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PRECIPITATION OF AMMONIUM DIURANATE**

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SUMÁRIO

Descreve-se um processo de descontaminação de urânio de impurezas constituídas por elementos de terras raras de alta seção de choque para absorção de neutrons lentos.

Na fase de precipitação do diuranato de amônio com amoníaco, adiciona-se, propositalmente, um elemento (ítrio , p.ex.) cujas propriedades químicas sejam semelhantes às das terras raras, mas que tenha baixa seção de choque para absorção de neutrons lentos. As terras raras e o ítrio são então complexados, antes da precipitação do diuranato de amônio, com ácido etilendiaminotetraacético (EDTA). O ítrio, na forma de complexo com o EDTA, age como carregador-retentor ("hold-back carrier") para os elementos de terras raras mantendo os mesmos, também complexados com EDTA, em solução.

São apresentados os valores dos fatores de descontaminação que são obtidos para as terras raras de alta seção de choque.

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RESUMÉ

On présente une méthode pour décontaminer l'uranium d'avec les terres rares possédant des hautes sections de choc pour l'absorption des neutrons thermiques.

Si l'on augmente la quantité totale de terres rares présentes dans des solutions d'uranium par la addition d'yttrium (un élément que se comporte comme les terres rares et qui a une faible section de choc pour neutrons thermiques) tout en complexant les terres rares et le yttrium par la addition de EDTA avant de précipiter l'uranium avec ammoniac, le facteur de décontamination pour les terres rares de haute section de choc se trouve augmenté.

On réporte les facteurs de decontamination pour les terres rares au cours de la précipitation de l'uranium comme diuranate d'ammonium.

SUMMARY

A way of decontaminating uranium from rare earths of high neutron absorption cross section is described. Increasing the total amount of rare earths present in uranyl solutions, by adding yttrium (a rare earth-like element of low neutron absorption cross section) and complexing the rare earths and yttrium with ethylenediaminetetraacetic acid, prior to precipitating uranium with ammonia, will raise the decontamination factor for the rare earths of high neutron absorption cross section. Yttrium will act as hold-back carrier for the rare earths. Decontamination factors for those rare earths, on

precipitating uranium as ammonium diuranate, are reported.

I. INTRODUCTION

Precipitation of uranium as ammonium diuranate (ADU) is a common unit operation on the purification of that element for nuclear purposes. Whatever the purification process used, such as solvent extraction or ion exchange, one of the final steps, almost universally adopted, and the one that precedes the operation of reduction of uranium VI to uranium IV, such as in UO_2 , or to metal, is precipitation of uranium with ammonium hydroxide. However, this precipitation is not specific for uranium and quite a large number of elements, if they are present together with uranium, will be entrained or coprecipitated with the ammonium diuranate precipitate.

One way to avoid the coprecipitation of a good number of impurities is to add a complexing agent, to the uranyl solution to be precipitate with ammonia, that will form complexes with the impurities and will not complex, or only to a very small extent, the uranyl cations. This procedure, largely used in analytical schemes (1), has been developed for precipitation of large amounts of uranium, in pilot plant size operation, by Lima and Abrão using ethylenediaminetetraacetic acid (EDTA) as complexing agent (2), and by Brill and Krumholz, in laboratory scale, using EDTA and Hydroxiethylenodiaminetriacetic acid (3).

Some Brazilian uranium sources, such as sodium diuranate obtained as by product from monazite sands do have rare earths as contaminants on the average-amount of 0.2% for total rare earths, 1% being the maximum value. One tenth of

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this proportion is represented by the rare earths of , high thermal neutrons absorption cross-sections, i.e., samarium , europium, gadolinium and dysprosium (3).

Precipitation of uranium as ammonium diuranate will not purify that element from rare earths, which will be entrained, practically quantitatively, by the diuranate precipitate. However, if a complexing agent such as EDTA is present the rare earths may be kept, partially, in solutions as soluble chelate compounds.

Taking into account that a purification procedure for uranium, such as solvent extraction or ion-exchange, may give decontamination factors, for rare earths elements, from 10^3 to 10^4 one may end up with a product containing 2 to 10 parts per million (ppm) of total rare earths, before precipitation of the ammonium diuranate. A product such as this would have around 0.2 to 1.0 ppm of the rare earths of high neutron cross-section. This amount is high for uranium to be used in natural uranium reactors.

In order to calculate the contribution given by such an amount of rare earths to the boron equivalent of uranium, it will be assumed, for the sake of calculation and as first approximation, that the proportion of rare earths of high cross section, in the purified uranium, is the same as the proportion of those rare earths in the monazite from which the uranium was separated. In general this proportion is 50:1:20:10 for Sm, Eu, Gd and Dy, in accordance with reference (3). The

contribution of these elements to the boron equivalent* of a uranium sample having 1 ppm of those rare earths is given in Table I.

TABLE -I-

Contribution to the boron equivalent (BE) of a uranium sample having 1 ppm of rare earths of high neutrons absorption cross sections.

	ppm	A**	S***	BE
Sm	0.62	150.4	5,500	0.33
Eu	0.01	152	4,600	0.004
Gd	0.25	156.9	46,000	1.05
Dy	<u>0.12</u>	162.5	1,100	<u>0.01</u>
	1.00			1.394

** Atomic weight
*** Cross section in barns

The upper limit for total boron equivalent for natural metallic uranium is set to a value of 2.88 ppm, and 0.9 to 1.2 for UO_2 (5). A contribution of 1.39 given by the rare earths listed in Table I is a too high value taking into account that other elements, besides the rare earths group, would be present and would contribute to the total boron equivalent.

* The definition of boron equivalent (BE) is

$$BE = m \frac{s / A}{69.77}$$

Where m is the mass in grams, of impurity, per 10^6 grams of uranium (ppm), s the cross section for thermal neutron absorption (in barns) of the element impurity, and A its atomic weight.

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Such being the case, it is seen that even when the purification procedure is such that the decontamination factor for rare earths is 10^4 , it is of high interest to devise a way of increasing the decontamination factor, which is practically equal to unity for the operation of precipitating uranium as ammonium diuranate, from 1 to 100 or even larger, if possible.

II. Entrainment of Rare Earths, Complexed With EDTA, by Ammonium Diuranate Precipitate.

If rare earths salts are present in solutions of uranyl nitrate or uranyl sulphate, even on the level of parts per million, and ammonia is added to the solution, the rare earths elements will be entrained by the precipitate of ammonium diuranate (ADU) in a quantitative way, practically.

However, as said before, if a chelating compound such as EDTA is present in the uranyl solution, the rare earth elements will be complexed and the amount that will coprecipitate with the ammonium diuranate is less than when no EDTA is used, the percentage of coprecipitation depending on the proportion of rare earths present. The percentage coprecipitated is very large for small amounts of rare earths originally present, such as when this amount is 2 ppm. When the amount of rare earth elements increases, the percentage coprecipitated with ADU, in the presence of EDTA, decreases. Also, it was observed that decontamination factors would depend on the amount of EDTA used, being larger for larger amounts of EDTA.

Since decontamination factors is larger for uranium containing, for instance, 100 ppm of total rare earths, as com

pared to uranium containing 2ppm of total rare earths it was thought that a process might be developed that would diminish the coprecipitation of high cross section rare earths and lowering, in this way, the contribution of these elements to the total boron equivalent for the impurities present in the uranium compound.

Suppose that a uranyl solution containing 2 ppm of total rare earths, one tenth of it, i.e., 0.2 ppm being represented by rare earth elements of high neutron cross section, is treated with ammonia in the presence of EDTA to complex the rare earths. Owing to the small amount of rare earths the decontamination factor will be close to one.

However, owing to the similarity of chemical behavior of the rare earth elements and of close values for stability constants of the complexes of rare earth elements and EDTA, it might be expected that the behavior of two EDTA-complexed rare earths, one of high neutron cross section (such as europium) and other of low neutron cross section (such as yttrium), on coprecipitation, would be the same.

In fact if yttrium is added in such an amount that total rare earth concentration will increase from 2 ppm to 100 ppm and if now the ammonia precipitation of uranium, in the presence of EDTA, is carried out, a decontamination factor from 50 to 1,000, depending on the concentration of the uranyl solution, may be obtained for total rare earth elements. The total amount of rare earth will not be diminished since 100 ppm, with a decontamination factor of 50, will be reduced to 2 ppm. However, the high cross section rare earth elements, originally in the amount of 0.2 ppm, will be decreased to $0.2/50=0.004$ ppm.

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The effect on the boron equivalent, of such an operation, will be of lowering the contribution, given to it by the high cross section rare earths, from 1.39 to $1.39/50=0.03$. Since the added rare earth element, yttrium, has a very low cross section (1.4 barns) as compared to samarium, europium, dysprosium and gadolinium, its contribution to the final total boron equivalent will be of no significance, amounting to about 0.0001 ppm in boron.

III. Decontamination of Rare Earth Elements of High neutron Absorption Cross Section.

In order to study in detail the coprecipitation behavior mentioned in II in such a way to take profit of it to decontaminate uranium from rare earths of high cross section, experiments were carried out with solutions of uranyl nitrate at the concentration of 5g/l, 25g/l, 50g/l and 125g/l; all concentrations are in terms of U_3O_8 .

To each uranyl nitrate solution labeled europium Eu^* (as nitrate), was added. In order to increase the amount of rare earths, yttrium nitrate was added to give solutions containing from 5 to 600 ppm of $(Y_2O_3+Eu_2^*O_3)$, as referred to U_3O_8 .

In another series of experiments the rare earths concentration was increased by adding europium, instead of yttrium, in such a way to have solutions from 5 to 600 ppm of $(Eu_2O_3+Eu_2^*O_3)$.

To each solution an amount of EDTA corresponding to 10 times the stoichiometric amount of $(Y_2O_3+Eu_2^*O_3)$, or $(Eu_2O_3+Eu_2^*O_3)$ was added.

In order to study the effect of increasing amount of complexing agent, solutions of uranyl nitrate at the concentration 25g/l, 50g/l and 125g/l, in U_3O_8 , were prepared and containing 1 ppm of $Eu_2O_3^*$. Increasing amounts of EDTA, from the stoichiometric amount for $Eu_2O_3^*$ up to 750 times that amount, was used.

Europium, labeled with Eu-152-154, was used as the representative element for the high cross section rare earths europium, samarium, gadolinium and dysprosium.

IV. Reagents

Nitric acid, Merck - PA d= 1.90, 65%.

Ammonium hydroxide, Colombina, PA- d=0.9, 25%.

EDTA chemically pure (Geigy), stock solution 100g/l.

Uranyl nitrate solutions made up by dissolving atomically pure U_3O_8 , (Analysis in Table II), in nitric acid; pH of solution equal to 1.4.

Yttrium nitrate solutions made up by dissolving chemically pure Y_2O_3 (Orquima), in nitric acid; pH of solution 0.8.

Europium nitrate solution made up by dissolving atomically pure Eu_2O_3 , (Orquima), in nitric acid.

Labeled europium was prepared by irradiating europium oxide in a thermal neutron flux of about 10^{13} neutrons/seg.cm² from 8 to 16 hours. Before use, a cooling period of four weeks was observed in order that the 9.3 hours half-life isomeric state of Eu-152 would decay to, practically, zero.

TABLE -II-

ANALYSIS OF U_3O_8 USED					
Element	ppm	Element	ppm	Element	ppm
Ag	< 0.5	Mn	< 4	Er	< 0.02
Al	< 20	Na	29	Eu	< 0.01
B	0.1	Ni	< 4	Gd	0.05
Cd	< 0.5	P	< 50	Ho	< 0.02
Cr	< 4	Pb	< 3	Lu	< 0.02
Cu	15	Si	20	La	0.13
Fe	10	Ca	70	Nd	0.25
K	44	Th	< 0.5	Pr	< 0.1
Li	< 5	Ce	0.25	Sm	0.04
Mg	< 20	Dy	0.4	Tb	< 0.1
				Tm	< 0.03
				Yb	< 0.005

V - Equipment

Measurement of Eu-152-154 was made by using a single channel gamma-ray spectrometer, Nuclear Chicago Corp., Model 182A, coupled to a well scintillation detector Model DS5, also from Nuclear Chicago Corp.

VI - Experimental

Uranyl nitrate solutions were prepared with the required amount of labeled europium, the retaining cation yttrium and EDTA; pH was ascertained to 1.4.

In order to carry on precipitation, 100 milliliters of the above solution was heated in a water bath up 75-78°C. Ammonia gas was passed through the solution with constant agitation until the pH value of 7.5, as determined by universal indicator paper, was reached. Ammonia gas was produced by heating a concentrated solution of ammonium hydroxide, analytical grade.

When the pH reached 7.5 the flow of ammonia gas was stopped, the ammonium diuranate precipitate decanted for 15 minutes at the temperature of 75-78°C, and filtered through sintered glass of fine porosity, (Jeanaer Glas 17-G4). The precipitate was washed five times with 4 to 5 milliliters portions of ammonium nitrate solutions at 2% made alkaline with ammonium hydroxide. The volume of the filtrate was reduced by heating and made up to 10 milliliters from which aliquots of 2 milliliters were taken for counting.

The precipitate was dissolved with nitric acid 1:1, the resulting solution was concentrated and made up to 10 milliliters in volumetric flasks, from which aliquots of 2 milliliters were also taken for counting. Alternatively, the precipitate was dissolved and europium carrier was added to the uranium solution. Uranium was precipitated with hydrogen peroxide and filtered; europium in the filtrate was precipitated

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with oxalic acid and prepared for counting.

A blank sample of uranyl nitrate solution, with no labeled europium, was also submitted to the same procedure in order to subtract background counting given by uranium daughters, when such was the case.

VII - Results

Measurements of Eu-152-154 present in each fraction (precipitate and filtrate) were made by measuring the area under the photopeak at 0.340 Mev or by integral gamma counting-above 0.900 Mev.

The percentage of rare earth entrained with the precipitate of ammonium diuranate is given by

$$100 \times (\text{activity of coprecipitated Eu}^*) / \text{activity of added Eu}^*$$

The decontamination factor is given by:

$$\text{activity of added Eu}^* / \text{activity of coprecipitated Eu}^*$$

Table III gives the results for the experiments in which the amount of Eu_2O_3^* was from 2 to 430 ppm. EDTA was used in excess of 10 times the stoichiometric amount required to complex europium. Figure I is the graphical representation of the percentage of europium coprecipitated as function of the fraction of europium initially present.

TABLE -III

Decontamination Factor and Percentage of Coprecipitation for Europium as Function of Amount of Europium.

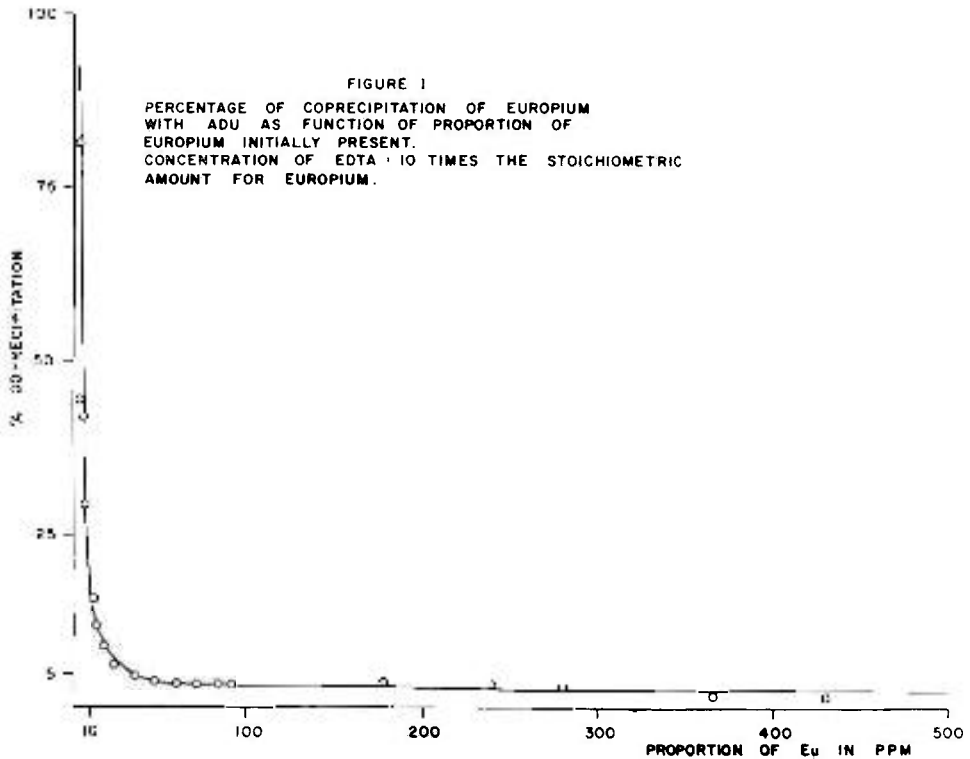
Eu ₂ O ₃ (ppm)	Coprecipitation (%)	DF*
2	81	1.2
4	44	2.2
6	29	3.4
12	12	8.3
18	9	11
24	5.5	18
36	5	20
47	3.7	27
60	3.4	29
72	3.3	30
84	3.2	31
92	3.0	33
280	2.9	35
366	1.7	59
430	1.5	67

*DF = Decontamination Factor

Quantity of EDTA used: 10 times the stoichiometric amount to complex the rare earth present.

Concentration of uranyl nitrate solution:

25g/l in U₃O₈.



As it can be seen from Figure I and Table III the percentage of rare earth coprecipitated is smaller, and the decontamination factor is larger, the higher the amount of rare earth initially present in the uranium. Evidently, this fact in itself will not diminish the amount of europium to levels below one part per million even when the decontamination factor is large.

If the total amount of rare earth is increased by adding yttrium to a uranium solution having, for instance, 4 ppm of europium as contaminant, large decontamination factors will also be obtained as in the case when only europium was present in high proportion.

In order to study the behavior of such a mixture, decontamination factors and percentage of coprecipitation were determined for mixtures of uranium and europium to which increasing amounts of yttrium, from 1 ppm up to 600 ppm, were added. The experimental technique was the same as the one described previously when europium was the sole rare earth present. Table IV and Figure II give the experimental results for decontamination factors and percentage of coprecipitation when yttrium was used as retaining ion.

Figure II shows that the percentage of europium coprecipitated, when using yttrium as retaining ion, follows the same general trend as when the only rare earth present was europium. It is seen that a very favourable decontamination of europium can be achieved by adding about 196 ppm of yttrium to the uranium sample containing 4 ppm of europium. The decontamination factor for total rare earth, Figure II, is about $1/0.025=40$. The amount of europium remaining in the uranium, after precipitation of ammonium diuranate, is $4 \text{ ppm}/40=0.1 \text{ ppm}$ of europium; yttrium will be present at the proportion of $196 \text{ ppm}/40 = 5 \text{ ppm}$. Since yttrium has a low neutron absorption cross section, this amount will not constitute any inconvenience to the natural uranium to be used as reactor fuel.

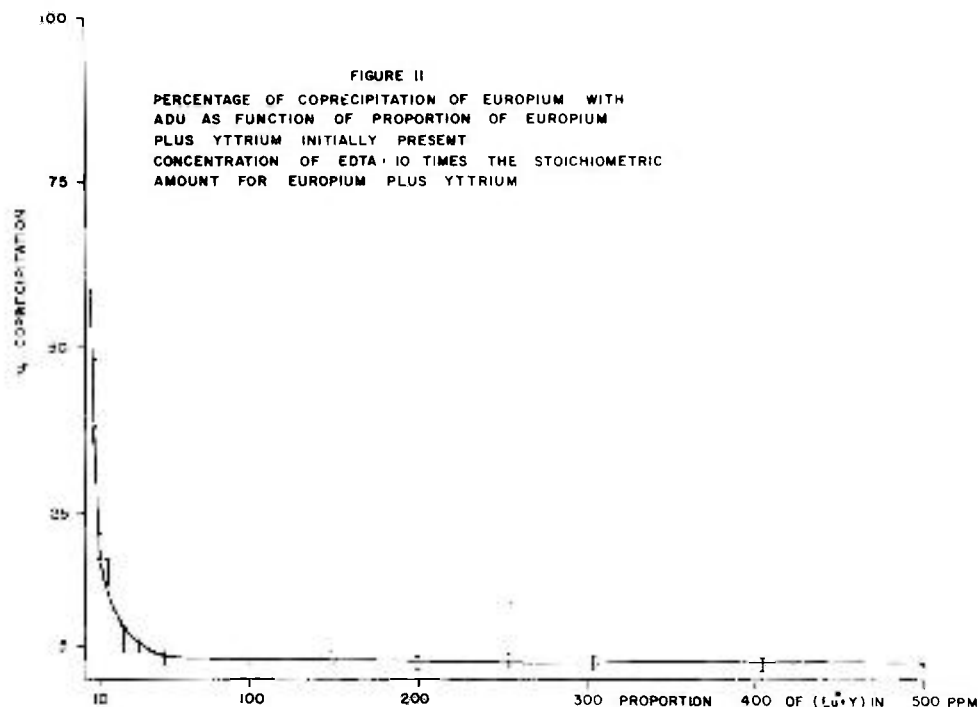
TABLE -IV-

Decontamination Factors and Percentage of Coprecipitation for Europium as Function of Amount of Yttrium Added.

Y_2O_3 (ppm)	Coprecipitation (%)	DF(a)
1	43	2
5	20	5
10	16	6
20	7	14
25	5	20
45	3	33
95	3	33
145	3	33
195	2.5	40
250	2.5	40
300	2.5	40
400	2.5	40
500	2.0	50
600	1.5	67

(a) DF: Decontamination Factor
Amount of $Eu_2O_3^*$: 4 ppm. Quantity of EDTA.
10 times the stoichiometric amount for total rare earth present. Concentration of uranyl nitrate solution: 25g/l in U_3O_8

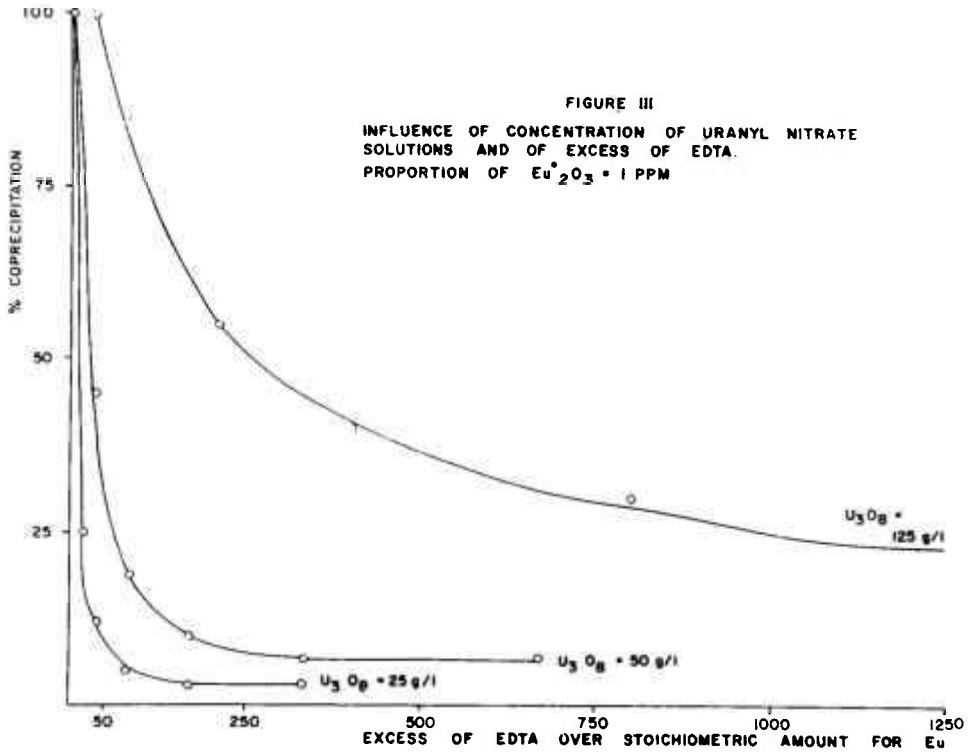
One notices from Figures I and II that the abscissa corresponding to the asymptotic values for coprecipitation, for both curves, is the same, i.e., about 50 ppm of europium, Figure I, or 50 ppm of europium plus yttrium, Figure II, giving the same percentage of total coprecipitation and hence the same decontamination factor, within experimental errors .



VIII. Influence of Amount of EDTA

In order to study the decontamination that might be reached by using increasing amount of EDTA, solutions of uranyl nitrate with constant amount of europium, i.e., one part per million, were prepared. EDTA was used in amounts from zero up to 750 times the stoichiometric amount required to complex the rare earth. Concentration of uranyl nitrate solutions were 25, 50 and 125 grams per liter in U_3O_8 .

Results are presented in Figure III. It is seen that the effect of using excess of EDTA is comparable to the effect of adding the rare earth of low neutron absorption cross section, yttrium. Coprecipitation is 100% when no EDTA is added.



The use of very large excess of EDTA is not convenient since uranyl nitrate solution in which the concentration of EDTA is too high, gives a bulky precipitate of ammonium diuranate very difficult to filtrate and to wash. Table V gives the volumes of precipitates of ADU precipitated from uranyl nitrate solutions of concentration 50 grams per liter and increasing amounts of EDTA.

TABLE -V-

Volume of Precipitate of ADU as Function of Amount of EDTA Present. Concentration of Uranyl Nitrate Solutions 50g/l in U_3O_8

EDTA/ U_3O_8 %	Volume of precipitate (milliliters)
0.02	11
0.2	11
1.0	16
2.0	20

IX. Influence of Concentration of Uranyl Nitrate Solutions

In order to study the influence of concentration of uranyl nitrate solutions on the entrainment of rare earth by the ADU precipitate, solutions were prepared with concentrations of 5, 25 and 50 grams per liter in U_3O_8 and 32 ppm of $Eu_2O_3^*$. The retaining ion used was yttrium and its proportion varied from 0 to 598 ppm of Y_2O_3 . The quantity of EDTA used was 10 times the stoichiometric amount required to complex europium plus yttrium.

Results are presented in Table VI and Figure IV. The general trends of the curves are the same as in the cases examined up to now, i.e., the larger the proportion of the retaining ion the smaller the percentage of coprecipitation and the higher the value for the decontamination factor. For a given value of retaining ion plus rare earth the percentage of coprecipitation is smaller the lower the concentration of uranyl nitrate solutions.

TABLE -VI-

Influence of concentration of uranyl nitrate solutions on percentage of coprecipitations

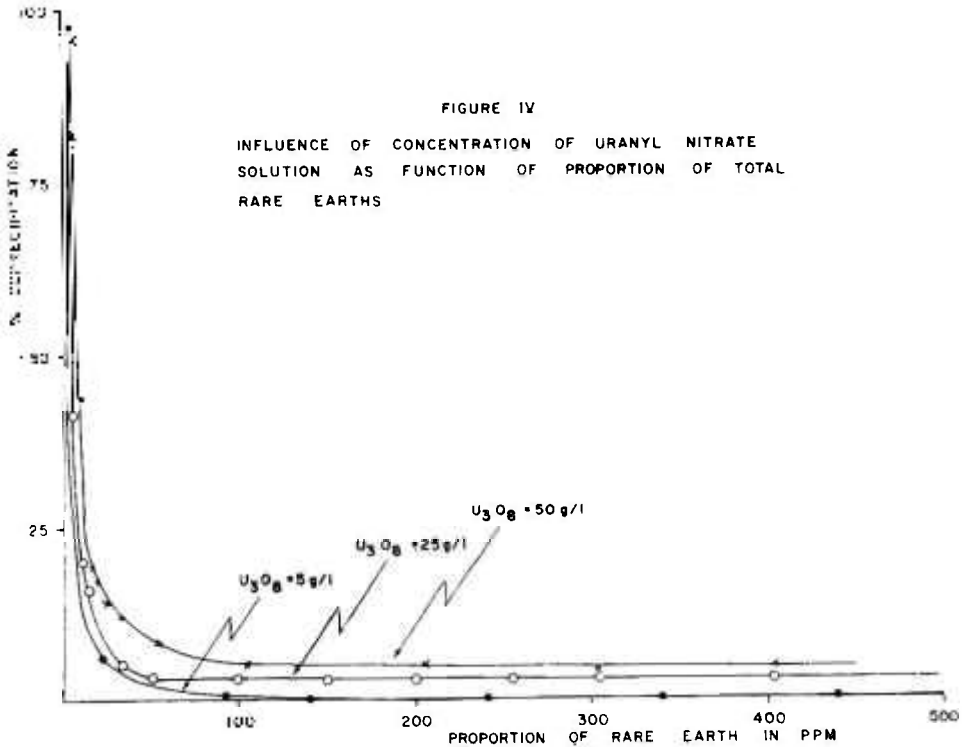
RE \ C	5	25	50
2	98%	98-99%	100%
5	20	50	80
10	13	20	35
15	10	15	19
20	6	10	17
30	4	6	13
40	3	4	10
50	2	3	9
60	1	3	8
70	1	3	6
80	1	3	5.5
90	0.5	3	5
100	0.5	3	5
150	0.3	3	5
200	0.2	2.5	5
300	0.2	2.5	5
400	0.1	2.5	4.5
500	< 0.1	2.0	4.5
600	< 0.1	1.5	4.5

Quantity of EDTA: 10 times the stoichiometric amount required to complex the rare earths present.

C - Concentration of uranyl nitrate solutions in grams per liter of U_3O_8 .

RE - ppm of $(Y_2O_3 + Eu_2O_3^*)$

Proportion of $Eu_2O_3^*$ = 32 ppm



X - Influence of Ageing of the ADU Precipitate

The influence of ageing of the ADU precipitate on the entrainment of the rare earth complexed with EDTA was studied by maintaining the precipitate and solution, after precipitation of ADU, in agitation for lengths of time that varied from zero up to 6 hours. Ageing took place at room temperature or at the temperature in which the ADU precipitate was formed. Concentration of uranyl nitrate solution was 25 grams per liter in U_3O_8 , proportion of rare earth 96 ppm of $Eu_2O_3^*$ and amount of EDTA equal to 10 times the stoichiometric amount required to complex the rare earth. Table VII presents the results.

TABLE -VII

Influence of Ageing of ADU Precipitates on the Entrainment of Rare Earth.

Time of ageing (hours)	Percentage coprecipitated	
	25°C	75°C
0	4	4
0.25	4.5	4
0.5	4	4
1.0	5	3
3.0	4.5	3
6.0	4	4

From Table VII it is seen that, within experimental errors, there is no influence of ageing of the ADU precipitate at room temperature or at precipitation temperature, i.e. 75°C.

XI. Conclusions

The proportion of rare earths of high neutron absorption cross section, i.e. samarium, europium, gadolinium and dysprosium, in uranium, can be reduced to levels below the hundredth of parts per million by complexing them with EDTA and adding a rare earth-like element of low neutron absorption cross section, such as yttrium, also as EDTA complex, to the uranyl nitrate solution prior to precipitating uranium with ammonia.

It is not necessary a large excess of EDTA over the stoichiometric amount required to complex the rare earths present. Five to ten times the stoichiometric amount is sufficient to give high decontamination factors. The expense in using excess of EDTA is not notable, since proportion of total rare earth, after solvent extraction or ion exchange purification procedures for uranium, is below 10 parts per million.

A very large excess of EDTA will have the same effect on the value of decontamination factors as of adding yttrium. However, the ADU precipitate formed in the presence of a large excess of EDTA is bulky and very difficult to filtrate. The best procedure is to use yttrium at the proportion of 100 to 200 ppm and an excess of 10 times of EDTA over the stoichiometric amount required to complex the retaining ion yttrium plus the other rare earth present.

Decontamination factors are larger the lower the concentration of uranyl nitrate solutions.

Ageing of the ADU precipitates, from zero up to 6 hours, will have no effect on the entrainment of the rare earth when ageing takes place at room or at the precipitation temperature.

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