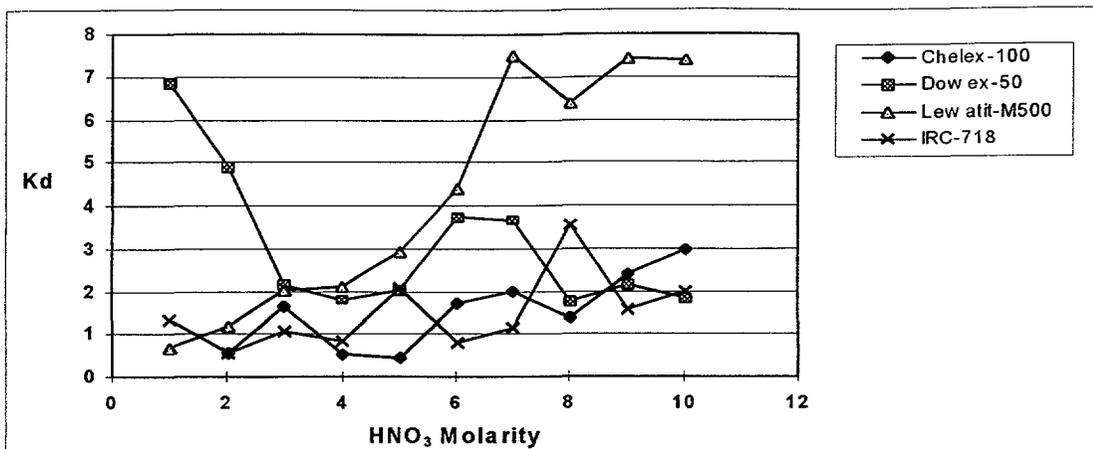


Figure 3 - Comparison of Kd Values for Chelex-100, Dowex-50, Lewatit-M500, and IRC-718

A cross check of the LSC data was done using a Varian Inductively Coupled Plasma, Atomic Emission Spectrometer (ICP-AES). The results obtained by this method were quite similar and a comparison of the two data sets for Duolite C-467 resin is shown in Figure 4.

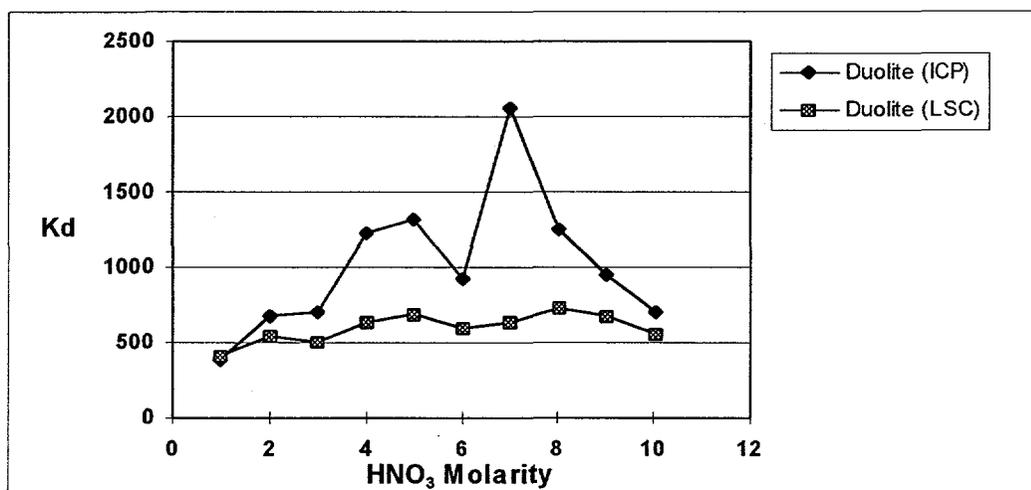


Figure 4 - Comparison of Kd Values Obtained for Duolite C-467 by ICP and LSC

Discussion

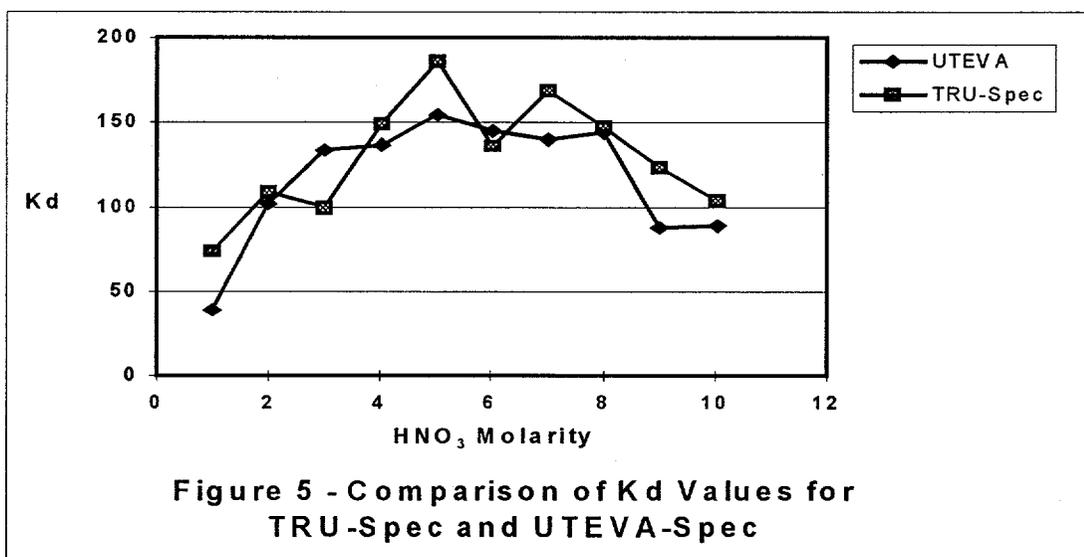
Another part of the Uranium Technology Development Program, lead by Kent Abney, Chemical Science and Technology Division, Group CST-11, is investigating commercial anion resins for the removal of uranium from nitric acid streams. (2) This work is based upon the fact that uranium (VI) or uranyl nitrate is typically an anion of the form $UO_2(NO_3)_6^{4-}$. A relatively new resin, Reillex™ HPQ (Reilly Industries Inc., Indianapolis, Indiana) has been demonstrated and is currently in use at the Los Alamos Plutonium Facility, TA-55, for recovery of plutonium from either nitric acid or

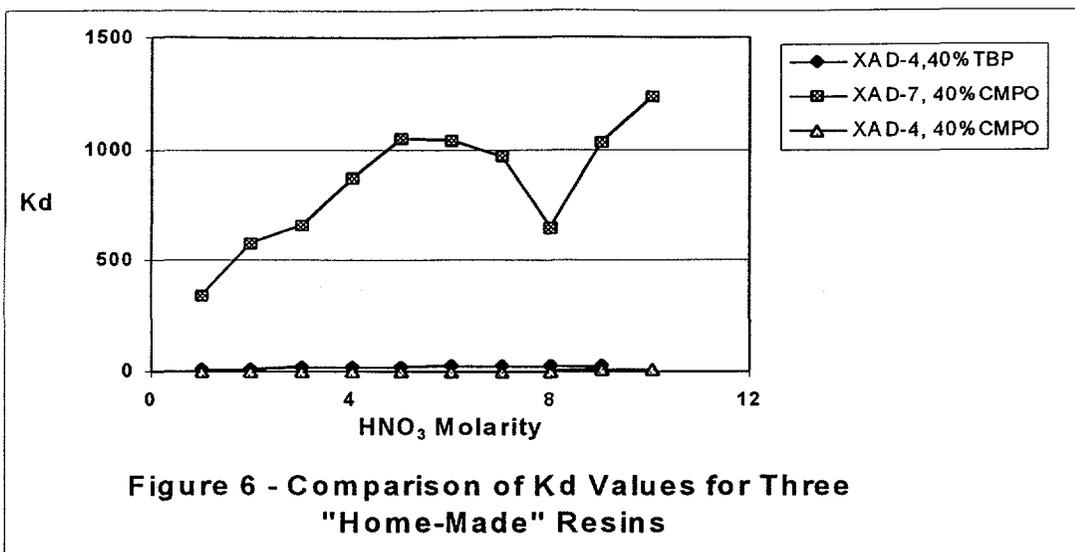
hydrochloric acid media. (3,4) It has also been reported that mixtures of strong base and strong acid resins, typically a mixture of anion and cation resins is quite useful for the removal of uranium at the ppm level from drinking water. (5) In addition to the low maximum Kd values obtained with both Dowex-1 (Kd = 20-24) and Reillex™ HPQ (Kd = 10-12),) this reference was a factor for trying a strong cation resin and in searching for other types of commercial resins which may have higher Kd values. This search was also directed towards experimentation with extraction chromatographic resins, namely TRU-Spec and UTEVA-Spec, and three other extraction chromatographic resins which we prepared in our laboratory.

Extraction Chromatography Resin Experiments

As mentioned above, experiments were done with both TRU-Spec, which contains 13% Octyl(Phenyl)-N,N-Diisobutyl Carbamoyl Methyl Phosphine Oxide (CMPO), and 27% TBP, UTEVA-Spec, containing 40% diamyl amyl phosphonate (DAAP). Also three resins of a similar nature were synthesized in our laboratory. These resins were 40% CMPO loaded onto both Amberlite XAD-7 and XAD-4 (Rohm & Haas Co., through Aldrich Chemical, Milwaukee, Wisconsin), with the third resin containing 40% TBP loaded onto XAD-4. The major difference between the XAD-4 and XAD-7 is the surface area and pore size, with XAD-7 having a larger pore size but a smaller surface area. XAD-4 is a polystyrene absorbent with a surface area of 725 m²/g and a pore size of 40 Angstroms. XAD-7 is a polyacrylate absorbent with a surface area of 450 m²/g and a pore size of 90 Angstroms. (6)

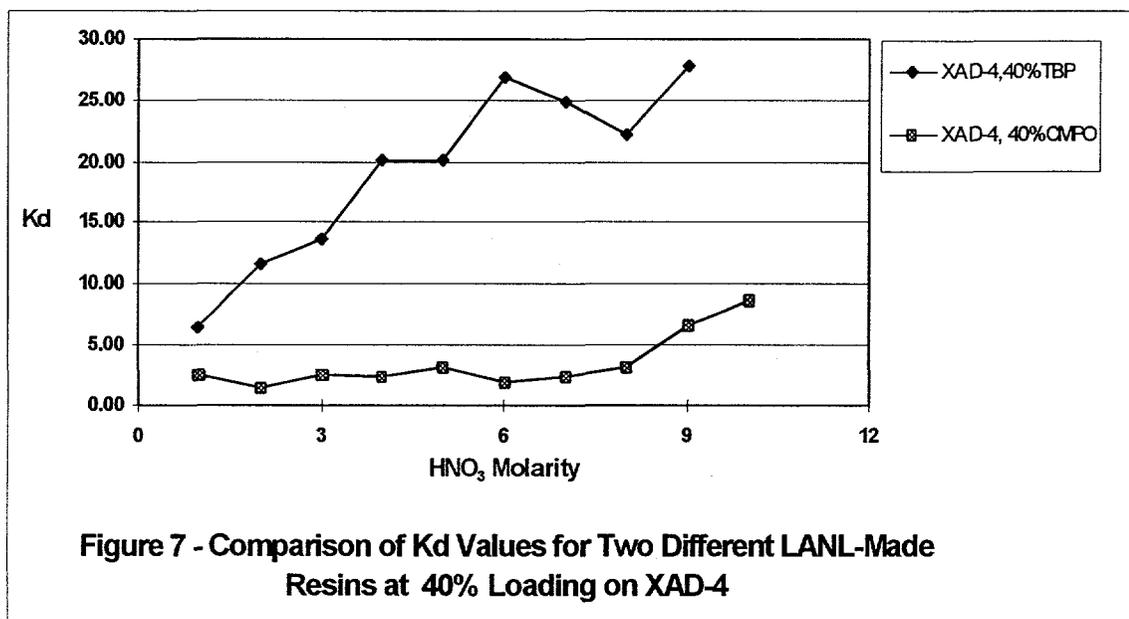
Although these resins are relatively expensive, they do offer the possibility of higher Kds and the fact that they can be eluted using low molarity acids after loading from high molarity acid. The Kd determinations were done in a similar fashion to those described above and the Kds were calculated and plotted using Excel. The plots of Kd versus acid molarity are shown in Figures 5 and 6.





Discussion

From Figures 5-7, we observe that relatively high Kds are obtained and the Kd increases with increasing acid molarity. Also the small Kds at low acid molarity indicate the possibility for elution of the uranium from these resins using low molarity (0.001-0.1M) HNO₃. These resins behave more like typical anion resins because they can be loaded at high acid molarity and can potentially be eluted with low acid molarity. Elutions of this type have great potential due to the fact that no other chemicals must be added, and the eluate containing the uranium is readily amenable to either peroxide precipitation or further neutralization to form ammonium diuranate. Further discussion on this subject occurs in the following section on elution experiments.



Elution Experiments

Once the resin is selected for sorption of uranium, then removal of the uranium from the resin becomes very important. If the uranium can not be removed or eluted from the resin, then the candidate resin must be rejected. It has been reported that uranium can be eluted from both Duolite C-467 and Diphonex using sodium carbonate (Na_2CO_3). (7,8) From Figures 2 and 3, we observe that it should be possible with some of these resins, particularly the Dowex-50 and Diphonex, to elute uranium from the resin by simply changing the acid molarity. Of these two, only Diphonex was tried due to the very low maximum K_d exhibited by Dowex-50. It should be noted here that Dowex-50 is a cation resin while Dowex-1 is the corresponding anion resin. It is typical for an anion resin to be loaded at high acid molarity(7-9 M) and eluted at a low acid molarity (0.1-1M). This is attributed to the complexation of the uranyl ion by nitrate anions at high acid molarities.

Table II gives an outline of the various resins and elution combinations that were tried. The elution experiments described below all used other complexing agents such as oxalate, citrate and ammonium, or sodium, or potassium carbonate. At this point only three resins were being tested. These were chosen as the top three resins based upon their maximum K_d values. A pre-evaluation step was done for these eluant/resin combinations, where 0.5 grams of resin, 0.1 mL of uranyl solution and 5 mL of 4, 5 or 6M acid were mixed for an hour, then the solutions was drained and 5 mL of the desired eluant was added to the tube and rotated again for 30-60 minutes. A mass balance was obtained using the LSC data from the original solution, the solution after loading, and the eluted solution. If these results were promising, then columns were loaded as just described. Instead of rotating the eluant solution for an hour, it was passed through the column in 3-5 mL aliquots and each fraction was counted by LSC methods.

Table II - Eluants Tested with the Various Resins

Eluant	Resin		
	Duolite C-467	TRU-Spec	UTEVA-Spec
0.0001-0.1M HNO_3	x	x	x
Water	x	x	x
Oxalic Acid, 0.1-1.0M	x	x	x
Ammonium Carbonate, 0.1, 0.5 & 1.0M	x	x	x
Sodium Carbonate, 0.1, 0.5 & 1.0M	x	x	x
Potassium Carbonate, 0.1, 0.5 & 1.0M	x	x	x
Citric Acid, 1.0 M	x	x	x
Tiron	x	x	-----

As expected, the elution of Duolite C-467 was quite easily accomplished using either 1M ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$), or sodium carbonate (Na_2CO_3). A

typical mass balance plot for elution of Duolite C-467 using 1 M Na₂CO₃ is shown in Figure 8.

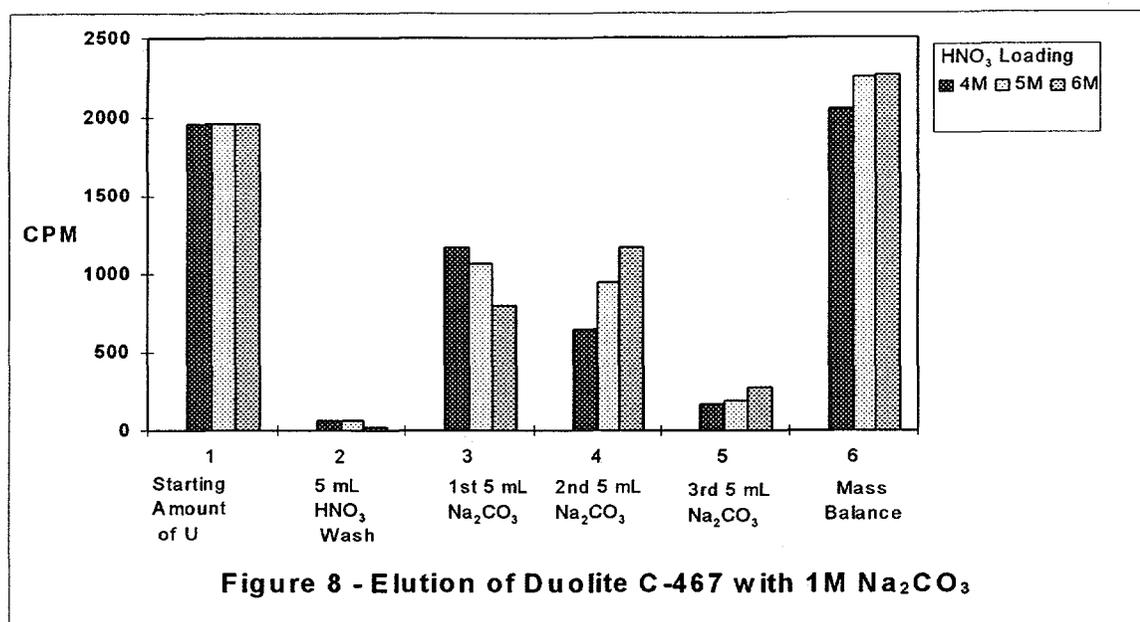


Figure 8 - Elution of Duolite C-467 with 1M Na₂CO₃

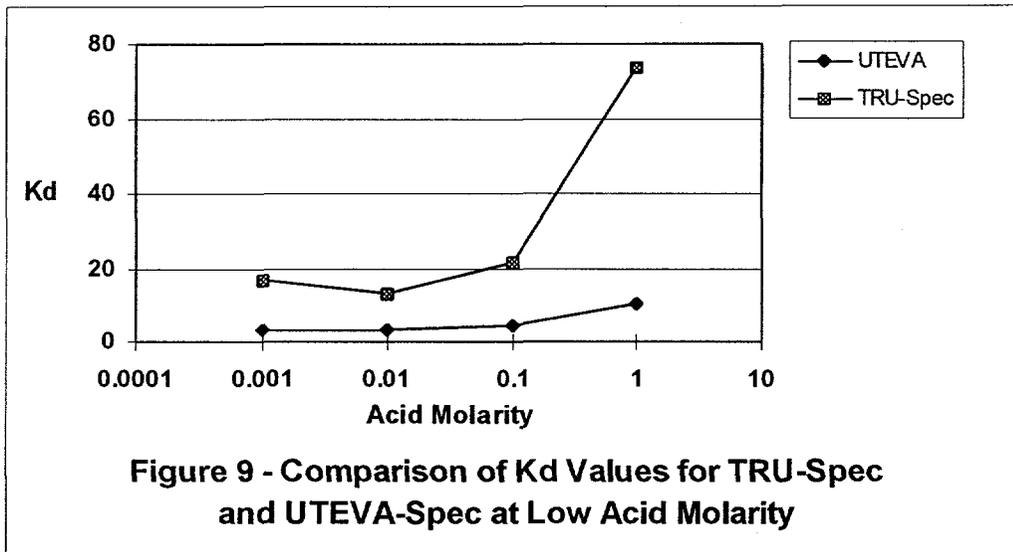
A major problem was encountered while using the carbonate solutions for elution of the uranium from the Duolite resin. This was the formation of carbon dioxide (CO₂), which occurred due to reaction between the Na₂CO₃ and the acid remaining in/on the resin from the loading step. A water wash was instituted between the acid wash step and the Na₂CO₃ elution step. At least 2-3 column volumes were required to reduce the acidity to a point where the formation of gaseous CO₂ was minimized. The use of a wash step appeared to work quite well on the small columns. However, when large columns (4 cm diameter, 60 cm long) were used, the Duolite C-467 retained sufficient acid such that it was almost impossible to prevent the formation of large gas bubbles which disrupted the column, led to channeling, and incomplete elution (therefore poor mass balance) of the uranium loaded onto the resin.

Attempts to elute the uranium from Diphonex by simply using acid of a different molarity from the acid molarity used for loading the uranium onto the column were not very successful. Although the uranium could be stripped by using high molarity acid after loading at ≤1M acid, the K_d is still high enough that a good mass balance could not be attained.

As shown in Table II, elution experiments were also done for both TRU-Spec and UTEVA Spec. As mentioned above, both of these resins exhibit characteristics that are quite similar to ordinary anion-exchange resins, namely that the uranium can be loaded onto the resin at high acid molarity and then eluted with 0.01-0.05M acid. This fact was supported by additional experiments using 0.0001-0.1M HNO₃ and water. In these cases, contact experiments indicate that the K_d values for both of these resins approached zero. This data is shown graphically in Figure 9.

Of the various eluants tried (Table II), only the various carbonate compounds were successful in achieving complete elution of the sorbed uranium. The HNO₃ and water elutions, as well as oxalate and citrate did not always give complete elution of

the uranium from the resins. Of the carbonate solutions, there was incomplete elution of the uranium for Na_2CO_3 solutions $<1\text{M}$, and the 1M solutions did not vary much with the different cations. Therefore, other than the gas evolution problem mentioned above, a 1M solution



of either sodium, potassium or ammonium carbonate gave complete elution and therefore a good mass balance.

Sorption of Other metals by Duolite C-467

Sorption of other metals, in particular the transition metals, which may be in the feed stream, was of interest because these metals might occupy potential uranium binding sites which would thus limit the amount of uranium that could be sorbed. A series of experiments were performed using single element solutions of cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), nickel (Ni), and neodymium (Nd). Most of these elements would arise from the corrosion of stainless steel and Nd was added to represent the lanthanide elements. Data is already available, through Eichrom Industries, on the sorption of various elements by both TRU-Spec and UTEVA-Spec resins. (9) In general, only Fe(III) is sorbed by the Eichrom resins, especially TRU-Spec, but even this can be minimized by reduction of any Fe(III) to Fe(II) using either hydrazine or ascorbic acid. One-tenth molar solutions of the metals mentioned above were made from the nitrate salts. These experiments proceeded in a similar manner as described earlier, where 0.5 grams of resin is contacted with 5 mL of a 4M HNO_3 solution contained a specific element. After rotation for an hour, the solution was drained from the column and analyzed by ICP-AES techniques.

Results

From the experiments outlined above, Table III shows that small amounts of Ni and Cr are removed while substantial amounts of both Fe and Nd are removed from 4M HNO_3 by the Duolite C-467 resin. It appears that both Fe and Nd would be

problem elements, however Nd is seldom in the feed streams that are encountered at Los Alamos. Fe will be contained in all of them and an appropriate feed treatment step is necessary to hold the Fe in solution as Fe(II). Experiments were performed to determine if Fe actually occupied sites that would normally sorb uranium. However, the results were unclear, and it was difficult to determine if the uranium sorption capacity was actually decreased in the presence of high amounts of Fe.

Table III - Extraction of Various Transition Metal Elements by Duolite C-467

Element	% Removed
Fe	94.0
Nd	82.0
Ni	2.3
Cr	2.0
Co	0.0
Cu	0.0

Flow Experiments

The flow experiments were done using either 1-inch diameter fritted glass columns or 1-2 inch diameter glass columns (Kontes Glass, Vineland, New Jersey, through Fisher Scientific) with a 10 micron nylon resin support. The solutions were simply poured into the top of the column, and gravity was used to provide the flow. This flow could be adjusted for the Kontes columns by insertion of a 1/4-28 machine screw into the bottom fitting. By adjusting the screw in or out, the flow could be adjusted from ~2 mL/min. to a flow >10 mL/min. The fritted glass columns did not have any flow control but nominal flow rates of 5-6 mL/min. were achieved. The top of the resin was covered with a 1-2 inch thick plug of glass wool to prevent agitation of the resin upon introduction of the feed (or any other) solution. The resin was packed into the column using water and then 600 mL of 4-6M acid was passed through the column. 600 mL corresponds to approximately 1-1.5 column volumes in the larger 2 inch-diameter columns.

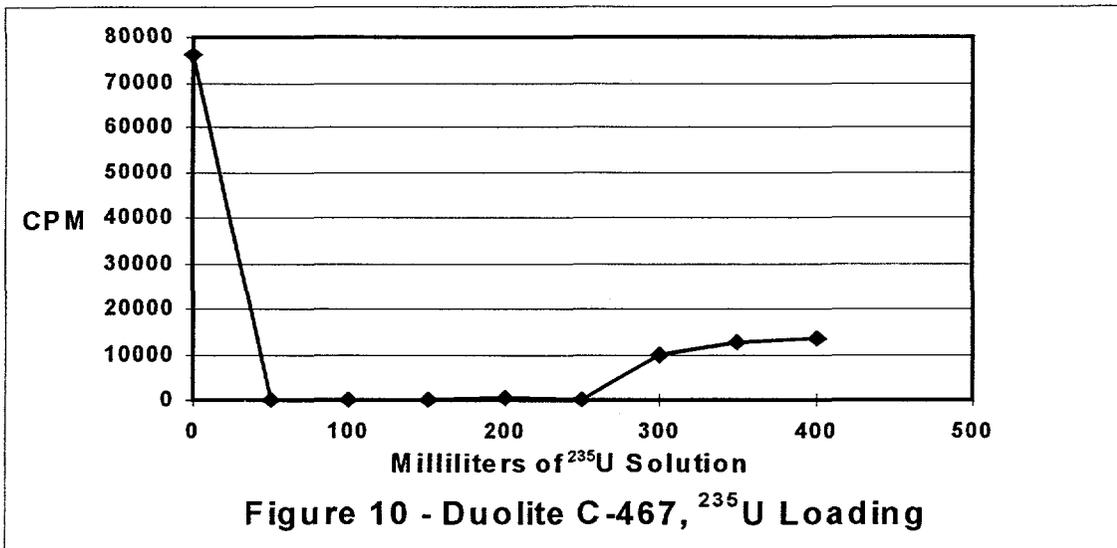
The entire procedure for loading, washing and eluting the columns is outlined below:

1. After the resin has been washed with the acid, as mentioned above, the 4-6M feed solution is poured into the column and the flow rate adjusted to ~3 mL/min. The solution is allowed to pass through the column until ~0.25 inch of liquid remains above the top of the resin,
2. 600 mL of 4-6M acid is poured into the top of the column (to wash away any impurities that are not sorbed by the resin) and allowed to pass through the resin until only 0.25 inch of liquid remains above the resin,
3. Two 600 mL batches of water are then run through the column to dilute the acid to approximately pH 2,
4. 600 mL of 1M Na₂CO₃ is then poured into the column and 100 mL aliquots are collected, until the yellow color of U(VI) becomes visible,

at which time smaller aliquots can be collected to better define the elution curve. Additional Na_2CO_3 is used until 1.5-2.0 L have been passed through the column.

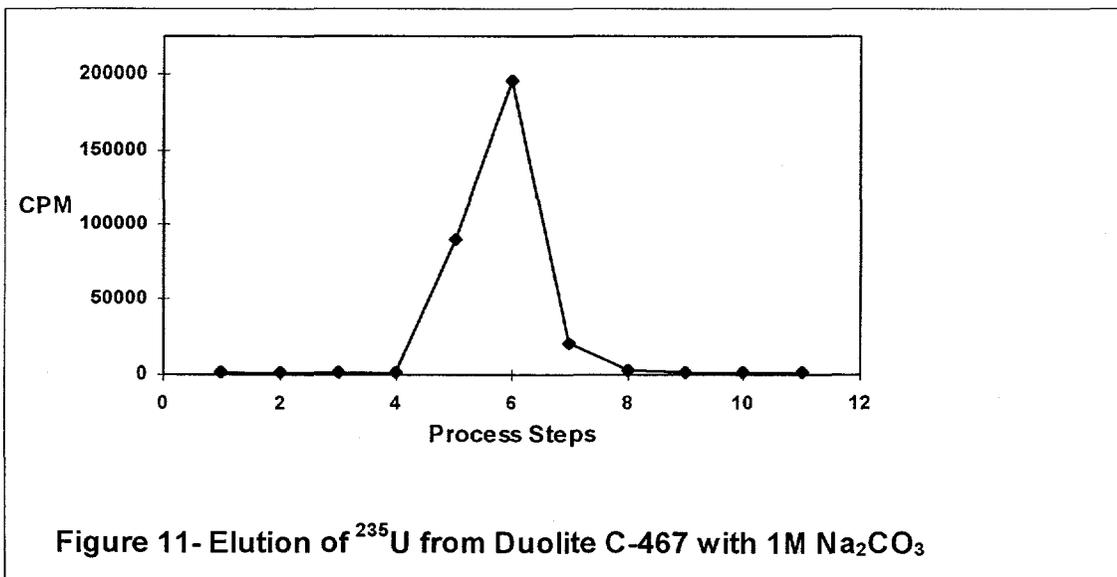
Results

A proof of principle experiment using several grams of ^{235}U was done in a one-inch, fritted glass column as a trial before larger scale (tens of grams) experiments. The results from loading are shown in Figure 10. One advantage to using HEU is that the



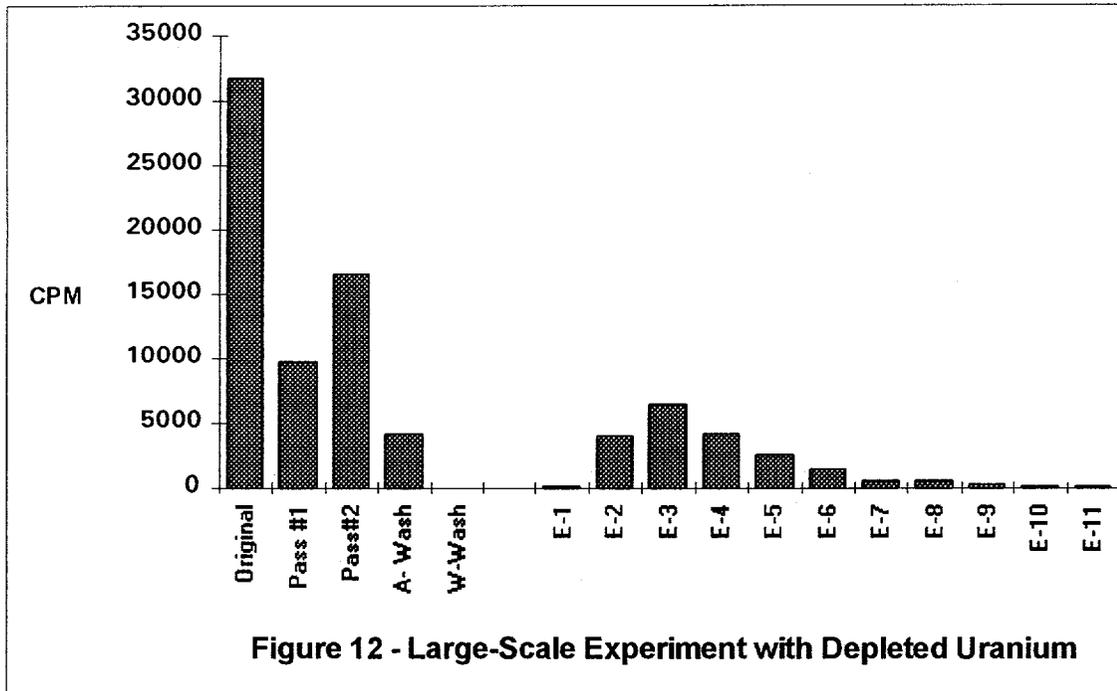
LSC data exhibits much greater CPM for a given sample size and therefore it is easier to determine break-through and the quantity of uranium that is "lost" in the acid and water wash solutions. Break through is evident from the curve.

The elution of the uranium from the column using 1M Na_2CO_3 is shown in greater detail in Figure 11. A volume of ~50 mL passes through the column before



the uranium begins to elute from the resin. This is partly due to simple neutralization of any remaining acid trapped in-between the resin beads (or extracted by the resin) and secondly, movement of the uranium from the top of the column to the bottom exit. The elution peak is quite sharp without tailing.

The larger-scale experiments gave similar results as shown in Figure 12.



However, the resin apparently extracts some acid and neutralization of this acid using 2-2.5L of water washes, was unsuccessful. Addition of Na_2CO_3 resulted in the formation of large CO_2 bubbles which virtually stopped the flow of liquid through the column and lead to the formation of channels. Under these conditions, elution of the uranium was difficult and the mass balance attained in these experiments ranged from 60-96%, therefore the uranium recovery from the resin was usually inconsistent and at times, extremely poor. These large bubbles would be a cause of great concern in a sealed or pressurized system. It is obvious that this is a dangerous situation and therefore would not be satisfactory for operation in either a hood or glove box. The problem can be overcome by sorbing the uranium onto the resin in the normal fashion, or by simply adding resin to a uranium solution in an Erlenmeyer flask, followed by separation of the resin from the solution by filtration. Acid and water washes can be done in either a column or in a flask followed by neutralization of any residual acid by Na_2CO_3 . The neutralization step must be done in either an open Erlenmeyer flask or beaker so that any CO_2 evolved, can easily escape. The Na_2CO_3 solution and resin are then poured into the glass column and the uranium is eluted from the resin into an appropriate flask. However, this type of operation is not really acceptable for process operation due to the large amount of hands-on labor required and the limitations imposed in handling large quantities of resin and/or solution.

Future Experiments

Current experiments are pursuing both new resins for removal of uranium from both nitric and hydrochloric acid media. Larger scale experiments are planned using Diphonex and either TRU-Spec or UTEVA-Spec. These experiments will eventually reach the 100 gram-scale. Kent Abney is investigating some newly developed resins from Reilly Industries for use with HNO_3 while we are looking at resins to give optimum performance for HCl media. Extraction of uranium from HCl solutions is much easier because of the formation of strong anions in HCl media and standard anion-exchange resins usually exhibit K_d values >1000 for the extraction of uranium.

(2)

Waste stream polishing should also be easier with HCl solutions. The extraction chromatography resins, i.e., TRU-Spec and UTEVA-Spec, can decrease the uranium content in HNO_3 media down to the discard limit. At Los Alamos, the discard limit for solutions to the Liquid Waste Treatment Facility, TA-50, is 0.5 microcuries per liter. For HEU, this translates to ~ 7 ppm of uranium in solution. Removal of the uranium to this level is difficult using ion-exchange techniques. However if the waste stream can be concentrated by a method, i.e., reverse osmosis (RO) or some other technique, then the discharge limits can be met. Experimentation into various methods for final waste stream polishing is being done in conjunction with the various processes being instituted and with the personnel performing the ion-exchange experimentation.

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