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CHEMICAL PROCESS DEVELOPMENT SECTION

LABORATORY PURIFICATION AND CONCENTRATION OF U^{233}

D.C. OVERHOLT

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TECHNICAL DIVISION

Chemical Process Development Section

LABORATORY PURIFICATION AND CONCENTRATION OF U^{233}

D. C. Overholt

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1.0 Abstract

This report presents a 23 final concentration and purification process (diisopropyl ether solvent extraction) to follow the plant-scale recovery of 23 from bombarded thorium.

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2.0 Introduction

Uranium 233 is produced by the following nuclear reaction:

$$_{90}Th^{232} \xrightarrow{(n,\gamma)} {}_{90}Th^{233} \xrightarrow[23.5\ min.]{\beta} {}_{91}Pa^{233} \xrightarrow[27.4\ d]{\beta} {}_{92}U^{233}$$

The proposed process[4] for the separation of 23 from thorium and fission products consists of the dissolution of bombarded thorium slugs in nitric acid, solvent extraction of the 23 in a continuous counter-current column employing one of three alternate solvents (diisopropyl ether, dibutyl cellosolve, or hexone), and stripping of the 23 from the solvent with water.

The principal functions of the concentration and purification step are to concentrate the product and to eliminate the corrosion products introduced in the initial separation of 23 from Thorium. In addition, the radioactivities carried over from this initial separation are also removed.

The scale of this laboratory concentration and purification procedure is limited by the critical mass of 23, and an appreciable safety factor has been allowed in the design of laboratory equipment to process 150 gram quantities per batch[6]. It is estimated that equipment of this capacity is sufficient to handle the 23 produced from one or more breeder piles at the neutron flux as presently envisioned[1].

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3.0 Summary

A laboratory final concentration and purification process for U^{233} has been developed to follow the recovery and decontamination of this material from bombarded thorium. This procedure employes a batch-wise solvent extraction cycle with diisopropyl ether, evaporation of the product to dryness, and ignition to the oxide.

The extraction loss of 23 in this process is about 0.03%. Spectrographic analysis of a product solution indicated less than 0.2 mg Al/mg of 23 and only traces (100 to 1000 ppm) of Th, Ca, Mg, and Si. Isotopic analysis using an energy range analyser showed 0 to 3% contaminating alpha activity. The beta activity in the product appears to be no greater than the (G.M. countable) activity normally associated with pure U^{233}.

Alternate concentration methods involving precipitation with hydrogen peroxide, and tetramethyl ammonium hydroxide gave adequate separation from ionic contaminants, but little or no decontamination from fission products.

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4.0 Experimental and Results

4.1 Diisopropyl Ether Extraction of 23

A total of 13.5 grams of 23 was concentrated and isolated in eighteen runs with an average loss of 0.03% employing a batch diisopropyl ether solvent extraction process (see Table 4.1-1). First cycle product solutions containing 0.1 to 90 mg/ml of Al and one to two mg/ml of Th as contaminants were evaporated to dryness to remove excess HNO_3. The feeds were then diluted to about 2.5M Al $(NO_3)_3$ and the acidity adjusted to a pH of 0.2 to 0.0. Eight to ten successive equal volume batch extractions were made with water washed diisopropyl ether at 20°C. This process was equally successful on decontaminating product solutions obtained from first cycle batch diisopropyl ether extraction, and first cycle continuous counter-current column extraction using either dibutyl cellosolve, diisopropyl ether, or hexone as solvents.

In the original first cycle batch process some scrub solution $(Al(NO_3)_3$ was mechanically carried over into the 23 product strip solution and the subsequent volume reduction possible was limited by the quantity of aluminum nitrate present (See Final Report Semi-Works Problem #TX5-11-63). However, in the case of column operations there was no mechanical carry-over of scrub solution and the volume reduction that could be obtained by evaporation of the first cycle product solution was limited only by the solubility and criticality of 23.

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Diisopropyl Ether Extraction of 23 (con't)

To obtain data on the maximum concentration of uranium that could be extracted per equal volume pass of diisopropyl ether at 20°C, a series of extractions was made with various amounts of uranium (30 to 86 grams per liter) in $Al(NO_3)_3$ aqueous feeds. At all concentrations investigated the diisopropyl ether retained the uranium long enough to make the necessary phase separation (5 to 10 minutes). At concentrations of 27-32 grams U/l in the diisopropyl ether no precipitation occurred within the first hour of standing. However, at concentrations of 73 grams per liter and above, precipitation started in about 10 minutes after phase separation and continued until the uranium concentration in the mother liquor was about 53 grams of uranium per liter.

In view of these uranium solubilities it was apparent that the final concentration and isolation of 23 produced in the proposed high flux pile was feasible on a laboratory scale, since it is planned to process 50 kilograms of bombarded thorium containing 75 grams of 23, in each charge of the first cycle separation[4].

Since good phase separation is essential for a high purity product the major problem involved in the 23 concentration process was the occasional formation of emulsions even though the feeds were filtered prior to extraction. It was found that if these emulsified phases were drawn through a coarse sintered glass disc after agitation excellent phase separation was obtained (settling time about 5 minutes as compared to 30-45 minutes without this treatment). The effectiveness of this step was further demonstrated by

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Diisopropyl Ether Extraction of 23 (con't)

filtering an emulsified feed which had been stabilized by the presence of 0.1 M $K_2Cr_2O_7$ and which had failed to coalesce after standing 72 hours. The phases separated quickly when the emulsion was drawn through the sintered glass disc. In view of these results, a coarse sintered glass filter was sealed into the bottom of the extraction vessel for routine use. Occasionally it was observed that a fine precipitate, or crud, formed on the filter plate which partially clogged the disc and resulted in a slow filtering rate. The crud formation was accelerated as successive passes of ether removed the small amount of free nitric acid present in the feed. This crud formation on the filter plate was eliminated by using diisopropyl ether pre-washed with 1.0M HNO_3 instead of distilled water. There was no evidence of solvent decomposition due to the acid wash. Subsequently, it was found that the 1.0M HNO_3 wash could be eliminated if the product feed solutions were adjusted to a pH of 0.6, filtered through a fine sintered glass filter, and then readjusted to a pH of 0.2-0.0 with 70% nitric acid prior to the first solvent pass. This procedure prevented the formation of crud on the filter disc and gave excellent filtering rates.

Some of the first cycle 23 strip product solutions, particularly those obtained from the dibutyl cellosolve continuous extraction column, contained large amounts of oily organic decomposition products which were only slightly soluble in diisopropyl ether. Since these oils contained appreciable quantities of 23 (about 10%), it was necessary to evaporate them to dryness and to treat

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Diisopropyl Ether Extraction of 23 (con't)

the residue with a three fold excess of 30% H_2O_2 to destroy the solvent decomposition products. A three-fold excess of H_2O_2 was necessary since all of the 23 $UO_2(NO_3)_2$ had to be converted to $UO_4 \cdot 2H_2O$. The $UO_4 \cdot 2H_2O$ was then reconverted to the nitrate with a two-fold excess of 70% HNO_3. The equations for the reactions are:

$$UO_2(NO_3)_2 \cdot 6H_2O + H_2O_2 \text{——} UO_4 \cdot 2H_2O + 2HNO_3 + 4H_2O$$

$$UO_4 \cdot 2H_2O + 2HNO_3 + 3H_2O \text{——} UO_2(NO_3)_2 \cdot 6H_2O + 1/2\ O_2$$

While this step has been incorporated in the flowsheet, it may not be necessary for 23 strip product solutions obtained from routine first cycle continuous column runs, since very small amounts of organic decomposition products are expected when the columns are operated at optimum conditions.

4.2 Stripping 23 from Diisopropyl Ether

Stripping of 23 from the ether extract was accomplished with four successive quarter volume passes of 2% HNO_3. No emulsion difficulty was encountered although small amounts of organic decomposition products from the ether were transferred to the aqueous strip. These decomposition products were destroyed in a subsequent volume reduction step with 30% H_2O_2. In all runs, the alpha count of the diisopropyl ether after stripping was at or below background.

4.3 Alternate Precipitation Methods for Concentration and Isolation of 23

Although a second solvent extraction cycle will accomplish the required thorium, aluminum, and fission product decontamination, there are certain advantages in substituting precipitation methods for this purpose.

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Investigation of some of the possible precipitating agents were made. Scouting experiments indicated that precipitation of thorium and uranium peroxides with H_2O_2 could be used to separate Al from 23 since Al does not form a peroxide. However, since Fe^{++} and Fe^{+++} catalyze the decomposition of H_2O_2, an iron removal step was necessary. Uranium losses for such a step were of the order of 0.2 to 0.4%. Using a synthetic feed of 0.2M $Al(NO_3)_3$, 0.02M $UO_2(NO_3)_2$ and 0.001M $Th(NO_3)_4$, it was found that the optimum HNO_3 concentration for this precipitation was in the pH range of 1.0 to 1.5. At a pH less than 1.0, the 23 loss was high (15-20%) while at a pH greater than 1.5 some of the Al precipitated.

An ammonium diuranate precipitation was not effective in separating Al from 23 and Th because of co-precipitation. This separation was possible if NaOH was used as the precipitant. However, since sodium is not desired in the final metallurgical process and since it is not easily removed from the sodium diurante precipitate, the use of this reagent is not recommended.

Tetramethylammonium hydroxide as a base is approximately as strong as NaOH. Precipitation of the diuranate with this compound with subsequent ignition of tetramethylammonium diuranate should yield pure 23 oxide. Experiments indicated that the 23 losses in such a precipitation were high (16-24%), but it was found that the reagent tetramethylammonium hydroxide contained appreciable quantities of carbonate which are known to complex uranium and therefore high waste losses might be expected. In order to use this reagent it would be necessary to prepare the carbonate free compound, and additional studies would be necessary to evaluate properly the use of this reagent.

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All of the precipitation methods described in this section gave little or no decontamination from radioactive elements and, consequently, are useful only as a means of separating 23 from inactive contaminants. In view of these data, it is concluded that the solvent extraction process utilizing diisopropyl ether afforded the best decontamination from active and inactive metals of any of the methods investigated.

4.4 Ignition to 23 Oxide

This phase of the process may be accomplished by the precipitation of $(NH_4)_2U_2O_7$ from $UO_2(NO_3)_2$, drying the precipitate, and firing at 1000°C to U_3O_8; or by evaporating $UO_2(NO_3)_2$ solution to dryness and decomposing to UO_3 at 250°C and then firing at 1000°C to the U_3O_8. A series of tests was conducted using one gram quantities of U.S. Bureau of Standards U_3O_8 which were dissolved in small volumes of HNO_3, evaporated to dryness, and converted to $(NH_4)_2U_2O_7$. The diuranate precipitates were quite voluminous and, to avoid spattering, it was necessary to dry the precipitates very slowly at about 100°C. The ignition was then completed by firing at 1000°C to U_3O_8. The measured yield through this cycle was 99.9%.

Direct ignition was accomplished on a one-gram scale by dissolving the standard U_3O_8 in HNO_3, evaporating to dryness, heating at 250°C until all the $UO_2(NO_3)_2$ was converted to UO_3, and finally firing to the U_3O_8 at 1000°C. The measured yield by this method was 99.99% and the latter procedure is recommended.

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4.5 Purity of Concentrated 23

Spectrographic analysis of a one gram sample of 23 product solution has indicated < 0.2 mg Al/mg of 23 (which was the lower limit of the spectrographic method) and only traces of Th, Ca, Mg, and Si. The source of the Ca and Mg is probably the distilled H_2O and the Si is from the thorium metal slugs. In all cases, the Al and Th content were below the limits of the spectrographic methods employed.

The product strip solutions from the second solvent extraction cycle, on evaporation to dryness, contained a small amount of organic residue which was eliminated by treating the concentrated 23 product twice with small portions (5-10ml) of 70% HNO_3 and, finally, with a 1-1 mixture of 70% HNO_3 and 30% H_2O_2. As further evidence of the absence of Al and Th salts, one gram of 23 was completely soluble in about 2ml of H_2O (the oxides of these metals would have been insoluble).

Further investigation of the 23 product purity was made using an energy range analyzer ("pulse analyzer"). Results of this analysis indicated that not more than 1% of the product had an alpha particle energy greater than 4.82 Mev and about 2% had an energy between 4.82 and 4.76 Mev. The value of the most recent measurements recorded in the literature for the energy of the U^{233} alpha particle is 4.82 Mev. Accordingly, the isotopic purity of the isolated 23 by this method of analysis is $\geq 97\%$ with a possible contaminant being U^{234} which has a 4.76 Mev alpha particle.

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Beta and gamma activities of the final product were well below tolerance. About 11,000 beta counts per minute per mg were associated with the 23. Twelve gamma counts per minute were detected in a one gram quantity of 23 which had been processed by this concentration procedure. The beta and gamma decontamination factors obtained for this concentration cycle were about 10^2 and 10^3, respectively. A six gram quantity of 23 extracted by the Chemistry Division continuous counter-current column using dibutyl cellosolve as the solvent, and subsequently concentrated to 25 ml by the procedure discussed in this report, measured 0.1 mr/hr of gamma radiation at contact.

The observed beta count of the product with a G-M tube when corrected to 100% geometry, but without correcting for absorption, was about 1 count/minute per 600 alpha disintegrations/minute. The actual count was made on the top shelf (30% geometry) through about 8 mg/cm^2 of cellophane, air and mica window. This count semms to be in good agreement with values obtained at Argonne National Laboratories with varying amounts of absorber, using radioactively pure U^{233} (see CC-2636 and CC-3056). Hence, it is concluded that the activity in this material is certainly not greater than the (G-M countable) activity associated with pure U^{233} (i.e. due to conversion electrons and photoradiation.)

4.6 Description of Equipment

The apparatus used for the concentration of 23 (see Figure 4.6-1) consisted of a jacketed extraction vessel with a total capacity of about 1500 ml. around which water from a constant temperature bath was circulated. This vessel was

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also equipped with a 10 x 1 inch interface stirrer and a coarse sintered glass filter which was sealed into the bottom. After agitation both organic and aqueous phases were drawn through the filter disc into a 1500 ml settling vessel. As the phases separated the aqueous was drawn off to a feed (1000 ml) hold-up vessel and the organic phase transferred to a stripping vessel. The aqueous phase was then returned to the extraction vessel and the cycle repeated. The stripping vessel consisted of a four liter conical shaped vessel in which the two phases were agitated by a 10 x 1 inch interface stirrer, from which the strip product was drawn off for final volume reduction and the organic waste stored for steam distillation and reuse. More recent equipment has been designed and constructed of stainless steel to process 150 gram quantities of 23 to operate as described above (See Figure 4.6-2).

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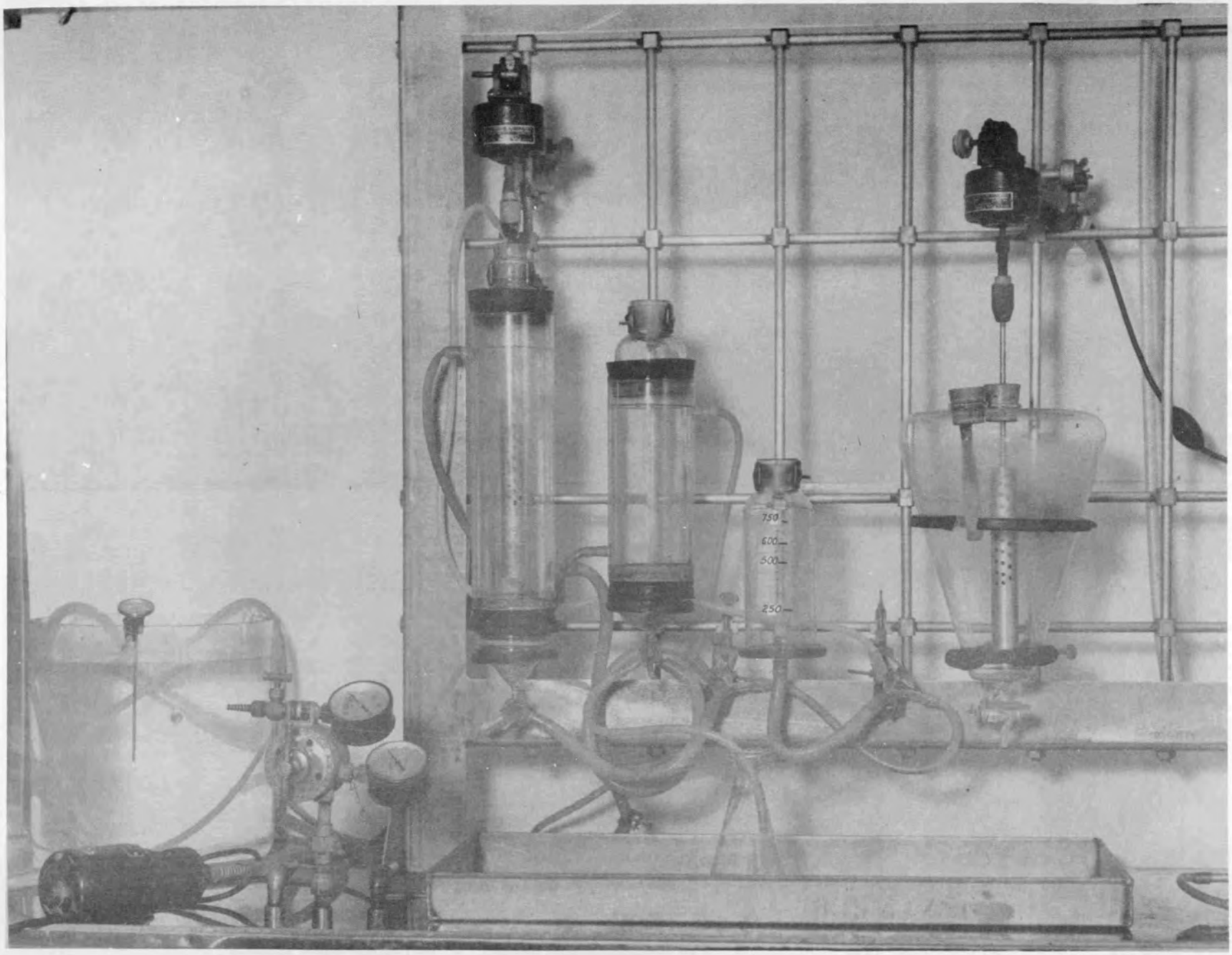

FIGURE 4.6-1

PHOTO 5049

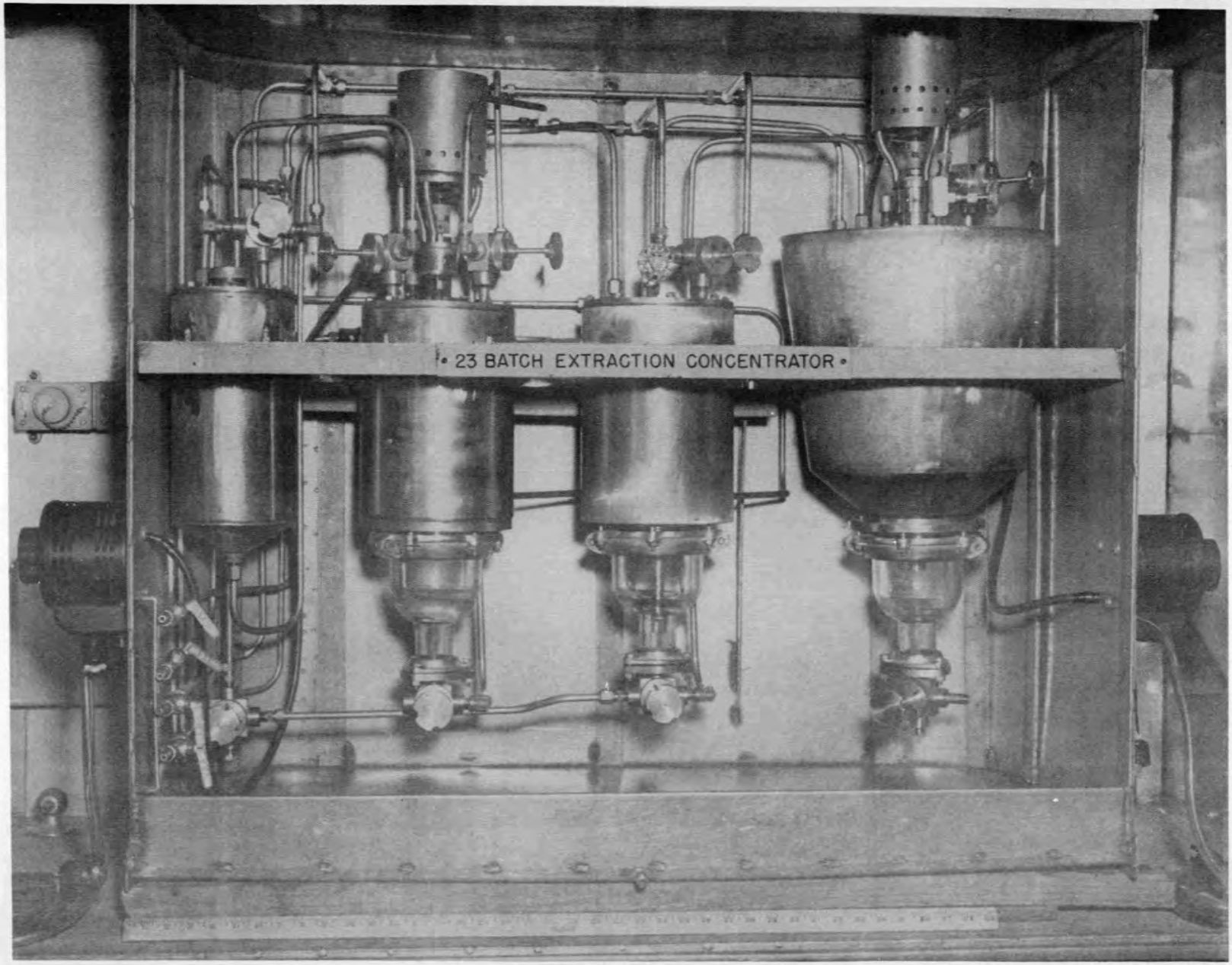

FIGURE 4.6.2

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TABLE 4.1-1

23 Laboratory Concentration Runs
(Batch Extraction)

Aqueous Feed: ca. 2.5M $Al(NO_3)_3$, 1.6M NaF

Organic Solvent: Diisopropyl ether, successive equal volume passes at 20°C, 500 ml Scale.

Concentration Run #	Number of Successive Solvent Passes	Total 23 in Starting Feed mg.	Total 23 Recovered after ext'n, stripping, & volume reduction mg.	Uranium Losses		Solvent & process used in first cycle **
				Extraction & stripping mg 23	Total %23	
1	6	2.72	2.68	0.045	1.66	A-1
2	7	11.40	11.2	0.022	0.28	A-1
3	8	1078.0	1010.0	0.428	0.04	A-1
4	9	515.0	504.0	0.143	0.03	A-1
5	10	564.0	550.0	0.41	0.08	A-1
6	7	315.0	338.0	0.11	0.02	A-1
7	7	203.0	203.0	0.05	0.02	A-1
8	8	550.0	558.0	0.015	0.003	B-2
9	9	776.0	850.0	0.013	0.002	B-2
10	9	185.5	200.0	0.20	0.10	A-2
11	9	6270.0	6078.0	0.218	0.036	B-2
12	9	64.0	68.1	0.03	0.044	A-2*
13	8	153.0	178.0	0.026	0.014	A-2*
14	7	289.0	292.0	0.028	0.009	A-2*
15	8	121.0	116.0	0.04	0.035	A-2*
16	9	800.0	815.0	0.09	0.012	A-2*
17	9	69.6	77.0	0.015	0.02	A-1
18	10	1730.0	1735.0	0.012	0.0007	B-2
TOTAL			13,585.98	1.895		

* Reprocessed 23 from A-1 runs
** A - diisopropyl ether

B - dibutyl cellosolve
1 - batch extraction - semi-works scale
2 - continuous counter-current extraction

[redacted]

[redacted]

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DWG 7620

CONDITIONS & REACTIONS

LABORATORY

1. FEED PREPARATION

FEED TANK
Jacketed, heating and cooling.
Hood installation.

$UO_2(NO_3)_2 \cdot 6H_2O + H_2O_2 \longrightarrow UO_4 \cdot 2H_2O + 2HNO_3 + 4H_2O$

$UO_4 \cdot 2H_2O + 2HNO_3 + 3H_2O \longrightarrow UO_2(NO_3)_2 \cdot 6H_2O + 1/2\ O_2$

2. EXTRACTION

EXTRACTOR
Jacketed, heating and cooling.
Agitated.
Hood installation.
Vacuum and air transfer.

TEMPERATURE: 20°C ± 2°C extraction.

3. STRIPPING

STRIPPER
Jacketed, heating and cooling
Agitated.
Hood installation
Gravity transfer.

TEMPERATURE: 20°C ± 2°C.

4. EVAPORATION-VOLUME REDUCTION

EVAPORATOR
Jacketed for heating.
Condenser.
Hood installation.

5. IGNITION

CALCINER
Electric resistance heating elements.
Off gas flue.

$UO_2(NO_3)_2 \xrightarrow[250°C]{heat} UO_3 + 2NO_2 + 1/2\ O_2$

$3\ UO_3 \xrightarrow[1000°C]{heat} U_3O_8 + 1/2\ O_2$

PROCEDURE

1. 23 FEED PREPARATION

A. Evaporate concentrated 23 strip solution to dryness, 150°C ± 5°C.
B. Add 3 fold excess 30% H_2O_2 to precipitate $UO_4 \cdot 2H_2O$.
C. Evaporate to dryness, 150°C ± 5°C.
D. Add 2 fold excess of 70% HNO_3.
E. Evaporate to dryness, 150°C ± 5°C.
F. Add 2 L 2.5M $Al(NO_3)_3$, 1.5M NaF previously filtered through a "fine" filter at pH 0.6.
G. Adjust feed to pH 0.1-0.0 with HNO_3.
H. Transfer aqueous feed to extractor.

2. 23 LABORATORY EXTRACTION

A. Add 2 L steam distilled diisopropyl ether.
B. Agitate 10 minutes.
C. Draw both phases through "F" stainless steel filter to settling vessel.
D. Settle 5 minutes.
E. Separate phases-transfer aqueous to extractor-organic to stripper.
F. Repeat A through E until 9 passes are made.
G. Transfer aqueous phase to waste.

3. 23 LABORATORY STRIPPING

A. Add 4.5 L 0.03M HNO_3 (0.2% HNO_3).
B. Agitate 10 minutes.
C. Settle 10 minutes.
D. Transfer aqueous phase to evaporator.
E. Repeat A through D until 4 passes are made.
F. Transfer organic phase to solvent recovery.

4. EVAPORATION-VOLUME REDUCTION

A. After addition of each strip, evaporate to 0.5 L.
B. Transfer to calciner tray.
C. Wash boiler three times with 0.2 L 3M HNO_3.
D. Transfer each wash to calciner tray.

5. IGNITION

A. Raise temperature slowly and evaporate to dryness.
B. Raise temperature to 250°C ± 5°C for 2 hours.
C. Raise temperature to 1000°C ± 5°C for 2 hours.

OPERATIONS / MATERIALS / QUANTITIES / REMARKS

Operation	Materials	Liters	Kgs.	Sp.Gr.20°	Fr.Pt.°C	Remarks
	One batch of concentrated 23 strip solution	2.0	2.1	1.1	0	75g. "23", (37.5 g/l)(160g. UNH)
FEED PREPARATION	Condensate	2.0	2.0	1.0	0	Three fold excess of 30% H_2O_2,
	H_2O_2	0.12	0.11	1.1	-	excess to destroy organic.
	Condensate	0.12	0.12	1.0	0	decomposition products.
	HNO_3	0.08	0.12	1.42	-	Two fold excess of 70% HNO_3
	Condensate	0.08	0.08	>1.0	<0	2.5M $Al(NO_3)_3$, 1.5M NaF
	$Al(NO_3)_3$, NaF solution	2.0	3.2	1.6	13	
	HNO_3	0.0012	-	1.41	-	
	Aqueous Feed	2.0	3.2	1.6	13	2.5M $Al(NO_3)_3$ 1.5M NaF 75g. "23"
EXTRACTION	Steam distilled diisopropyl ether (9 passes)	18	13.1	0.73	-15	Organic/aqueous volume ratio 1:1 per pass.
	Aqueous raffinate to waste	2.0	3.1	1.6	13	Aqueous raffinate: 2.5M $Al(NO_3)_3$, 1.5M NaF 7 mg "23" (0.01%)
	Organic extract	18.0	13.3	0.74	-15	
STRIPPING	0.2% (0.03M) HNO_3 (4 passes)	18.0	18.0	1.0	0	Aqueous/organic volume ratio 1:4. Four passes strips all 23 from solvent. 0.01% of total 23 lost.
	Stripped solvent to solvent recovery	18.0	13.1	0.73	-15	
	23 Strip	18.0	18.0	1.0	0	0.03M HNO_3 75g. "23" (160g. UNH)
EVAPORATION	Condensate (Contains trace of HNO_3 and all of diisopropyl ether carried over to the evaporator).	17.5	17.5	1.0	ca.0	After addition of each strip evaporate to 0.5 L to minimize 23 caking on boiler walls. Ca. 9 fold volume reduction factor: 36.
	Concentrated 23 strip solution	1.1	1.26	ca. 1.2	ca. 0	75g. "23", (160g. UNH) ca. 3M HNO_3 Trace of $Al(NO_3)_3$
CALCINATION	Condensate	1.1	1.16	1.06	<0	Condensate includes boiler washes from Evaporation-Volume Reduction.
	Flue gas		0.3			
	U_3O_8		0.089			89g. U_3O_8, (75g. "23") 0.01% loss. Gamma activity: 1.5 mr/hr/75g. "23" at contact.

"23" LABORATORY CONCENTRATION - BATCH SOLVENT EXTRACTION

Solvent: Diisopropyl Ether Basis: 75g. "23"

Salting Agent: Aluminum Nitrate

SHEET 1 of 1 SHEET

REF. DWG. TD-NONE

REV. NO.	REVISION	AP'P'D. BY	DATE

Drawn By	Ckd. By	App'd. By
RE-A	DK-O.	RL.

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