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BMI-243

FINAL REPORT

on

RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SAND

VOLUME I

by

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G. D. Calkins
R. B. Filbert, Jr.
A. E. Bearse
J. W. Clegg

September 15, 1950

BATTELLE MEMORIAL INSTITUTE

Columbus, Ohio

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Metallurgy - Raw Materials
Special Distribution

RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SAND

VOLUME I

by

G. D. Calkins
R. B. Filbert, Jr.
A. E. Bearse
J. W. Clegg

ABSTRACT

A process for recovering high yields of uranium and pure thorium from monazite sand concentrates is described. The monazite is decomposed by treatment with aqueous caustic soda and the resulting hydrous metal oxides are dissolved in a mineral acid. A separation from rare earths is accomplished by partial neutralization of the acid solution. The resulting precipitate containing the thorium and uranium is dissolved in nitric acid. The thorium and uranium are subsequently recovered and separated by solvent-extraction techniques using tributyl phosphate. An alternative separation technique involving ion exchange was also evaluated. By-products of the monazite process are rare earths, in a high state of purity, and trisodium phosphate.

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SECRETINTRODUCTION

The purpose of this project, under Contract No. AT-30-1-Gen-228, was to develop a process for recovering thorium and uranium from monazite sand concentrates. The specifications for the process were:

- (1) That the over-all recovery of thorium and uranium be in excess of 95 per cent.
- (2) That the thorium product contain less than one part each of uranium and rare earths per million parts of thorium.
- (3) That the uranium product be as pure as possible, without placing an undue burden on the over-all process.
- (4) That the bulk of the rare earths be recovered in a usable form.

Monazite sand consists mainly of the orthophosphates of the rare earth metals, together with thorium and smaller amounts of uranium. The major impurities associated with the commercial concentrates of monazite sand are titanium (as rutile), iron, and silica. A typical analysis is:

	<u>Per Cent</u>
ThO ₂	6.50
U ₃ O ₈	0.17
(RE) ₂ O ₃	59.2
P ₂ O ₅	26.0
TiO ₂	1.75
Fe ₂ O ₃	0.51
SiO ₂	2.16

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Monazite sand is the most important source of thorium, cerium, and other rare earths. The best known deposits are found in Brazil and India, but there is some monazite in the United States. The beach sands are upgraded by ore-dressing methods to produce the concentrates used in the present study.

The prior art consisted mainly of digestion with sulfuric acid to open the monazite sand, and fractional precipitations and crystallizations to recover the rare earths and thorium. Little regard was given to the recovery of uranium.

The data in this report and the companion report, BMI-243A, were originally discussed in topical, quarterly, and monthly reports (Appendix, Volume II).

Other reports which describe parts of the work on this project are:

- (1) BMI-242, August 15, 1950, Anion-Exchange Studies on the Separation and Purification of Uranium and Thorium.
- (2) BMI-244, September 15, 1950, Estimated Manufacturing Costs for the Recovery of Thorium and Uranium From Monazite Sand.
- (3) BMI-245, September 15, 1950, Solubility of Sodium Phosphate in Sodium Hydroxide Solutions.

This final report consists of two volumes: Volume I (BMI-243) summarizes the results of the monazite investigation, and Volume II (BMI-243A) presents experimental details and data and includes descriptions of alternative methods of attack.

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The experimental work described in this report was carried out during the period January 1, 1948, to June 30, 1950.

SUMMARY

A process has been developed for the recovery of pure thorium and uranium from monazite sand concentrates. This process consists of the following operations, Figure 1:

- (1) Reaction of the monazite sand with a hot concentrated aqueous solution of sodium hydroxide. This converts the metal phosphates of the sand to hydrous metal oxides and trisodium phosphate.
- (2) Separation of the hydrous metal oxides from the sodium phosphate and excess sodium hydroxide.
- (3) Dissolution of the hydrous metal oxides in hydrochloric acid.
- (4) Separation of the rare earths by selective hydroxide precipitation of the thorium and uranium.
- (5) Preparation of a feed solution for solvent extraction by dissolution of the hydroxide precipitate in nitric acid.
- (6) Solvent extraction of the nitrate solution to
(a) separate the thorium and uranium from impurities and (b) separate the uranium from the thorium.
- (7) Final precipitation of the thorium as thorium oxalate and of the uranium as uranyl hydroxide.

A method alternative to solvent extraction for purifying the thorium was investigated. This consisted of ion-exchange procedures.

In addition to accomplishing the major objective of developing a process for the recovery of thorium and uranium from monazite sand, this project has pointed out a new process for recovering rare earths. It is apparent that the first part of the monazite process developed

FIGURE 1. SIMPLIFIED SCHEMATIC DIAGRAM OF PROCESS FOR THE RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SAND

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under this contract could be made available to the rare earth industry without giving out information on the subsequent steps involving purification of thorium and uranium. An outstanding advantage to the Atomic Energy Commission of informing the industry of the caustic soda process is that the industry would then be in a position to produce a high-grade thorium-uranium hydroxide cake, which would be much more attractive for future processing than the present oxalate and fluoride sludges.

The over-all process described in this report was developed from data obtained in laboratory experiments and laboratory production-unit investigations. These data have been organized into a flowsheet and diagram, Figure A and Table A, Appendix, which show, as clearly as possible, the suitability of the process for commercial application.

The process is described in summary form in the following sections of this volume. The numbers appearing in parentheses in the text refer to the numbers of Figure A and Table A, Appendix, Volume I.

The experimental details and results on which this process was based are described in detail in Volume II (BMI-243A).

Grinding of Monazite Sand

For the process described in the following paragraphs, it was necessary to have a monazite sand feed with a particle size distribution as shown in Table 1. The size of the sand as received, before grinding, is also shown.

The sand is hard (5.5 Moh) but brittle, and was reduced by wet ball-milling in a Porox mill using flint pebbles. The sand (1)⁽¹⁾, with

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(1) Parenthetical numbers refer to numbered streams in Table A and Figure A, Appendix.

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TABLE 1. PARTICLE SIZE OF MONAZITE SAND (BRAZILIAN)

U. S. Sieve Series, Mesh Size	Weight Per Cent	
	Ground	Unground
-30 to +50	-	27.0
-50 to +70	-	36.5
-70 to +100	-	34.9
-100 to +200	-	1.6
-200 to +325	3.5	-
-325	96.5	-

water and pebbles, was charged to the mill and ground for the length of time needed to reduce it to the size range desired. In a large installation, continuous wet classifying with return of oversize would be desirable (3).

Since the discharge (2) from the mill contained excessive amounts of water, it was allowed to settle, and supernatant water was removed until the sand-water slurry contained 39 per cent of water. This sand slurry (4) was then fed to the reactor vessel.

Reaction of Monazite Sand With Caustic Soda Solution

The stainless-steel reaction vessel used had a capacity of about 10 gallons, and was fitted with external electrical heaters, a variable-speed agitator, a flush-bottom valve, and a thermocouple. Fed to the vessel at the start of a run were the sand slurry, commercial anhydrous caustic soda, water, and recovered caustic soda solution (6) recycled from a later step. In a commercial plant, the water and anhydrous caustic soda would be fed simultaneously as liquid caustic soda (5) containing 73 per cent NaOH. The amounts of materials charged were controlled so that initially the following conditions were obtained in the reactor:

Sand charge:	10 pounds (dry basis).
Ratio of NaOH to sand:	1.5.
Ratio of water to sand:	1.7.

This mixture was then heated to a temperature of 280°F. and maintained there, with moderate agitation, for three hours. This was sufficient time for the sand to react completely with the caustic soda.

Among constituents of the sand which behave differently than indicated by the equation are silica and uranium. The former forms a soluble sodium silicate and the latter, an insoluble sodium uranate.

After complete reaction, the mixture was diluted with a wash solution (8) from a later step, to keep the trisodium phosphate in solution when the temperature was lowered. The slurry was digested at 220°F. for one hour to facilitate later filtration. Its volume at this time was about 0.7 gallon per pound of sand charged. After digestion, the slurry was filtered.

Filtration of Reaction Slurry and Washing of Hydrous Oxide Cake

Filtration of the digested slurry to remove the hydrous oxides from the solution of trisodium phosphate and excess caustic soda was performed at a temperature of 180°F. under pressure. The steel filter consisted of one horizontal plate with a steam-jacketed chamber. The filter medium was a piece of 500-by-28-mesh Monel wire cloth. The slurry was delivered by a centrifugal pump to the filter and maintained at a gage pressure of about 60 pounds per square inch.

The filtrate (9), after it became clear, was sent to the evaporator for concentration and removal of trisodium phosphate. At completion of filtration, the hydrous oxide cake was washed in the filter with water (10) until practically all of the soluble elements in the cake were removed. The wash solution was saved and recycled to a subsequent reaction (8)

---

(1) RPO_4 represents monazite sand.

for its dilution prior to digestion. The cake (11) was blown dry with compressed air, and sent on to the acid-dissolution vessel. During blowing, some of the cerous oxide in the cake was oxidized to ceric oxide.

At this point, the hydrous oxide cake contained the following quantities of the constituents charged in the monazite sand:

<u>Constituent</u>	<u>Per Cent Accounted for in Hydrous Oxide Cake</u>
Uranium	96.9
Thorium	100
Rare earths	100
Phosphorus	0.3

Recovery of Excess Caustic Soda and Trisodium Phosphate

The filtrate (9) from the reaction contained unchanged about two-thirds of the caustic soda charged. The other third had been converted to trisodium phosphate. Both materials remained dissolved in the filtrate, provided its temperature did not fall below about 140°F. Below this temperature, a trisodium phosphate hydrate separated incompletely from the solution.

In order to achieve nearly complete separation of the phosphate, it was necessary to concentrate the filtrate by evaporation. At the same time, the caustic soda liquor remaining after removal of phosphate was prepared for recycling to a later reaction with monazite sand.

The evaporation was carried out in an open still kettle, steam jacketed and fitted with an agitator and thermocouple. The filtrate was evaporated until its boiling point reached 275°F., corresponding to an

NaOH concentration of about 47 per cent. At this concentration, 99 per cent of the trisodium phosphate had crystallized out of solution, and could be removed by filtration. After filtration, the caustic soda liquor (13) was ready for use either in sand reactions, or for later neutralization of acid solutions of hydrous oxide cake. The trisodium phosphate crystals (15, 17) were dried, and constituted a by-product of the process. Typical compositions of the dried crystals and liquor are shown in Table 2.

The recovered caustic soda liquor was divided into two parts; 45 per cent of it was recycled to the sand reaction (6), and 55 per cent was diluted with water and used for neutralization purposes later in the process (14).

By recycling only 45 per cent of the liquor, the maximum silica concentration reached in the reactor was held within allowable limits. When all the liquor had been recycled, the silica concentration built up to the point where it interfered with uranium recovery, and the uranium loss in the phosphate crystals became prohibitively high.

Dissolution of Hydrous Oxide Cake in Hydrochloric Acid

The hydrous oxide cake (11) was next placed in a 5-gallon steam-jacketed Pfaudler kettle fitted with a flush-bottom valve, agitator, and thermocouple. To it was added 37 per cent hydrochloric acid (18), 1.5 pounds of acid per pound of sand. The mixture was heated at 175°F. for 1 hour. This was sufficient time to allow complete dissolution of all the essential material in the cake. Presence of ceric oxide in the cake at this point caused evolution of chlorine gas from the mixture. A portion

TABLE 2. TYPICAL COMPOSITION OF DRIED TRISODIUM PHOSPHATE CRYSTALS AND
RECOVERED CAUSTIC SODA LIQUOR

Constituent	Composition, Per Cent	
	Crystals	Liquor
U	0.001	0.003
Na_3PO_4	86.8	0.53
Na_2SiO_3	0.39	1.48
NaOH	10.2	47.4

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of the cake, consisting mostly of unreacted rutile and zircon, remained insoluble in the acid and was carried along as suspended solid matter.

The acid solution (19) was then transferred to the neutralization vessel, and diluted with water (20). Concentration of a typical solution after dilution is shown in Table 3.

Separation of Thorium and Uranium by Selective Precipitation From Hydrochloric Acid Solution With Sodium Hydroxide

The diluted hydrochloric acid solution of hydrous oxide cake was partially neutralized by the addition of a sodium hydroxide solution. This operation was carried out at room temperature in an open stoneware vessel fitted with a high-speed agitator.

The sodium hydroxide solution (22) used was a portion of the caustic soda liquor recovered from the evaporation step (14), diluted with water (21). It was added to the acid solution until an equilibrium pH of about 5.8 was reached, as measured with a glass-calomel electrode system and a Beckman meter. The dilution of the caustic soda solution and of the acid solution with water was to make the precipitation at this point more nearly homogeneous. The slurry formed was quite fluid, and could be agitated easily. The effect of high localized pH during precipitation was minimized.

At pH 5.8, all of the thorium and uranium were precipitated, and about three per cent of the total amount of rare earths. The balance of the rare earths remained in solution as chlorides. The distribution of constituents between solution and precipitate at pH 5.8 is shown in Table 4.

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TABLE 3. COMPOSITION OF TYPICAL HYDROCHLORIC ACID
SOLUTION OF HYDROUS OXIDE CAKE, AFTER ADDITION OF
DILUTION WATER.

Constituent	Concentration, Grams Per Liter
U	0.21
Th	8.4
R.E.	74
Fe	0.85
Ti	1.3
P	0.05
Cl	24

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TABLE 4. DISTRIBUTION OF CONSTITUENTS BETWEEN SOLUTION AND PRECIPITATE
AFTER SELECTIVE PRECIPITATION OF THORIUM AND URANIUM AT pH 5.8

Constituent	Fraction, Per Cent of Total	
	In Solution	In Precipitate
U	0.7	99.3
Th	0.3	99.7
R.E.	97.7	2.3
Fe	0	100
Ti	0	100
P	0	100
Cl	99.9	0.1

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After neutralization, the slurry (23) was allowed to settle prior to decantation and filtration to separate the solids from the solution.

Sedimentation, Filtration of Slurry; Repulping and Washing of Thorium-Uranium Cake

The high degree of separation of constituents shown in Table 4 could not be realized unless occluded and retained liquor was efficiently removed from the solids by washing. For this purpose, a series of three filtrations and two washings was set up. Before each filtration, the slurry was settled and as much supernatant liquor as possible removed by decantation. After the first two filtrations, the cake was repulped and vigorously stirred in water. The filtrations were performed with an Eimco rotary drum vacuum filter, covered with heavy cotton duck cloth, and operated at 15 inches of mercury vacuum. The cake was continuously removed by a scraper, and repulped for the next filtration. After the third filtration, the cake was dried. The flowsheet in the Appendix, Figure A, clearly shows these operations.

The initial slurry (23) was settled, decanted, and filtered. The decantate (25) and filtrate (26) were combined, and the wet cake (27) was repulped in the combined decantate (34) and filtrate (35) from the third filtration cycle. The repulped slurry was again settled, decanted, and filtered. The decantate (29) and filtrate (30) were combined with the liquors from the first filtration. The wet cake (31) was repulped in water (32) containing 0.5 per cent of sodium nitrate, settled, decanted,

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and filtered. The final wet cake (36) was dried, the decantates (25, 29) and filtrates (26, 30) were combined (50) for subsequent rare earths recovery.

In general, one-half to two-thirds of the total liquor could be separated clear from the settled slurry by decantation. The thick slurry remaining yielded, on filtration, a cake containing about 20 per cent dry solids, which included all the material insoluble in acid, along with the thorium-uranium precipitate. The addition of sodium nitrate to the wash water was to improve the flocculating and settling characteristics of the solids. A brief heating of the slurries further hastened their settling.

The final wet cake (36) was oven-dried at 220°F. to constant weight. A typical analysis of such a cake (38) is given in Table 5.

Dissolution of Thorium-Uranium Cake in Nitric Acid; Preparation of Solvent-Extraction Feed Solution

The dried thorium-uranium cake was next dissolved in nitric acid to make a solution suitable for use in the solvent-extraction operation which followed. The final solution was to be 0.3 molar in thorium and 7.5 molar in free HNO_3 . Furthermore, at an intermediate step, it was necessary to heat the solution and filter off the acid-insoluble material and titanium phosphate which precipitated. The titanium separation could best be made at a 9.0-molar free HNO_3 concentration. Consequently, knowing the composition of the cake, it was possible to calculate the quantities of water and nitric acid needed to prepare the final solution.

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TABLE 5. COMPOSITION OF TYPICAL THORIUM-URANIUM
CAKE AFTER WASHING AND DRYING

Constituent	Composition, Per Cent
U	0.74
Th	36.4
R.E.	7.45
Fe	2.21
Ti	6.73
Si	4.47
P	0.44
Cl	0.36
Acid insoluble	23

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To the dried cake (38) was added all the nitric acid needed, which came from two sources: 10-molar acid (48.5 per cent HNO_3 by weight) from the nitric acid-recovery still (39), and 16-molar acid (71 per cent HNO_3 by weight) from storage (40). Then enough water was added to make a solution 9.0 molar in free acid and 0.36 molar in thorium. This mixture was heated at 175°F. for one hour, cooled, and filtered. The titanium phosphate which precipitated, and the insoluble gangue material, formed the cake (46), which was washed on the filter with water (45). Just enough water was used so that, on adding the wash (48) to the filtrate (44), the required feed solution (49) was produced.

The filter cake (46) was discarded, and the nitric acid solution (49) sent on to the solvent-extraction operation. Compositions of a typical cake and solution are shown in Table 6.

The recoveries of thorium and uranium in the solvent-extraction feed solution were 99 per cent and 96 per cent, respectively, based on the total amount of each accounted for in all exit streams from the process. The concentrations of these metals (particularly thorium) in the trisodium phosphate cake, recovered caustic soda liquor for neutralization, filtrates, washes, and gangue material were all quite low and their determination subject to greater accuracy than similar determinations of recoveries where concentrations were high. The combined losses at the above points were actually determined to be about 1 per cent for thorium and 4 per cent for uranium.

The actual recoveries of the metals were somewhat lower than indicated by the difference between the losses and 100 per cent. Actual

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TABLE 6. COMPOSITIONS OF TYPICAL SOLVENT-EXTRACTION FEED SOLUTION
AND GANGUE FILTER CAKE(1)

Constituent	Solution, Grams Per Liter	Cake, Weight Per Cent
U	1.4	0.01
Th	69.6	0.2
R.E.	12.4	1.5
Fe	3.7	0.4
Ti	3.9	7.4
Si	-	7.1
P	0.3	0.5
Zr(2)	-	0-15
Cl	0.8	-
HNO ₃ (free)	472	-
H ₂ O	-	63

(1) Not including filter-aid.

(2) Some monazite samples contained significant amounts of zircon.

thorium recovery was about 90 per cent, and uranium, 75 per cent, of the total charged with the sand. These latter figures do not include handling losses, and were taken over a period of two months, during which time 340 pounds of monazite were processed.

Recovery of Rare Earth Hydroxides

The combined decantates and filtrates (50) from the separation and washing of the selectively precipitated thorium-uranium cake contained about 97 per cent of the rare earths as chlorides in solution. They were isolated by precipitation with sodium hydroxide solution (51) recovered during the evaporation step. About 62 per cent of the unrecycled recovered caustic soda liquor (14) was used for this step. The other 38 per cent was used for the selective precipitation of thorium and uranium.

After precipitation of the rare earth hydroxides, the resulting slurry (52) was filtered and the hydroxide cake dried (57). The composition of a typical, dried, rare earth cake is given in Table 7. The filtrate (53, 54) from this separation contained only sodium chloride and sodium hydroxide in small quantities, and was discarded.

Solvent Extraction

The final purification of the thorium was accomplished by solvent extraction of the nitric acid solution (101). The operation consisted of three steps of countercurrent liquid-liquid extraction.

TABLE 7. COMPOSITION OF A TYPICAL DRIED RARE EARTH CAKE

Constituent	Composition, Weight Per Cent
U ⁽¹⁾	0.005
Th	0.05
R.E.	73
Fe ⁽¹⁾	0.02
Si ⁽¹⁾	0.4
P ⁽¹⁾	0.1
Cl	7.9

- (1) These constituents introduced partially with re-covered caustic liquor used to precipitate the rare earth hydroxide.

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The organic solvent used was a mixture of 50 per cent by volume of tributyl phosphate and Gulf Spray naphtha⁽¹⁾, hereafter abbreviated as 50-50 TBP-GSN. The tributyl phosphate was the active member of this solvent. The Gulf Spray naphtha, a highly purified kerosene fraction, served as a diluent, reducing the viscosity and density of the solvent for satisfactory countercurrent column operations.

The equipment used to obtain intimate contact between the aqueous and organic phases for each step was a one-inch-I.D. Scheibel-type multistage column⁽²⁾. The column consisted of a series of 14 mixing stages, separated by calming sections. The mixing was accomplished by agitators attached to a shaft situated in the center of the column. The calming sections were rolls of fine wire mesh (stainless steel) wrapped around the shaft.

The three steps of the solvent-extraction operation were:

- (1) Extraction of the thorium and uranium.
- (2) Selective stripping of the thorium.
- (3) Stripping of the uranium.

Extraction of Thorium and Uranium

The extraction step consisted of the separation of the impurities, rare earths, iron, and titanium, by transfer of the thorium and uranium to the organic solvent. In this case, advantage was taken of the higher distribution coefficients, organic over aqueous, of the thorium and uranium, as compared with those of the impurities from a concentrated

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- (1) Gulf Oil Company.
  - (2) Otto H. York Company, East Orange, New Jersey.

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(6.0-molar, 32 per cent) nitric acid solution. The distribution ratio for thorium is approximately 100 times, and for uranium, about 1000 times, greater than those for the impurities.

The aqueous feed solution (101) was introduced about a third of the way from the top of the column. The organic extractant (103), 50-50 TBF-GSN, entered at the bottom of the column. A scrub solution (102), 6-molar nitric acid, was used at the top of the column to wash the small amounts of extracted impurities out of the solvent. The volume ratios of scrub:feed:solvent were 0.5:1.0:1.5.

A typical extraction run produced an organic phase containing less than 44 parts of rare earths, 100 parts of iron, 200 parts of titanium, and less than 70 parts of phosphorus ($\text{PO}_4^{\equiv}$) per million parts of thorium, Table 8. The extraction of the thorium and uranium was greater than 99 per cent.

The stage efficiency in the extraction section of the column was approximately 60 per cent. This indicated a height of an equivalent theoretical stage of about 4.2 inches compared to an actual stage height (of mixing and calming sections) of 2.5 inches.

Selective Stripping of Thorium

The next step in the solvent-extraction operation was the separation of the thorium from the uranium and the organic solvent. This was accomplished by selectively stripping the thorium into an aqueous solution while the uranium remained in the organic solvent. In this case, advantage was taken of the fact that, from a dilute nitric acid

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TABLE 8. PURIFICATION OF THORIUM AND URANIUM BY EXTRACTION

		Aqueous Feed to Column	Organic Solvent From Column
Th, per cent		5	5.3
U, parts per million of Th		20×10^3	20×10^3
R.E.,	Ditto	18×10^4	<44
Ti,	"	56×10^3	200
Fe,	"	53×10^3	100
$\text{PO}_4^=$ as P,	"	62×10^2	<70
HNO_3 , per cent		34	7.9

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solution (about 0.5-molar), the thorium distributes in favor of the aqueous phase, while the uranium distributes in favor of the organic phase. The difference in distribution ratios is a factor of ten.

For this step, the organic feed solution (105), which was about 1.0 molar in nitric acid, was introduced near the center of the Scheibel column described in the preceding section. The strip solution, deionized water (109), entered at the top of the column. A scrub solution consisting of 50-50 TBP-GSN which had been made 0.4 molar in nitric acid (108) was used at the bottom of the column to wash small amounts of uranium from the aqueous phase back to the organic phase. While nitric acid was not added directly to the aqueous stripping solution, a sufficient amount of the acid transferred from the organic phase to the aqueous phase in the top section of the column to keep the uranium in the organic solvent. The volume ratios of the strip:feed:scrub were 1.5:1:0.5.

Analyses of typical products from the selective-stripping step indicated that the concentration of the uranium in the aqueous thorium product solution (110) had been reduced to 15 parts per million parts of thorium and that the concentration of the thorium in the uranium had been reduced to less than 2000 parts per million parts of uranium, Table 9.

While the concentration of the uranium in the thorium product solution was not reduced to the specified value of less than one part per million parts of thorium, this could have been accomplished if a column with a greater number of stages had been used.

Stripping of Uranium

The final step in the solvent-extraction operation was the removal of the uranium from the organic solvent. This was done in the Scheibel

TABLE 9. SEPARATION OF THORIUM FROM URANIUM BY SELECTIVE STRIPPING⁽¹⁾

	Organic Feed Solution	Aqueous Solution, Out of Column	Organic Solution, Out of Column
Th, per cent	5.3	2.9	-
U, per cent	0.11	-	0.07
U, parts per million of Th	20×10^3	15	-
Th, parts per million of U	50×10^6	-	<2000

(1) Also see Table 10, Analysis of Thorium Oxalate Product, and Table 11, Analysis of Uranyl Hydroxide Product.

column by stripping the solvent (116) with deionized water (117). The nitric acid concentration of the solvent had been reduced during the selective-stripping step to a value (<0.05 -molar) low enough that it was possible to strip the uranium into a volume of water one-third that of the organic phase.

Recycle, Pretreatment, and Losses of Organic Solvent

Following the removal of the uranium, the organic solvent may be recycled (123).

While insufficient data are available to indicate the number of times a given batch of solvent may be recycled without special treatment, it has been found in other work⁽¹⁾ that, in a similar operation, the solvent was still satisfactory after five cycles.

Raw solvent, as received, usually contains small amounts of mono- and dibutyl phosphate esters. These esters react with thorium and uranium, forming compounds which are extremely difficult to strip from the solvent. These esters may be removed from the solvent by converting them to sodium salts with a solution of sodium hydroxide, and subsequently washing with water.

Until further information is available, it is recommended that the solvent be treated as the raw solvent after each five cycles of the solvent-extraction operation (127, 129, 131).

The raw solvent pretreatment⁽¹⁾ consisted of the following:

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(1) ORNL 557, January, 1950.

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- (1) A wash with 0.2 volume of 1.5-molar nitric acid.
- (2) Two washes with 0.2 volume of 1.0-molar sodium hydroxide.
- (3) Water washes until neutral.

The over-all losses of solvent during the extraction process have been estimated to be 0.5 per cent of the tributyl phosphate and 0.2 per cent of the Gulf Spray naphtha. The loss of tributyl phosphate was greater, because it is soluble to a small extent in the aqueous phases.

The losses would be made up by feeding in fresh solvent (125 and 126).

Recovery of Nitric Acid

Process economics require that as large a portion of the nitric acid as possible be recovered for recycle purposes. The bulk (84 per cent) of the nitric acid used in the solvent-extraction operation was found in the aqueous raffinate from the extraction column (104). This solution was approximately 6 molar in nitric acid.

It has been estimated, with the aid of experimental evidence, that about 85 per cent of the nitric acid in the raffinate from the first column could be recovered by rectification in the following solutions:

- (1) Eight per cent of the HNO_3 as a 1.5-molar solution to be used for solvent pretreatment and equipment clean-up (137).
- (2) Thirty-three per cent of the HNO_3 as a 6-molar solution to be used as the scrub solution for the extraction column (136).
- (3) Forty-four per cent of the HNO_3 as a 10-molar solution to be used for the preparation of the solvent-extraction feed solution (135).

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The acid residue from the distillation (138) may be neutralized with the excess sodium hydroxide from the solvent pretreatment (139). The neutralized solution may then be discarded (140).

Preparation of Thorium Product

Previous investigations of other laboratories⁽¹⁾ have indicated that the oxalate is the most desirable form for the metallurgical treatment required to produce thorium metal. In view of this fact, thorium oxalate was selected as the end product of this process.

The precipitation of thorium oxalate from an acid solution resulted in some purification, because the oxalates of some of the impurities (U, Ti, Fe) are soluble in acid solution.

Thorium Oxalate Precipitation

The thorium oxalate was precipitated directly from the aqueous thorium product solution (110), from the selective-stripping column. This solution contained about 4 per cent of nitric acid. A 5-per cent excess of a 10-per cent oxalic acid solution (111) was used for the precipitation.

The resultant thorium oxalate slurry (112) was filtered, and the precipitate was washed to improve the removal of soluble impurities and dried.

Analysis of Thorium Oxalate Product

The thorium oxalate, the end product of this process, was analyzed spectrographically for all impurities except uranium, which was determined fluorometrically, Table 10.

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(1) TID 66, January, 1949.

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TABLE 10. ANALYSIS OF THORIUM OXALATE PRODUCT

Element	Thorium Oxalate, Parts Per Million Parts of Thorium	Oxalate Filtrate, Parts Per Million Parts of Thorium Separated
U	2(1)	13 ⁽¹⁾
RE	<44(2)	<100 ⁽³⁾
Ti	5(2)	195(3)
Fe	20(2)	95(3)
P	<20(3)	<70 ⁽³⁾

- (1) Analyzed fluorometrically.
(2) Analyzed spectrographically.
(3) Analyzed chemically.

Precipitation of Uranium Product

The uranium was removed from the aqueous uranium product solution (118) by precipitation with ammonium hydroxide.

Analysis of a typical uranyl hydroxide precipitate is shown in Table 11.

Ion Exchange, An Alternative Method
for Separating Uranium and Thorium

During the investigation of methods for separating thorium and uranium, ion-exchange procedures were developed as an alternative to solvent extraction. The details of this study are discussed in report BMI-242, August 15, 1950, and in Volume II (BMI-243A) of this report.

The operation consisted of the selective adsorption of the uranium by an anion-exchange resin, Amberlite IRA-400⁽¹⁾, from a concentrated chloride solution. This method was successful with the hydrochloric acid solution (19). The uranium concentration was reduced to the order of less than one part per million parts of thorium.

The processing after this operation would be similar to that shown in the flowsheet, Figure A, except that the selective-stripping step of the solvent-extraction operation would be eliminated.

Radiation Hazards

Since monazite sand contains both uranium and thorium, and more important, their radioactive decay products, there is a radiation hazard associated with its processing. Fortunately, the hazard appears to be

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(1) Rohm and Haas Company.

TABLE 11. ANALYSIS OF URANYL HYDROXIDE PRODUCT

<u>Element</u>	<u>Parts Per Million Parts of Uranium</u>
Th	<2000(1)
RE	<4000(2)
Ti	<1000(2)
Fe	300(2)
P	100(2)

(1) Analyzed chemically.

(2) Analyzed spectrographically.

serious at only a few points in the process described. They are:

- (1) handling and storage of dry monazite sand, particularly when ground;
- (2) handling and storage of the dried thorium-uranium cake (38) after separation from the rare earths; and (3) disposal of the raffinate from the extraction column after nitric acid recovery (138, 140). Any process such as this one, operating over a considerable period of time, would, necessarily, involve possible exposure of operators to radiation.

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APPENDIX - VOLUME I

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FIGURE A. FLOWSHEET FOR THE RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SAND

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TABLE A. FLOWSHEET DATA-RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SAND

Stream No. (See Figure 4)	Name of Stream	Distribution, per Cent of Total in Monazite Sand(1)										Composition				
		Th	U	R.E.	Ti	Fe	Si	P	Na ⁺ as NaOH	Cl ⁻ as HCl	HNO ₃ as HNO ₃	Th	U	R.E.	Ti	Fe
1	Monazite sand	100	100	100	100	100	100	100	-	-	-	5.71	0.12(2)	50.6	1.05	0.36
2	Ball-mill discharge	100	100	100	100	100	100	100	-	-	-	2.86	0.06(2)	25.3	0.53	0.18
3	Classifier-settler return	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	Wet sand charge	100	100	100	100	100	100	100	-	-	-	3.48	0.07(2)	30.8	0.64	0.22
5	Make-up caustic soda - 73% liquid	-	-	-	-	-	-	100	-	-	-	-	-	-	-	-
6	Recycle caustic soda liquor	-	2.2	-	-	3.6	31	0.7	40.9	-	-	-	0.003	-	-	0.014
7	Water-vapor loss from reactor	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
8	Dilution wash solution	-	0.2	-	-	0.2	3	2.9	4.9	-	-	-	-	-	-	-
9	Caustic filtrate	-	5.3	-	-	8.6	75	100.4	137.7	-	-	-	0.001	-	-	0.004
10	Wash water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
11	Hydrous oxide cake	100	96.9	100	100	95	56	0.3	8.4	-	-	4.46	0.09	39.5	0.82	0.27
12	Water vapor from evaporator	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
13	Recovered caustic soda liquor	-	4.9	-	-	8	63	1.6	91.0	-	-	-	0.003	-	-	0.014
14	Neutralization caustic soda liquor	-	2.7	-	-	4.4	38	0.9	49.9	-	-	-	0.003	-	-	0.014
15	Wet sodium phosphate crystals	-	0.4	-	-	0.6	6	98.8	46.7	-	-	-	0.001	-	-	0.003
16	Water from crystal dryer	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
17	Dry sodium phosphate crystals	-	0.4	-	-	0.6	6	98.8	46.7	-	-	-	0.001	-	-	0.003
18	Hydrochloric acid - 37%	-	-	-	-	-	-	-	-	100	-	-	-	-	-	-
19	Chloride solution	100	96.9	100	100	95	56	0.3	-	100	-	2.06	0.045	18.2	0.38	0.12
20	Dilution water for acid solution	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
21	Dilution water for neut. caustic soda	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
22	Caustic soda solution for partial neut.	-	1.0	-	-	1.6	14	0.3	18.7	-	-	-	0.002	-	-	0.01
23	Partial neut. slurry	100	97.9	100	100	96.6	70	0.6	-	100	-	0.68	0.014	6.0	0.12	0.04
24	Settled slurry No. 1	99.7	97.3	11.1	-	-	-	-	-	-	-	1.65	0.034	1.63	-	-
25	Decantate No. 1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
26	Filtrate No. 1	0.3	0.6	88.9	-	-	-	-	-	90.9	-	0.002	0.0001	6.1	-	-
27	Wet cake No. 1	99.7	97.3	11.1	-	-	-	-	-	-	-	5.33	0.109	5.27	-	-
28	Settled slurry No. 2	99.7	97.3	3.4	-	-	-	-	-	-	-	1.98	0.041	0.60	-	-
29	Decantate No. 2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
30	Filtrate No. 2	0.3	0.6	8.8	-	-	-	-	-	9.0	-	0.002	0.0001	0.60	-	-
31	Wet cake No. 2	99.7	97.3	3.4	-	-	-	-	-	-	-	7.41	0.152	2.25	-	-
32	Wash water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
33	Settled slurry No. 3	99.4	96.7	2.3	-	-	-	-	-	-	-	2.32	0.047	0.48	-	-
34	Decantate No. 3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
35	Filtrate No. 3	0.3	0.6	1.1	-	-	-	-	-	1.1	-	0.002	0.0001	0.079	-	-
36	Wet cake No. 3	99.4	96.7	2.3	-	-	-	-	-	0.1	-	7.78	0.16	1.60	-	-
37	Water from cake dryer	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
38	Dry cake	99.4	96.7	2.3	-	-	-	-	-	0.1	-	36.4	0.74	7.45	6.73	2.21
39	10-molar nitric acid solution - 48.5%	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
40	16-molar nitric acid solution - 71%	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

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SILM AND URANIUM FROM MONAZITE SAND

Distribution, per Cent of Total in Monazite Sand (1)										Composition of Stream, Per Cent										Quantity Per Pound of Thorium Produced			
Th	U	R.E.	Ti	Fe	Si	P	NaOH	as HCl	NO ₃ as HNO ₃	Th	U	R.E.	Ti	Fe	Si	P	NaOH	as HCl	NO ₃ as HNO ₃	H ₂ O	Pounds	Gallons	
100	100	100	100	100	100	100	-	-	-	5.71	0.12(2)	50.6	1.05	0.36	1.00	11.34	-	-	-	0	17.7	-	
100	100	100	100	100	100	100	-	-	-	2.86	0.06(2)	25.3	0.53	0.18	0.50	5.67	-	-	-	50	35.4	2.56	
100	100	100	100	100	100	100	-	-	-	3.48	0.07(2)	30.8	0.64	0.22	0.61	6.90	-	-	-	38.2	29.1	1.80	
-	-	-	-	-	-	-	100	-	-	-	-	-	-	-	-	-	73.0	-	-	25	26.3	1.85	
-	2.2	-	-	3.6	31	0.7	40.3	-	-	-	0.003	-	-	0.014	0.34	0.10	47.4	-	-	50	16.1	1.31	
-	0.2	-	-	0.2	3	2.9	4.5	-	-	-	-	-	-	-	0.007	0.07	0.74	-	-	100	14.1	1.69	
-	5.3	-	-	5.6	75	100.4	137.7	-	-	-	0.001	-	-	0.004	0.11	1.64	14.8	-	-	75.8	84.1	9.83	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	100	123	11.6	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	100	88.5	10.5
100	96.9	100	100	95	56	0.3	8.4	-	-	4.46	0.09	38.5	0.82	0.27	0.44	0.03	-	-	-	33	22.7	-	
100	4.9	-	-	8	53	1.6	91.0	-	-	-	0.003	-	-	0.014	0.34	0.10	47.4	-	-	100	73.6	8.85	
100	2.7	-	-	4.4	38	0.9	49.9	-	-	-	0.003	-	-	0.014	0.34	0.10	47.4	-	-	50	35.8	2.92	
100	0.4	-	-	0.6	6	95.8	46.7	-	-	-	0.001	-	-	0.003	0.08	14.4	9.0	-	-	50	19.7	1.61	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	13	13.9	-	
-	0.4	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	100	1.72	0.21	
-	0.4	-	-	0.6	6	95.8	46.7	-	-	-	0.001	-	-	0.003	0.09	16.4	10.2	-	-	63	12.2	-	
100	96.9	100	100	95	56	0.3	-	100	-	2.06	0.045	18.2	0.38	0.12	0.20	0.012	-	37	-	-	26.6	2.69	
-	-	-	-	-	-	-	-	100	-	-	-	-	-	-	-	-	-	20	-	-	49.2	3.94	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	100	89.6	11.7	
-	1.0	-	-	1.6	14	0.3	18.7	-	-	-	0.002	-	-	0.01	0.23	0.06	32.4	-	-	100	9.2	1.05	
97.9	97.3	11.1	100	96.6	70	0.6	-	100	-	0.68	0.014	6.0	0.12	0.04	0.08	0.01	-	6.6	-	-	56	10.6	0.96
99.7	97.3	-	-	-	-	-	-	-	-	1.65	0.034	1.63	-	-	-	-	-	-	-	-	150	15.3	-
-	0.6	88.9	-	-	-	-	-	90.9	-	0.002	0.0001	6.1	-	-	-	-	-	6.85	-	-	-	61.1	6.11
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	38.5	9.51	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	42.1	4.53	
99.9	97.3	11.1	-	-	-	-	-	9.1	-	5.33	0.109	5.27	-	-	0.65	-	-	4.75	-	70.4	18.9	-	
99.9	97.3	3.4	-	-	-	-	-	-	-	1.98	0.041	0.60	-	-	-	-	-	-	-	-	51.0	5.86	
0.3	0.6	3.8	-	-	-	-	-	9.0	-	0.002	0.0001	0.60	-	-	-	-	-	0.68	-	-	93.3	11.1	
99.7	97.3	3.4	-	-	-	-	-	1.2	-	7.41	0.152	2.25	-	-	0.91	-	-	-	-	-	37.3	4.44	
99.4	96.7	2.3	-	-	-	-	-	-	-	2.32	0.047	0.48	-	-	-	-	-	-	-	79.3	13.6	-	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	100	125	15.0	
0.3	0.6	1.1	-	-	-	-	-	1.1	-	0.002	0.0001	0.079	-	-	-	-	-	0.86	-	-	43.3	5.10	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	94.9	11.3	
99.4	96.7	2.3	-	-	-	-	-	0.1	-	7.78	0.16	1.60	-	-	0.96	-	-	0.76	-	-	30.4	3.63	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	80.4	12.9	-	
-	-	-	-	-	-	-	-	-	-	36.4	0.74	7.45	6.73	2.21	4.47	0.44	-	0.36	-	100	10.1	1.21	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0	2.76	-	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	51.5	7.46	0.69	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	29	6.75	0.57	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	48.5	71	-	

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TABLE A. (Continued)

Stream No. (See Figure A)		Distribution, Per Cent of Total in Monazite Sand ⁽¹⁾									Composition of Stream, Per Cent						
		Th	U	R.E.	Ti	Fe	Si	P	Na ⁺	Cl ⁻	NO ₃ ⁻	Th	U	R.E.	Ti	Fe	Si
									as NaOH	as HCl	as HNO ₃						
41	Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
42	Nitrate solution	99.4	96.7	2.3	100	96.6	70	0.6	-	0.1	100	5.52	0.113	1.13	1.02	0.33	0.67
43	Filter aid slurry	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
44	Filtered nitrate solution	92.9	90.1	1.9	28.2	79.9	-	0.19	-	0.09	93.8	5.63	0.115	1.01	0.31	0.3	-
45	Wash water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
46	Wet insoluble residue ⁽³⁾	0.4	0.7	0.3	70	11.6	70	0.4	-	-	-	0.23	0.01	1.54	7.43	0.42	7.06
47	Adjusting nitric acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
48	Adjusting wash water	6.1	5.9	0.1	1.8	5.1	-	0.01	-	0.01	6.2	1.8	0.04	0.3	0.1	0.1	-
49	Solvent-extraction feed solution	99.0	96.0	2.0	30	85	-	0.2	-	0.1	100	4.97	0.101	0.69	0.28	0.27	-
50	Combined decantates and filtrates	0.6	1.2	97.7	-	-	-	-	-	99.9	-	0.002	0.0001	3.35	-	-	-
51	Caustic soda solution for rare earth pptn.	-	1.7	-	-	2.8	24	0.6	31.2	-	-	-	0.002	-	-	0.01	0.23
52	Settled rare earth slurry	0.6	2.9	97.7	-	2.8	24	0.6	-	99.9	-	0.004	0.0004	6.06	-	0.001	0.03
53	Decantate waste	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
54	Filtrate waste	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
55	Wet rare earth cake	0.6	2.9	97.7	-	2.8	24	0.6	-	-	-	0.01	0.001	16.2	-	0.003	0.08
56	Water from cake dryer	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
57	Dry rare earth cake	0.6	2.9	97.7	-	2.8	24	0.6	-	-	-	0.05	0.005	73.1	-	0.014	0.35

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Distribution, Per Cent of Total in Monazite Sand (1)

	Composition of Stream, Per Cent										Quantity Per		
	Th	U	R.E.	Ti	Fe	Si	P	NaOH	Cl ⁻ as HCl	NO ₃ ⁻ as HNO ₃	H ₂ O	Pounds Produced	Gallons
.....	96.7	96.7	2.3	100	96.6	70	0.6	-	0.1	100	100	1.18	0.14
.....	90.1	90.1	1.9	28.2	79.9	-	0.19	-	-	46.1	-	18.2	1.45
.....	-	-	-	-	-	-	-	-	-	47.1	90	3.0	0.32
.....	-	-	-	-	-	-	-	-	-	-	100	16.7	1.35
.....	0.7	0.7	0.3	70	11.6	70	0.4	-	-	-	-	3.48	0.42
.....	-	-	-	-	-	-	-	-	-	-	62.6	1.75	-
.....	5.9	5.9	0.1	1.8	5.1	-	0.01	-	0.06	71	29	0	0
.....	96.0	96.0	2.0	30	85	-	0.2	-	0.02	14.9	-	3.48	0.38
.....	1.2	1.2	97.7	-	-	-	-	-	0.05	41.5	-	20.2	1.73
.....	-	-	-	-	-	-	-	-	3.76	-	-	261	29.3
.....	1.7	1.7	-	-	2.8	24	0.6	31.2	-	-	66	18.2	1.64
.....	2.9	2.9	97.7	-	2.8	24	0.6	-	-	-	-	144	15.3
.....	-	-	-	-	-	-	-	-	-	-	-	-	15.7
.....	2.9	2.9	97.7	-	2.8	24	0.6	-	3.9	-	-	136	10.3
.....	-	-	-	-	-	-	-	-	1.8	-	81	54.1	-
.....	-	-	-	-	-	-	-	-	-	-	100	41.9	5.03
.....	2.9	2.9	97.7	-	2.8	24	0.6	-	8.1	-	-	12.2	-

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Distribution, Per Cent of Total In Monazite Sand										Composition of Stream, Per Cent							Composition, Parts per Million					Quantity Per Pound of Thorium Produced	
Th	U	R.E.	Ti	Fe	P as PO ₄	Th	U	R.E.	Ti	Fe	P as PO ₄	H ₂ O ₃	TBP	GSN	H ₂ C ₂ O ₄	U	P.E.	Ti	Fe	P as PO ₄	Pounds	Gallons	
99	96	2.0	30	85	0.2	5.0	0.10	0.90	0.3	0.3	0.03	34	-	-	-	20x10 ³	10x10 ⁴	50x10 ³	5.3x10 ³	6x10 ²	23	1.7	
-	-	-	-	-	-	-	-	-	-	-	-	31	-	-	-	-	-	-	-	-	1.6	1.80	
0.1	0.1	2.0	30	85	0.2	-	-	-	-	-	-	-	55	44	-	-	-	-	-	-	19	2.6	
99	96	<5x10 ⁻⁴	0.1	0.2	<2x10 ⁻³	5.3	0.11	0.7	0.2	0.2	0.02	31	0.01	-	-	20x10 ³	-	-	-	-	27	2.6	
-	-	-	-	-	-	-	-	-	-	-	-	7.9	52	42	-	-	-	-	-	-	20	2.6	
-	-	-	-	-	-	-	-	-	-	-	-	-	55	44	-	-	-	-	-	-	9.5	1.3	
-	-	-	-	-	-	-	-	-	-	-	-	71	-	-	-	-	-	-	-	-	0.39	0.033	
-	-	-	-	-	-	-	-	-	-	-	-	2.3	54	43	-	-	-	-	-	-	10	1.3	
-	-	-	-	-	-	-	-	-	-	-	-	3.9	0.02	-	-	-	-	-	-	-	33	3.9	
99	7x10 ⁻²	<5x10 ⁻⁴	0.1	0.2	<2x10 ⁻³	2.9	4x10 ⁻⁵	-	-	-	-	-	-	-	-	15	<44	200	100	<70	34	3.9	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	10	-	-	-	-	-	11	1.3	
99	7x10 ⁻²	<5x10 ⁻⁴	0.1	0.2	<2x10 ⁻³	2.9	4x10 ⁻⁵	-	-	-	-	3.8	0.02	-	-	15	<44	200	100	<70	45	5.2	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	34	4.0	
99	1x10 ⁻²	<5x10 ⁻⁴	5x10 ⁻³	4x10 ⁻²	<2x10 ⁻³	56	1x10 ⁻⁴	-	-	-	-	-	-	-	44	2	<44	5	20	<50	1.8	-	
-	6x10 ⁻²	<5x10 ⁻⁴	0.1	0.2	<2x10 ⁻³	<1x10 ⁻⁴	2x10 ⁻⁵	-	-	-	-	2.0	0.02	-	0.6	-	-	-	-	-	70	0.2	
Composition, Parts per Million																							
Composition, Parts per Million										Composition, Parts per Million													
Th	U	R.E.	Ti	Fe	P	Th	U	R.E.	Ti	Fe	P	Th	U	R.E.	Ti	Fe	P	Th	U	R.E.	Ti	Fe	P
96	96	<1x10 ⁻³	<1x10 ⁻³	1x10 ⁻²	1x10 ⁻⁴	<1x10 ⁻⁴	7x10 ⁻²	-	-	-	-	0.1	54	43	-	<2000	<4000	<1000	300	100	29	3.9	
-	-	-	-	-	-	-	-	-	-	-	-	<0.05	0.01	-	-	<2000	<4000	<1000	300	100	11	1.3	
96	96	<1x10 ⁻³	<1x10 ⁻³	1x10 ⁻²	1x10 ⁻⁴	<3x10 ⁻⁴	3x10 ⁻²	-	-	-	-	-	-	-	-	<2000	<4000	<1000	300	100	0.007	0.003	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<2000	<4000	<1000	300	100	11	1.3	
96	96	<1x10 ⁻³	<1x10 ⁻³	1x10 ⁻²	1x10 ⁻⁴	<3x10 ⁻⁴	3x10 ⁻²	-	-	-	-	-	-	-	-	<2000	<4000	<1000	300	100	0.024	-	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<2000	<4000	<1000	300	100	11	1.3	
96	1x10 ⁻³	-	-	-	-	-	-	-	-	-	-	-	0.01	-	-	-	-	-	-	-	23	3.2	
-	2x10 ⁻²	-	-	-	-	-	-	-	-	-	-	-	55	44	-	-	-	-	-	-	5.8	0.78	
-	6x10 ⁻³	-	-	-	-	-	-	-	-	-	-	-	55	44	-	-	-	-	-	-	0.073	0.01	
-	-	-	-	-	-	-	-	-	-	-	-	-	99	-	-	-	-	-	-	-	0.025	0.004	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5.9	0.79	
-	5x10 ⁻³	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.4	0.16	
-	-	-	-	-	-	-	-	-	-	-	-	8	0.01	0.01	-	-	-	-	-	-	5.8	0.78	
-	-	-	-	-	-	-	-	-	-	-	-	-	56	43	-	-	-	-	-	-	2.8	0.31	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5.8	0.78	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	7.5	0.69	
-	-	-	-	-	-	-	-	-	-	-	-	-	55	44	-	-	-	-	-	-	5.8	0.78	
-	-	-	-	-	-	-	-	-	-	-	-	-	0.01	0.01	-	-	-	-	-	-	1.8	0.22	
-	-	-	-	-	-	-	-	-	-	-	-	-	55	44	-	-	-	-	-	-	5.8	0.78	

TABLE A. (Continued)

Stream No. (See Figure A)	Name of Stream	Distribution, Per Cent of Total in Monazite Sand						Composition of Stream, Per Cent										
		Th	U	P.E.	Ti	Fe	P as PO_4	Th	U	P.E.	Ti	Fe	P_{2O_5}		UO ₂	TBP	GSN	H ₂ O ₂
													PO ₄	P ₂ O ₅				
126	GH HNO ₃ recycle	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
137	1.5M HNO ₃	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Solvent wash	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Miscellaneous use	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
132	HNO ₃ waste	0.1	0.1	2.0	30	85	0.2	3x10 ⁻²	5x10 ⁻³	5	2	2	0.2	55	0.1	-	0.04	0.01
139	NaOH waste to neutralization	-	5x10 ⁻⁵	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
140	Neutralized waste slurry	0.1	0.1	2.0	30	85	0.2	2x10 ⁻²	3x10 ⁻³	3	1	1	0.1	-	-	0.65	-	-

(1) Does not refer to materials not found in monazite sand, i.e., H_2O , H_2O_2 , and HNO_3 . Figures for these are based on streams which show 100 per cent of particular material.

(2) These head compositions are based on total uranium found in process streams.

(3) Does not include filter aid.

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Distribution, Per Cent of Total in Ionazite Sand										Composition of Stream, Per Cent					Composition, Parts Per Million				Quantity Per Pound of Thorium Produced				
U		F.E.		Ti		Fe		P as PO_4		Th	U	P.E.	Ti	Fe	P	Th	U	F.E.	Ti	Fe	P	Pounds	Gallons
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	4.5	0.87
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.4	0.16
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	6.2	0.71
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	3.4	0.17
0.1	0.1	2.0	30	85	0.2	5×10^{-2}	5×10^{-2}	5	2	2	0.2	55	0.1	0.04	0.01	-	-	-	-	-	-	2.8	0.31
-	6×10^{-3}	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5.2	0.45