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THE DECONTAMINATION OF URANIUM FROM FISSION PRODUCTS BY THE USE OF THE URANYL OXALATE PRECIPITATION REACTION

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CHEMISTRY - SEPARATION PROCESSES FOR PLUTONIUM AND URANIUM

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ABSTRACT

Decontamination factors of the order of 10^4 were obtained for β and γ emitters present as fission products when uranium was precipitated from 50 mC activity level solutions as uranyl oxalate under normal uranium yield conditions for three cycles (~60%).

Factors of the order of 10^3 were obtained by the use of this reaction with similar solutions under relatively high uranium yield conditions for three cycles (~90%).

The uranium peroxide precipitation reaction proved to be of no value on such solutions, yielding decontamination factors of only 17.1 for β emitters and 1.2 for γ emitters for three cycles.

References: P. A. No. CMR-8-4 (SRP#8); LA Notebook 2537.

I - Introduction

In connection with the RaLa program at this laboratory, it was proposed that 3 kg. of enriched uranium be substituted for the two tons of normal uranium now irradiated in the Hanford Pile to produce the required quantity of active Ba^{140}. This would relieve the problem of storing large volumes of highly active normal uranium solutions after the Ba^{140} had been removed. However, the monetary value of enriched uranium would necessitate its recovery and decontamination so that it could be recycled whenever additional Ba^{140} was required.

Estimates of the quantity of Ba^{140} needed for single experiments range up to 10,000 C, which would mean initial activities of the irradiated uranium on the order of 10^6C. Decontamination factors (after removal of the Ba^{140}), therefore, should approach this value so that the enriched uranium could be re-cycled through standard processes to finished metal shapes with a minimum of shielding of the operations involved.

Although successful decontamination of uranium from fission products has been achieved through the use of solvent extraction processes, it was further proposed that precipitation reactions be investigated as to their ability to decontaminate uranium under these conditions. Certain process advantages are obvious for precipita-

tion-type reactions.

The uranium peroxide reaction (30% H_2O_2 as precipitant) and the uranyl oxalate reaction ($H_2C_2O_4 \cdot 2H_2O$ as precipitant) are both used extensively at this laboratory for regular chemical purification. This report describes their use for the separation of uranium from fission products.

II - Experimental Considerations

Limitations

1. Nature of activity at the time of attempted decontamination.

It was assumed at the outset of the problem that a cooling period would be allowed prior to attempted decontamination. This would decrease the initial activity by perhaps half and leave some medium-lived, but chiefly long-lived, isotopes. This cooling period could be of the order of 3 months.

2. Limit of decontamination.

Decontamination of irradiated uranium to the extent of the β and γ activity in an equal quantity of normal uranium would be sufficient.

3. Activity level of experiments.

Since the facilities available for this study afforded no protection from high level irradiation, the experiments performed

would have to be at such starting level as to permit direct handling of the material within certain time limits, and with the use of simple tongs.

Calculations.

This work was carried out on normal uranium solutions to which small quantities of fission product activity were added. Since normal uranium (especially old preparations) also contains β and γ activity from certain isotopes in the natural decay chain, this radiation was subtracted from the fission product activity. To obtain these blank values, "hot" experiments were duplicated by "cold" experiments -- actually preceded by them, thus also providing technique experience, especially in the use of tongs, remote vacuum transfers, etc.

Solution aliquots were counted before a precipitation was made. Then, after the cake was washed, converted to an oxide, re-dissolved, and made up to a definite volume, solution aliquots were again counted. This value became the starting count for the next precipitation, etc. In this manner, any value divided by its succeeding value provided the decontamination factor for that precipitation; or, the initial value divided by the final value of a series of precipitations would equal the total decontamination factor obtained for that series.

Sample Calculations.

The following calculations, shown in Table 1, were made for Experiment D-2, and are typical of the calculations made for all the experimental work, with the exception of some of the early experiments where no γ activity determinations were made.

TABLE 1

β Decontamination Results

Sample	Aliquot	Sample Result, (mean) c/m	Total Activity, c/m	Corrected Total Activity, c/m	Decontamination Factor
starting soln., "hot"	1/25000	4462	1.11×10^8	1.09×10^8	
starting soln., "cold"	1/25000	90	*2.25×10^6		
first ppt., "hot"	1/25000	96	2.40×10^6	1.12×10^6	97
first ppt., "cold"	1/25000	51	1.28×10^6		
second ppt., "hot"	1/5000	33	1.65×10^5	***	6.8
second ppt., "cold"	1/5000	119	5.95×10^5	1.65×10^5	
third ppt., "hot"	1/5000	59	2.95×10^5	1.10×10^5	1.5
third ppt., "cold"	1/5000	37	1.85×10^5		

Total DF = 991

γ Decontamination Results

Sample	Aliquot	Sample Result, (mean) c/m	Total Activity c/m	Corrected Total Activity, c/m	Decontamination Factor
starting soln., "hot"	1/2500	700	1.75×10^6	1.73×10^6	36
starting soln., "cold"	1/2500	8	**2.00×10^4		
first ppt., "hot"	1/2500	39	9.75×10^4	4.75×10^4	4.0
first ppt., "cold"	1/2500	20	5.00×10^4		
second ppt., "hot"	8/5000	19	1.19×10^4	***	4.8
second ppt., "cold"	8/5000	53	3.32×10^4	1.19×10^4	
third ppt., "hot"	8/5000	6	3.75×10^3	2.50×10^3	
third ppt., "cold"	8/5000	2	1.25×10^3		

Total DF = 692

*Average total β activity found for 50 gms. normal U was 2×10^6 c/m.

**Average total γ activity found for 50 gms. normal U was 2.5×10^4 c/m.

***Since the blank value is higher than the hot value, it must be neglected.

Corrections.

β and γ counting of the samples was done by the RaLa chemistry group (CMR-10). Counting results were corrected for background and coincidence before being reported to this group. No further correc-

tions were applied to the calculations other than the subtraction of blank values. Decay of the activity over the experimental period was small enough to be neglected. (See "Sources of Activity".) A small fraction of the decontamination is attributable to uranium yield itself -- e.g., if a decontamination factor of 4 were obtained after a precipitation whose uranium yield was 50%, then only half that decontamination would be due to chemical purification. This is a severe example, however, and on the basis of uranium yields and accompanying decontamination factors given later in this report, yield effects were negligible and no corrections were made for them.

Sample Preparation.

Aliquots were so arranged that one ml of solution contained enough activity for a reasonably accurate beta count. This volume of solution was evaporated in a glass cup 7/8" I.D. and 1/4" deep. Duplicate samples were prepared and the cups placed in the counting chamber. The mean, corrected result was used in the calculations as noted.

10 ml solution aliquots were given directly to Group CMR-10 for gamma counting. This group carried out the sample preparation for these aliquots using annular type counting bottles.

Sources of Activity.

Two sources of fission products were used to supply the β and γ activity for the experiments:

1. Uranyl nitrate solution from the Los Alamos water boiler.

This solution contained 450 mg. of uranium (14.9% U-235) per ml. Samples received after 3 months' decay emitted approximately 1.8 mC of γ activity per ml, measured by survey meter*, and at 7 months, 0.7 mC of γ activity per ml, measured in the same way. A sample taken for β count at the beginning of the experiments showed the following decay with respect to time:

Days	c/m, β
0	9305
4	9087
5	9245
6	9073
7	9048
10	8810

These data are plotted in Figure 1.

2. Uranyl nitrate solution from the dissolution of a Chalk River Pile slug.

Four 50 mC samples were received after about 3 months' decay, containing the following weights of U and Pu per sample:

total U = 2.65 gms.
U-235 = 0.016 gms.
Pu = 0.00225 gms.

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*Based on the common method of measuring γ-ray intensity: One curie = one r/hr. at one meter distance.

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Samples taken for β and γ count showed the following decay with respect to time:

Days	c/m, β	c/m, γ
0	3743	57,200
4	3723	-----
6	----	55,000
7	3633	-----
14	3517	-----

These data are plotted in Figure 2.

III - Experiments

A. Decontamination by the Use of the Uranium Peroxide Reaction at Various Acidities.

Two 500 ml, 0.42 M uranyl nitrate solutions were made from the dissolution of U_3O_8 in nitric acid. To one was added a mC of water boiler solution before the final volume adjustment was made. Aliquots were then taken from each solution for β counting.

A uranium peroxide precipitation was made on each solution by the simultaneous addition of a 30% excess of 30% H_2O_2 and 1:1 ammonium hydroxide at a constant pH of 2.5. After a 5 minute digestion period and a short settling period, the slurry was filtered through a fine, fritted glass Büchner funnel with a minimum of washing. The funnel was placed in a small crucible furnace and the UO_4 converted to UO_3

at 300°C for several hours. This oxide was dissolved in nitric acid, made up to 500 ml volume again, and aliquotted for re-counting for the β activity.

The experiment was repeated at pH 3.5 and 1.5 on "hot" solutions only, using the final solution from the pH 3.5 experiment as the starting solution of the pH 1.5 experiment.

For pH 1.0, the same scheme was used again, this time including the "cold" precipitation to provide a blank value for correction of the "hot" result.

Decontamination results are given in Table 2. Although uranium yields are not given, the reaction is essentially quantitative in this pH range.

TABLE 2

Decontamination Achieved by the Uranium Peroxide Reaction at Various Acidities

pH	Total β Activity in Starting Soln., c/m	Total β Activity After 1 Pptn., c/m	Decontamination Factor
3.5	6.28×10^7	5.16×10^7	1.2
2.5	4.51×10^7 (corrected)	2.12×10^7 (corrected)	2.1
1.5	5.16×10^7	1.11×10^7	4.7
1.0	13.2×10^7 (corrected)	1.30×10^7 (corrected)	10.1

Decontamination factors are plotted against pH in Figure 3.

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B. Preliminary Investigation of the Effect of the Uranyl Oxalate Precipitation on Decontamination.

1. The 500 ml solution remaining from the "hot" precipitation at pH 1.5 (see Exp. A), containing 1.11×10^7 c/m β, total, was made normal with respect to free HNO_3 and heated to steam bath temperature. The uranium was precipitated as uranyl oxalate by the addition of 150 ml of hot, aqueous oxalic acid solution (containing 29 gms. $H_2C_2O_4 \cdot 2H_2O$). The slurry was stirred for about 1 1/2 hours until it had reached room temperature. The precipitate was filtered out, transferred to a platinum crucible, and ignited overnight at 800°C. This oxide was dissolved in nitric acid, made up to 500 ml volume, and aliquotted for β counting. The result was a total count of 8.75×10^5. This is a decontamination factor of approximately 13.

By obtaining the weight of the crucible before the U_3O_8 was dissolved, the uranium yield was found to be 90% for this precipitation.

2. The 500 ml solutions remaining from both the "hot" and "cold" precipitations at pH 1.0 (see Exp. A) were precipitated as uranyl oxalate as above. The corrected starting count was 1.30×10^7 c/m β; the corrected final count was 7.0×10^5 c/m β, giving a decontamination factor of 19.

Again the uranium yield for the precipitations was about 90%.

C. The Effect of Hold-Back Carriers on Decontamination.

Two aspects of this technique were investigated. In the first experiment Al, Mg, and Fe were added to determine whether they would

adsorb the active isotopes to any appreciable extent, thus holding them in solution when the uranium precipitated. These particular metals were chosen because data were available on the purification of uranium from them by means of the peroxide precipitation.

A uranyl nitrate solution containing 50 gms. of uranium was prepared. 0.5 gm. each of Al^{+++}, Mg^{++}, and Fe^{+++} (as nitrates) was added to it. The solution was made active by the addition of 5 mC of water boiler solution, made up to 500 ml volume, and aliquotted for β counting. The uranium was precipitated as uranium peroxide at pH 2.5 as described in Exp. A (except that a small amount of citric and malonic acids was added to complex the Fe^{+++}). The UO_4 was filtered out, ignited to UO_3, dissolved in acid, and aliquotted for a second β count. A second precipitation was then made to bring the Al, Mg, and Fe content to <10 ppm. The final precipitate was converted to oxide, dissolved, and aliquotted for the final β count.

The procedure was duplicated without the addition of tracer to provide blank values. The corrected β results are given in Table 3.

TABLE 3

Sample	Total β Activity (Corrected), c/m	Decontamination Factor
starting solution	1.74×10^8	---
dissolution of first ppt.	1.09×10^8	1.6
dissolution of second ppt.	8.00×10^7	1.4

In the second experiment, 0.5 gm. each of Sr, Ce, and Cs were added as nitrates, and Rb as the chloride, to the solutions remaining from the first hold-back carrier experiment in an effort to replace the active ions carried down by the uranium with their corresponding inactive ions. Although a radio-chemical assay was not made to determine the specific isotopes supplying the major activity in water boiler solution, the literature indicated Sr, Cs, Rb, and several rare earth isotopes (of which it was hoped Ce would be representative) to be among the principal offenders.

Two successive peroxide precipitations were made at pH 2.5 on both the "hot" and "cold" solutions as in the case of the Al-Mg-Fe experiment. The corrected β results are given in Table 4.

TABLE 4

Sample	Total β Activity (Corrected), c/m	Decontamination Factor
starting solution	8.00×10^7	---
dissolution of first ppt.	2.30×10^7	3.5
dissolution of second ppt.	1.50×10^7	1.5

D. A Systematic Study of the Decontamination Factors Obtained from Successive Peroxide and Oxalate Precipitations at Various Activity Levels.

1. Three successive uranium peroxide precipitations were made at pH 1.0 (as described in Exp. A) on a 500 ml starting solution con-

taining 50 gms. of uranium and 0.5 mC of water boiler activity. β and γ activities were counted initially and after each precipitation. The procedure was duplicated on a similar "cold" solution to provide blank values.

New starting solutions were provided for identical studies at the 5.0 and 50 mC levels. The corrected results and decontamination factors appear in Table 5.

2. Three successive uranyl oxalate precipitations were made on a 500 ml starting solution, N with respect to free HNO_3 (as described in Exp. B), containing 50 gms. of uranium and 0.5 mC of water boiler activity. This study was also repeated at the 5.0 and 50 mC levels. The corrected results and decontamination factors are given in Table 5.

E. Decontamination Results Obtained with Successive Uranyl Oxalate Precipitations under Normal and High Uranium Yield Conditions, Using Pile "Dissolver" Solution as the Activity Source.

These experiments were done for two reasons: (1) to determine whether the physical characteristics and the activity of water boiler solution were representative of dissolver solution from a pile insofar as decontamination was concerned, and (2) to try to improve the uranium yield with a minimum loss in decontamination efficiency.

1. A new starting solution was made up which contained 50 gms. of uranium, 50 mC of dissolver solution, and was normal with respect to free nitric acid. It was diluted to 500 ml and aliquotted for β and γ counting.

Table 5

	SAMPLE	Decontamination Factors Received From Successive Uranium Peroxide Precipitations						Decontamination Factors Received From Successive Uranyl Oxalate Precipitations					
		0.5 mC Level		5.0 mC Level		50 mC Level		0.5 mC Level		5.0 mC Level		50 mC Level	
		Total c/m, Corrected	DF	Total c/m, Corrected	DF	Total c/m, Corrected	DF	Total c/m, Corrected	DF	Total c/m, Corrected	DF	Total c/m, Corrected	DF
β Results	Starting Solution	1.33×10^{8}	—	1.33×10^{9}	—	1.14×10^{10}	—	1.09×10^{8}	—	1.00×10^{9}	—	8.74×10^{9}	—
	Dissolution of 1st. Ppt.	1.49×10^{7}	8.9	1.29×10^{8}	10.3	1.23×10^{9}	9.3	1.12×10^{6}	97	4.48×10^{6}	223	3.29×10^{7}	266
	Dissolution of 2nd. Ppt.	1.57×10^{7}	0.9	9.95×10^{7}	1.3	7.45×10^{8}	1.7	1.65×10^{5}	6.8	2.20×10^{5}	20	2.35×10^{5}	140
	Dissolution of 3rd. Ppt.	8.83×10^{6}	1.8	9.10×10^{7}	1.1	6.65×10^{8}	1.1	1.10×10^{5}	1.5	2.10×10^{5}	1	1.75×10^{5}	1.3
	Total DF	—	15.1	—	14.6	—	17.1	—	991	—	4.76×10^{3}	—	5.0×10^{4}
γ Results	Starting Solution	6.18×10^{6}	—	2.61×10^{7}	—	2.14×10^{8}	—	1.73×10^{6}	—	1.53×10^{7}	—	9.85×10^{7}	—
	Dissolution of 1st. Ppt.	3.42×10^{6}	1.8	1.92×10^{7}	1.4	1.49×10^{8}	1.4	4.75×10^{4}	36	9.12×10^{4}	168	4.69×10^{5}	210
	Dissolution of 2nd. Ppt.	2.07×10^{6}	1.1	2.04×10^{7}	0.9	1.76×10^{8}	0.8	1.19×10^{4}	4.0	1.81×10^{4}	5.0	6.44×10^{4}	7.3
	Dissolution of 3rd. Ppt.	3.51×10^{6}	0.9	2.17×10^{7}	0.9	1.84×10^{8}	1.0	2.50×10^{3}	4.8	4.38×10^{3}	4.1	6.88×10^{3}	9.4
	Total DF	—	1.8	—	1.2	—	1.2	—	692	—	3.50×10^{3}	—	1.4×10^{4}

The uranium was precipitated as described in Exp. B, calcined to U_3O_8, dissolved in HNO_3, made up to 500 ml, and aliquotted for re-counting of the β and γ activity. The cycle was repeated two more times and the entire procedure duplicated on a "cold" solution to provide the results shown in Table 6. Uranium yield results were obtained by weight of the "hot" U_3O_8; these yields include transfer losses.

2. For the second experiment*, 200 ml of 2 M uranyl nitrate solution, 2 N with respect to free HNO_3, and containing 50 mC of dissolver solution, was aliquotted for β and γ counting. This solution was heated to steam bath temperature and the uranium precipitated by a 10% excess of hot, 50% oxalic acid solution. The precipitate was digested hot for 1/2 hour with constant stirring. The slurry was then chilled in an ice bath to 5-10°C and 400 ml of cold, saturated oxalic acid was added. The chilled slurry was stirred for 10 minutes and then filtered. The cake was transferred to a crucible for conversion to U_3O_8, which was then dissolved and re-counted. The cycle was repeated twice more, duplicated on a "cold" solution, and uranium yield data taken on the "hot" U_3O_8, as in the previous experiment. The results are given in Table 7.

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*The uranyl oxalate precipitation conditions necessary to achieve maximum uranium yield were based on unpublished work by J. P. Bertino.

TABLE 6

Sample	Total β Activity, Corrected, c/m	Decontamination Factor	Total γ Activity Corrected, c/m	Decontamination Factor	Uranium Yield per Precipitation, %
Starting soln.	8.69×10^9	---	1.14×10^8	---	---
First ppt.	3.51×10^7	250	2.82×10^6	40	81
Second ppt.	1.76×10^6	20	2.57×10^5	11	86
Third ppt.	5.60×10^5	3.1	6.99×10^4	3.7	83
Total for 3 cycles	---	1.6×10^4	---	1.6×10^3	58

TABLE 7

Sample	Total β Activity, Corrected, c/m	Decontamination Factor	Total γ Activity, Corrected, c/m	Decontamination Factor	Uranium Yield per Precipitation, %
Starting soln.	1.04×10^{10}	---	1.59×10^8	---	---
First ppt.	1.41×10^8	74	5.11×10^6	31	95
Second ppt.	5.23×10^6	27	3.98×10^5	13	96
Third ppt.	1.46×10^6	3.6	1.88×10^5	2.1	95
Total for 3 cycles	---	7.1×10^3	---	850	87

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F. Investigation of the Presence of Radio-Colloids in Water Boiler Solution.

During a discussion of the problem with Dr. G. K. Rollefson*, he suggested that some isotopes may exist in water boiler solution as negative radio-colloids which would continually be carried down with the uranium precipitates. He further suggested that, if present, they might be removed by the addition of positive colloids such as TiO_2, ZrO_2, or CeO_2 in a filtration step. Accordingly, the following experiments were tried:

1. Four 200 ml solutions of dilute HNO_3 containing 0.5 mC each of water boiler solution were aliquotted for β and γ activity. The pH of the solutions was then adjusted to 3.0, 2.0, 1.0, and 0, respectively, by adding dilute NH_4OH. After being allowed to stand at room temperature for at least 22 hours, each solution was slurried with 20 gms. of finely divided TiO_2. The supernatant was filtered off and the procedure repeated twice with 100 ml portions of acidified wash water at the same pH. The filtrate and washings were combined, aliquotted, and re-counted for β and γ activity. The results may be found in Table 8.

2. A single 200 ml dilute HNO_3 solution containing 0.5 mC of water boiler solution was made up and aliquotted for β and γ counting.

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*Consultant to the Chemistry-Metallurgy Division.

[redacted]

About 1/4 gm. of sodium silicate was added to the solution, and the pH was adjusted to 3.5. At the end of 24 hours, a small amount of silica had precipitated. The solution was then filtered through a 3/8" damp cake of TiO_2 on a 150 ml fine, sintered glass funnel, followed by two 100 ml portions of wash water at pH 3.0. The filtrate and washings were recounted for β and γ activity. These results are also given in Table 8.

TABLE 8

β Results

Exp.	pH	Standing Time, hrs.	Starting Soln. Activity, c/m	Filtrate Activity, c/m	Decontamination Factor
F-1-a	3.0	22	8.52×10^7	7.85×10^7	1.09
F-1-b	2.0	23	8.26×10^7	8.15×10^7	1.01
F-1-c	1.0	41	8.38×10^7	7.86×10^7	1.07
F-1-d	0	44	8.65×10^7	8.18×10^7	1.06
F-2	3.5	24	8.60×10^7	8.23×10^7	1.04
			γ Results		
F-1-a	(as above)		1.03×10^6	7.63×10^5	1.35
F-1-b			9.52×10^5	4.10×10^5	2.32
F-1-c			9.44×10^5	3.40×10^5	2.77
F-1-d			9.22×10^5	3.45×10^5	2.67
F-2			6.62×10^5	5.80×10^5	1.14

[redacted]

[redacted]

IV - Discussion and Conclusions

Experiment A showed that the decontamination of uranium with respect to the β activity in fission products is dependent upon the pH at which the peroxide reaction is carried out. This is clearly shown by the curve in Figure 3. However, it is known from a study of the uranium peroxide precipitation reaction (Report LA-1089) that pH 1.0 is the highest acidity at which the precipitate can be conveniently handled. It may be assumed, then, that a β decontamination factor of 10 (per cycle) is maximum for the reaction. This value is not unprofitably small if it could be repeated during subsequent cycles. This is not the case, as Exp. D showed. Here, a constant factor was not obtained during three successive precipitations at pH 1.0 at any activity level. Furthermore, the reaction provided for virtually no decontamination from γ activity.

The decontamination efficiency of the peroxide precipitation was not improved by the use of hold-back carriers. The factors obtained in Exp. C were negligible. At this point it was concluded that the uranium peroxide precipitation reaction had no decontamination value, and further experimental work was abandoned.

With respect to the uranyl oxalate precipitation reaction, the results of Exp. B were so encouraging that a systematic study of decontamination from various activity levels was immediately undertaken (Exp. D). By referring to Table 5, it is seen that decontamination was

[redacted]

obtained to below the level of an equal quantity of normal uranium. (Also see the Sample Calculations for the 0.5 mC level, page 7.) Total γ decontamination was not as successful as total β reduction, but was of the same order of magnitude.

In Exp. E it was found that the oxalate reaction was several times less effective on solutions containing activity from pile dissolver solution. The reason for this is not known, although two suggestions for the difference in results have been offered: (1) that pile dissolver solution, being less pure than Los Alamos water boiler solution, contained some colloidal silica which carried activity along with the precipitated uranium, and (2) that the concentrations of the various active isotopes differed slightly between the two activity sources, so that, due to the individual decontamination characteristics afforded each species by the reaction, the total factors were also different. The decrease in total factors for Exp. E-2 as compared to Exp. E-1 is more easily accounted for. In the case of high uranium yield, the percentage separation of filtrate from cake is considerably less per cycle than for the so-called "normal" uranium yield for the uranyl oxalate reaction. If suitable calculations were made to correct for this difference, the factors from Exp. E-2 and Exp. E-1 would probably compare very favorably. Such calculations are not presented here because the necessary data were not taken.

Radio-colloids do not appear to exist to any appreciable extent in water boiler solution. The results of Exp. F showed essentially

no decrease in total β activity after attempted removal of such colloids, although total γ activity was reduced by a factor of 2-3. This reduction is not worth the effort as compared to 10^4 obtained directly by precipitation. Pile dissolver solution was not tested for the presence of colloids.

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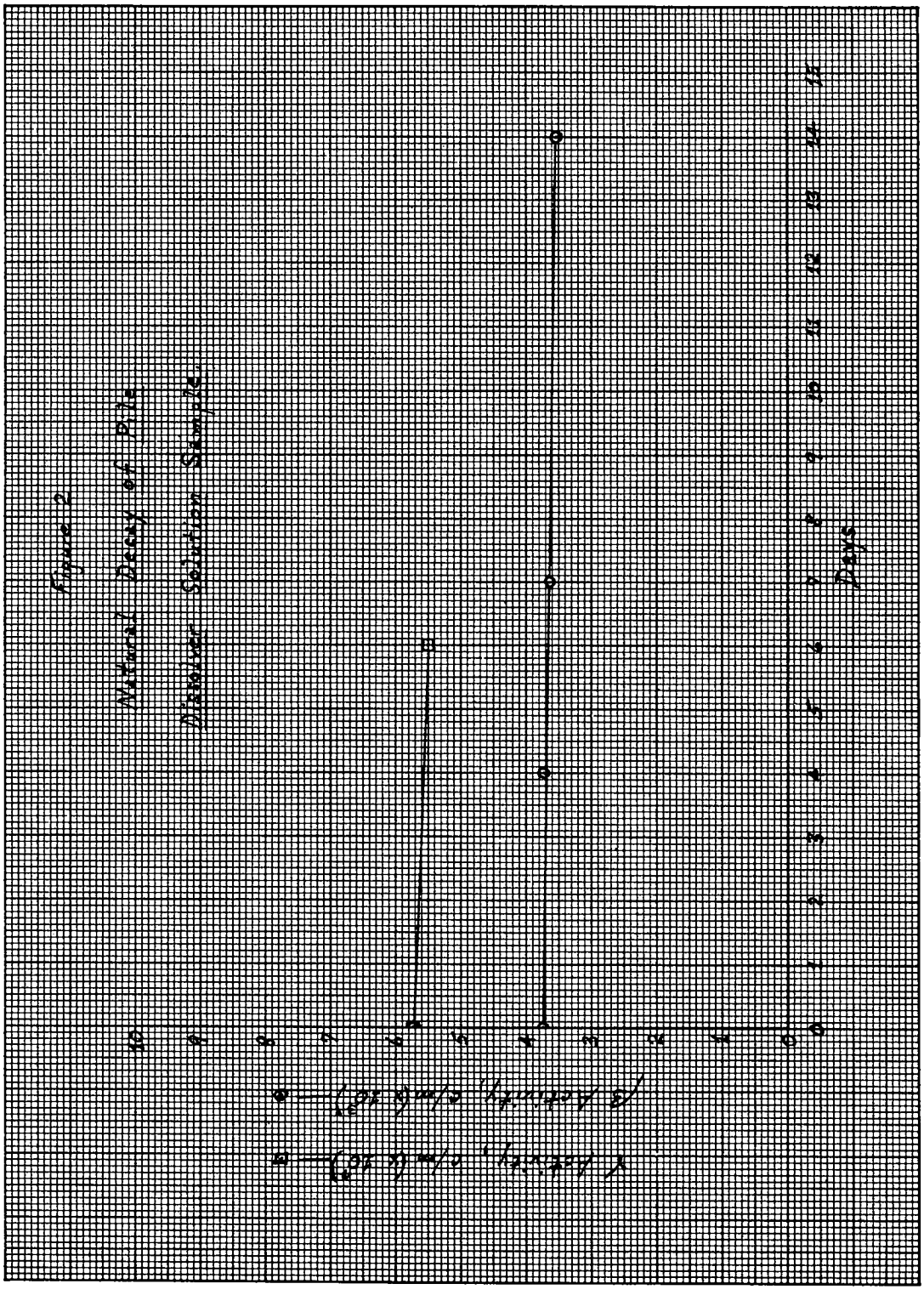

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