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Chemistry-Separation Processes for Plutonium and Uranium

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CHEMICAL PROCESSING OF IRRADIATED THORIUM OXIDE

STATUS REVIEW FOR MAY 1956 TO MAY 1957

E. M. Shank

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CHEMICAL PROCESSING OF IRRADIATED THORIUM OXIDE STATUS REVIEW FOR MAY, 1956 TO MAY, 1957

E. M. Shank

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0.0 ABSTRACT

Pilot plant processing of irradiated thorium, recent process development, and the remaining development program are discussed. The original one-cycle Thorex flowsheet has been modified to include a two-cycle co-decontamination flowsheet. Using the co-decontamination flowsheet, homogeneous-reactor-level-irradiated thorium decayed about 400 days has been processed to provide thorium and uranium products meeting tentative activity specifications for subsequent metal fabrication; these specifications exceed those for direct recycle of the products to a homogeneous reactor. A minimum of two solvent extraction cycles are necessary for processing short-decayed material. An alternate process being investigated on laboratory-scale is the preferential absorption of fission products on an inorganic ion-exchanger. Flowsheet modifications being investigated are emphasizing improved decontamination factors, solvent degradation, iodine removal, and recycle techniques. The remaining pilot plant program will consist of continued evaluation of the co-decontamination flowsheet, the processing of homogeneous-reactor-level-irradiated thorium decayed 30 days, and the demonstration of the recovery of isotopically pure U-233 by solvent extraction reprocessing of the decayed Pa-233 waste.

1.0 INTRODUCTION

The Thorex Pilot Plant has been in operation 28 months and has processed about 30 tons of irradiated thorium using the originally developed one-cycle flowsheet and a recently developed two-cycle flowsheet. In addition, about 0.6 tons of short-decayed thorium was processed in a scouting run using the one-cycle flowsheet. Although thorium oxide has not been processed, the discussion to be presented for thorium metal processing is directly applicable. The capacity of the pilot plant is sufficient to process the blanket material from three 450 megawatt homogeneous reactors.

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A review is given of the pilot plant operations during the past year, the recent process development, and the remaining development program. This review was presented at the Civilian Power Reactor Conference held at Oak Ridge in May 1957; the classified data included here were deleted for the Conference.

2.0 FLOWSHEET MODIFICATIONS

The original Thorex process was discussed at the Civilian Power Reactor Conference held at Oak Ridge in March 1956. This flowsheet consisted of dissolving the thorium metal in an excess of nitric acid; feed adjustment of the dissolver solution to remove the excess acid, to dehydrate silica, and to improve fission product decontamination factors; one solvent extraction cycle for decontaminating and separating the thorium and U-233, uranium isolation by ion-exchange for final purification and concentration; continuous thorium product concentration by evaporation; and continuous solvent recovery. On the basis of pilot plant data, it was concluded that the single-solvent-extraction cycle Thorex flowsheet would be adequate to process long-decayed homogeneous blanket material, but becomes marginal if adequate recycle handling provisions are not made. For this reason, a two-cycle co-decontamination flowsheet was developed in the laboratory and installed in the pilot plant.

The co-decontamination flowsheet is shown in Fig. 2-1. The dissolving and feed preparation steps have not been modified. The basic feature of the co-decontamination flowsheet is the substitution of an extraction-stripping cycle for the first cycle with the second cycle essentially the original flowsheet. Co-stripping of the products in the first cycle and partitioning of the thorium from the uranium in the second cycle permits two decontamination cycles for both products with

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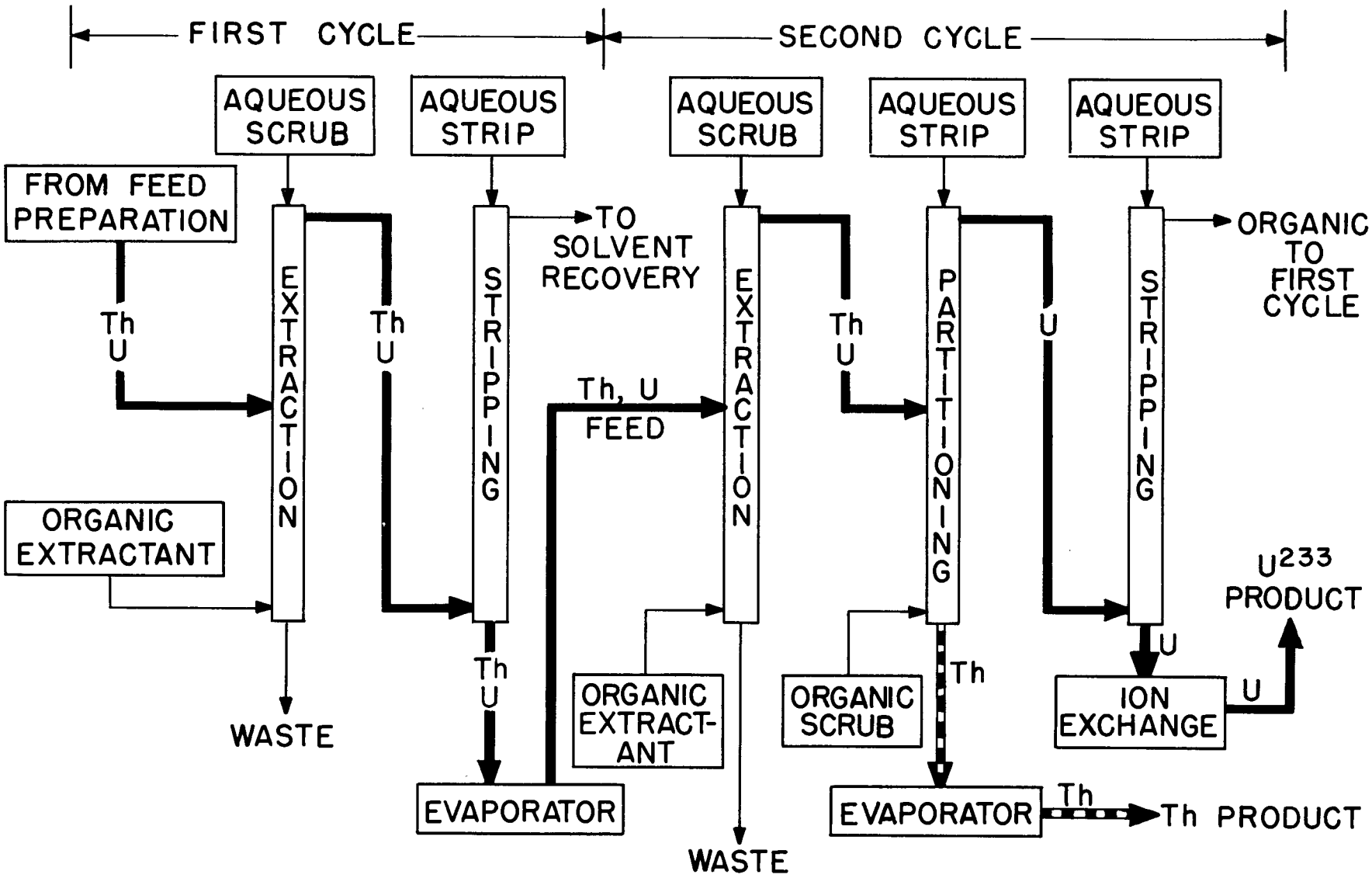

THOREX CO-DECONTAMINATION FLOWSHEET

FIGURE 2.1

a minimum of equipment. The co-stripped products are continuously concentrated to 1.5 M thorium by an intercycle evaporator; partly denitrated aluminum nitrate is added prior to concentration to provide the correct $Th:Al:OH^-$ concentration. Solvent from the second cycle is used as the extractant for the 1A-column. The solvent from the first cycle is routed to solvent recovery prior to use as the extractant for the 2A-column.

3.0 PILOT PLANT OPERATION

The Thorex Pilot Plant program has been divided into three general phases: the first phase was the one-cycle processing of thorium irradiated to about one-half the level anticipated in the homogeneous reactor (~1500 g/t), decayed 200 days; the second phase is the two-cycle processing of homogeneous-reactor-level-irradiated thorium (3500 g/t), decayed 400 days; and the third phase is the processing of 3500 g/t thorium, decayed 30 days, using the optimum flowsheet conditions developed.

During the past year, 11.5 tons of irradiated thorium was processed through one cycle during three extended runs, 0.6 tons of short-decayed thorium was processed during a scouting run, and 5 tons of thorium was processed by the co-decontamination flowsheet.

3.1 Observed Results

Following are some operating data for the first two phases and the scouting run for the third phase, a brief discussion of some of the operating experiences and conclusions, and an indication of the current effort in trying to solve some of the problems anticipated for phase 3 and in developing alternate processes.

The observed decontamination factors for the thorium product are shown in Table 3-1.

Table 3-1

Thorex Pilot Plant

Average Decontamination Factors - Thorium Product

Phase	1	2	3
g/t	1500	3500	2200
Decay, days	~ 200	~ 400	30
Constituents			
Gross γ	$3x10^3$	$1x10^5$	400
Pa	$3x10^3$	$1x10^4$	$1x10^4$
Ru	600	$4x10^3$	2
Zr-Nb	$3x10^3$	$3x10^5$	$1x10^3$
TRE	$2x10^5$	$2x10^6$	$1x10^3$

The decontamination factors shown for phase 1 were observed during the extended operation with the one-cycle flowsheet. The material varied widely as to irradiation level and decay time; however, the values of 1500 g/t, decayed 200 days indicated are the average for the material processed. The decontamination factors shown for phase 2 were observed during five extended development runs using the co-decontamination flowsheet. The irradiation and decay history was considerably less varied for these runs. It may be observed that the second cycle provides an additional decontamination factor of about 30 for gross gamma emitters, the major improvement observed being for Ru and Zr-Nb. The increased decontamination has enabled the pilot plant to consistently meet the tentative specifications for the thorium product required for subsequent processing to the metal. The decontamination factors shown for phase 3 were observed during a single scouting run using the one-cycle flowsheet. The major significance of the run was the almost complete loss of ruthenium decontamination.

The activity spectra of the thorium product for the material just described are shown in Table 3-2.

Table 3-2

Thorex Pilot Plant

Activity Spectra - Thorium Product

Phase		1	2	3
g/t		1500	3500	2200
Decay, days		~200	~ 400	30
Constituents	Specs.	Activity, cts/min/mg		
Gross γ	260	800	160	$5x10^5$
Pa	30	400	6	$1x10^5$
Ru	40	160	6	$6x10^5$
Zr-Nb	45	200	40	$2x10^4$
TRE β	40	80	5	$1x10^4$
Th-234 β	100	300	110	$2x10^4$

The specifications indicated were established from considerations of the biological hazards encountered during subsequent processing. With the decontamination factors just presented, the product from the one-cycle operation (phase 1) exceeded specifications by factors of 2 to 13. The processing of higher g/t, but longer decayed thorium (phase 2), through two solvent extraction cycles provided a product which met the tentative specifications. The product from the phase 3 scouting run exceeded specifications from 300 to greater than 10^4 (ruthenium). The Th-234 β specification requires decay time and cannot be helped by the process chemistry.

The decontamination factors observed for the uranium product are shown in Table 3-3.

Table 3-3

Thorex Pilot Plant

Average Decontamination Factors - Uranium Product

Phase	1	2	3
g/t	1500	3500	2200
Decay, days	~ 200	~ 400	30
Constituents	Decontamination Factors		
Gross γ	$3x10^4$	$3x10^5$	$4x10^4$
Pa	$2x10^5$	$3x10^5$	$1x10^6$
Ru	$4x10^3$	$2x10^5$	300
Zr-Nb	$5x10^4$	$8x10^5$	$5x10^4$
TRE	$2x10^7$	$9x10^8$	$3x10^7$

During phase 1 and 2 processing, gamma decontamination factors ranged from $4x10^3$ to $8x10^5$. While the decontamination factors observed for the uranium product from the phase 3 scouting run compare more favorably than those for the thorium product, the uranium product had a radiation intensity of 65 r/hr for about 300 g contained in 1.5 liters of solution.

The activity specifications for both the thorium and uranium products are considerably more stringent for subsequent metal fabrication than for recycle to the homogeneous reactor. Both products have consistently met the more stringent radiation specifications when processing homogeneous-reactor-level-irradiated thorium (3500 g/t) decayed about 400 days. Consequently, adequate recycle material should be provided for the homogeneous reactor even when processing 30-day decayed blanket material.

The activity spectra for the uranium product is shown in Table 3-4. The only specification for the uranium product is that 1.5 liters of

uranium solution containing about 300 g of uranium will not exceed 300 mr/hr at contact as measured by a hard-shell cutie-pie. The activity specifications have been calculated to serve as a guide. As may be observed, the one-cycle flowsheet (phase 1 and 3) did not provide specification uranium product. However, the two-cycle flowsheet has consistently provided a uranium product meeting specifications.

Table 3-4

Thorex Pilot Plant

Activity Spectra - Uranium Product

Phase		1	2	3
g/t		1500	3500	1500
Decay, days		~ 200	~400	30
Constituents	Specs.	Activity, cts/min/mg		
Gross γ	$9x10^3$	$3x10^4$	$3x10^3$	$2x10^6$
Pa	$1x10^3$	$1x10^3$	35	$4x10^4$
Ru	280	$2x10^4$	130	$2x10^6$
Zr-Nb	$8x10^3$	$3x10^3$	600	$1x10^5$
TRE	450	600	1	130
Radiation, mr/hr	<300	>300	150	$\sim 6.5x10^4$

Of interest to power reactor fuel reprocessing requirements are the ionic contaminants and the process losses. The major ionic contaminants observed in the Thorex products are shown in Table 3-5.

Table 3-5

Thorex Pilot Plant

Product Ionic Contaminants

	Thorium Product (ppm Th)		Uranium Product (ppm U)	
Contaminant	1-Cycle	2-Cycle	1-Cycle	2-Cycle
Al	30	70	1600	30
B	-	-	30	20
Ca-Mg	15	4	1700	8000
Fe, Cr, Ni	200	200	500	600
Others	100	100	200	200
U	10	15	-	-
Th	-	-	500	500

No firm specifications have been established for either product. The acceptability of these impurities will depend upon the specifications for the reactor fuels and upon the path of each during subsequent processing. To date, thoria prepared from the thorium product has been sufficiently pure chemically for use in the ORNL homogeneous reactor program. The uranium product has likewise been of adequate purity.

The losses observed during 1-cycle and 2-cycle operation are shown in Table 3-6.

Table 3-6

Thorex Pilot Plant

Process Losses

	Losses, %			
	Thorium		Uranium	
	1-Cycle	2-Cycle	1-Cycle	2-Cycle
Extraction	0.50	1.20	0.12	0.06
Partitioning	< 0.01	0.02	0.10	0.16
Stripping	< 0.01	0.07	0.03	0.06
Ion-Exchange	< 0.01	< 0.01	< 0.01	< 0.01
Product Recovery	< 0.01	< 0.01	< 0.01	< 0.01
TOTAL	0.52	1.30	0.26	0.29

The extraction losses for the thorium are not considered satisfactory. About 50% of this loss is attributed to the acid deficiency in the extraction sections and the remainder is attributed to recycle material to the first cycle and to degradation products from the intercycle evaporator in the second cycle. Means of reducing this loss are being studied in the laboratory. The uranium losses are considered satisfactory since about 50% of the loss is to the thorium product and is therefore recoverable.

3.2 Operating Experience

The Thorex Pilot Plant has been in operation for 28 months. Equipment performance during this period, while not trouble-free, has been good except for three major items. The uranium isolation system, although never the cause of plant shutdown, initially required almost constant maintenance to retain its operability and was the source of considerable personnel radiation exposure and alpha contamination in the plant. A redesign of this system eliminated essentially all of the problems encountered initially.

The most serious equipment malfunction, from the standpoint of on-stream efficiency, was the operation of the two first-cycle concatenated pulse columns during the one-cycle operation. The plant was essentially inoperable for about eight weeks as a result of the two concatenated columns air locking; the sources of air inleakage were located and eliminated.

The third equipment item to give trouble in operation has been the feed adjustment system. Here, the difficulties can be attributed principally to the process requirement of high temperature digestion and high concentration solutions. Recycle material containing dissolved organic material can be handled only under rigidly controlled conditions.

The modification of the plant for the co-decontamination flowsheet was accomplished by rerouting lines between existing equipment. It was necessary to increase the strip rate to the 1C-column for thorium stripping by 600%; this increased the total flow to the column to 133%, which exceeded the flooding capacity of the column at plant design capacity. Consequently, the plant capacity has been reduced by 25% when operating with the co-decontamination flowsheet.

The results observed from the sustained operation and the processing of the 0.6 ton of short-decayed thorium, although somewhat disconcerting, provided an excellent opportunity to evaluate the flowsheet under severe conditions and to determine areas where improvement is necessary. These areas might be classified into a few categories as follows:

1. General improvement in decontamination from fission products.
2. Waste and off-specification material recycle.
3. Solvent degradation and recovery.
4. Diluent specification and iodine buildup in the recovered solvent.

As these problems arose, several corrective measures were taken:

1. The feed adjustment step was modified to include a 1-hr digestion period at 155°C to complete the destruction of organic substances entrained in the recycled material and of complexing agents, and to increase ruthenium decontamination.
2. The practice of periodic removal of "interfacial cruds" from the columns was adopted.

Following the adoption of these measures, a part of the loss in decontamination was recovered.

The decontamination during the sustained runs was insufficient to produce a uranium product suitable for direct handling in subsequent processing. Accordingly, it became necessary to process the first cycle uranium product through a second solvent extraction cycle for further decontamination. The results of this emergency program may be summarized as follows:

1. The uranium product met the radiation intensity specification for direct handling, which is, that the radiation intensity of a polythene bottle containing 1.5 liters of 200 g U-233/liter solution shall not exceed 300 mr/hr at the surface of the bottle.
2. In reaching the above objectives the second uranium cycle demonstrated a gross decontamination factor of 10 and a factor of 10^3 for ruthenium.

Some of the observations made during the short-decayed scouting runs are:

1. The solvent was deteriorating at a much higher rate than previously experienced at any time as a result of the high radiation level of the feeds.
2. The concentration of solvent degradation products was higher in the process streams than experienced at any time previously.
3. The accelerated rate of buildup of highly radioactive solids at the column interfaces was probably due in part to the generally higher concentration of solvent degradation products and that in some instances interfered with proper operation of the columns.
4. The iodine content of the 30-day decayed feed was a factor of 10^4 greater than previously observed. During processing of the 30-day-decayed thorium, approximately 40 per cent of the iodine that was present in the feed was extracted into the organic solvent. Of this, only 0.5 per cent was stripped into the thorium and uranium products. The solvent recovery system did not remove the extracted iodine so that the recovered solvent became progressively more contaminated as the run continued.

4.0 PROCESS DEVELOPMENT

4.1 Thorex Process

The laboratory scale program has consisted of two main efforts: (1) pilot plant assistance and flowsheet improvement, and (2) evaluation of alternate processes. This development is directed towards decreasing the required decay time and has emphasized improved decontamination factors, solvent degradation, iodine removal, and recycle limitations.

The problem of improved decontamination has been studied and several flowsheet modifications were made. Also, additional fission product decontamination is obtained by the second solvent extraction cycle.

The recycle problem is not severe during short runs, since waste and off-specification material can be accumulated and reprocessed independently. These recycle materials can result from equipment or process malfunctions, elution operations, and interfacial cruds which are periodically jetted from the columns. The chemical behavior of these materials is little known, but the observed effects are generally increased solvent extraction losses, decreased decontamination factors, poorer column operating characteristics, and a more rapid buildup of highly radioactive solids which tend to be carried with the product streams. The requirements for handling recycle material are being investigated.

The problems of solvent degradation and recovery, and of diluent specifications were partly anticipated, but perhaps not to the extent as was noted during the short-decayed scouting run. Iodine buildup in the solvent was not prevented by any method of solvent recovery which had been developed.

Decalin is being investigated as an alternate diluent for Amsco. The major advantage to Decalin is the increased loading of the TBP with thorium. This increased loading permits a factor of about 5 increase in decontamination and assists mechanical operation by the elimination of the third phase.

A continuous interface purge system has been developed and will be installed in the pilot plant. This system involves the continuous removal of the interface through a glass wool filter medium and a phase separator. The clarified solution is then reintroduced into the column.

4.2 Alternate Processes

At the present time the only practical method for processing irradiated thorium oxide slurried in heavy water is to convert the oxide to a light water-nitrate solution and process by Thorex. Although this method is adequate, it is expensive unless one plant is built to process ThO_2 from several full-scale power reactors. Therefore, simple alternate methods of processing which can be operated as part of each reactor are being investigated. Attempts to leach uranium-233 and protactinium from irradiated ThO_2 were only partly successful since both are bound within the ThO_2 particles. As much as 70-80% of the U and Pa was removed by refluxing the ThO_2 in 12 N H_2SO_4 for 4 hr. However, this also converted most of the ThO_2 to $Th(SO_4)_2$.

Calculation shows that the recoil energy of fission products is sufficient to eject most of the fission products into the aqueous phase if the ThO_2 particles are $< 10\,\mu$. This means that it may be possible to remove fission product neutron poisons without destroying the ThO_2 particles. Scouting tests indicate that the fission products will adsorb irreversibly on the surface of the ThO_2 particles. Adsorption can be reduced by pretreating the ThO_2 with 0.05 M H_2SO_4 at room temperature for 24 hr, or by firing the ThO_2 at 1200-1600°C. However, it does not appear that adsorption can be completely eliminated by such pretreatment. Scouting tests have also shown that by pretreating the ThO_2 with dilute H_2SO_4 and co-slurrying the ThO_2 with an inorganic ion-exchanger such as magnesium silicate, the fission products will preferentially adsorb on the ion-exchanger. Separation of the ThO_2 and inorganic exchanger presents a problem. It may be possible to separate them in a classifier such as the hydroclone. At the present time the possibility of making a colloidal ion-exchanger, stable at 250°C, is being investigated.

5.0 SUMMARY AND PROGRAM

The following is a review of the status of the program and an indication of the program for the remainder of the development phase.

The demonstration of a flowsheet to process homogeneous-reactor-level-irradiated thorium (3500 g/t) decayed in excess of 400 days has been completed. On the basis of the results determined to date, it was concluded that the single-solvent-extraction cycle Thorex flowsheet, which includes ion-exchange isolation of U-233 becomes somewhat marginal if adequate provisions are not made for handling recycled material originating in sustained production. Additional cycles for both thorium and uranium are necessary for processing short-decayed material.

Sustained operation and preliminary evaluation of the processing of short-decayed material has indicated the need for further development in the following areas:

1. Improvement in the decontamination factors.
2. Reduction of recycle volumes from uranium isolation, minimizing crud formations at the column interfaces, and the elimination of solids carryover into the product streams, which produce off-specification products.
3. Solvent recovery--including iodine removal.
4. Improved diluent and means of obtaining diluents meeting these specifications.

The schedule for the remainder of the pilot plant program may be summarized as follows:

1. Continue evaluating the co-decontamination flowsheet during the processing of about six tons of long-decayed irradiated thorium.

2. The evaluation of short-decayed processing, using about 5 tons of thorium irradiated to 3000 to 4000 g/t, decayed 20 to 30 days. This thorium will be delivered to ORNL July 1957. Specifically, the pilot plant will be interested in decontamination, solvent stability, product purities, and protactinium-bearing waste storage.
3. The evaluation of improved solvent recovery methods, a new diluent, the third uranium cycle, and continuous interface crud removal equipment.
4. And finally, the demonstration of the recovery of isotopically-pure U-233 by solvent-extraction reprocessing of the decayed protactinium waste.