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Subject Category: CHEMISTRY—SEPARATION PROCESSES FOR PLUTONIUM AND URANIUM

UNITED STATES ATOMIC ENERGY COMMISSION

CHEMICAL PROCESSING OF TWO-REGION THERMAL BREEDER REACTORS

By
F. R. Bruce

Price \$ 0.35

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

March 22, 1956

Oak Ridge National Laboratory
Oak Ridge, Tennessee

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CHEMICAL PROCESSING OF TWO-REGION
THERMAL BREEDER REACTORS

By

F. R. Bruce

March 22, 1956

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DATE APR 17 1957

For The Atomic Energy Commission

H. F. Canale
Chief, Declassification Branch

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CHEMICAL PROCESSING OF TWO-REGION THERMAL BREEDER REACTORS

F. R. Bruce

INTRODUCTION

This discussion will be concerned with the chemical processes which have been developed for two-region thermal breeder reactors employing an enriched uranyl sulfate fuel and a thorium oxide slurry blanket.^{1,2} Table 1 shows the characteristics of the specific reactor which will be discussed. During reactor operation a number of events occur which make chemical processing necessary.

1. While U^{233} is being consumed for heat production in the core, a slightly greater quantity of Pa^{233} , which decays to U^{233} , is produced in the blanket. Most of this U^{233} must be removed from the blanket and returned to the core as fuel; the balance represents the breeding gain. At the same time, the thorium blanket material must be purified from corrosion and fission products which accumulate.

2. In the reactor core, fission products are produced, and through corrosion, zirconium, iron, chromium, nickel and manganese are introduced into the system. The fission products and stainless steel corrosion products have high cross sections and impair the neutron economy of the reactor. Therefore, they must be removed.

3. Many of the contaminants produced in the core are insoluble under reactor conditions. Among the fission products the rare earths, strontium, barium, zirconium and niobium fall in this category; while of the corrosion products, zirconium, iron, and chromium precipitate. The presence of these

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Table 1

Two-Region Thermal Breeder Reactor

Core System

Power	320 MW
Volume	23,000 liters
Average flux	3.2×10^{14}
U ²³³ concentration	1.24 g/liter

Blanket System

Power	144 MW
Volume	21,000 liters
Average flux	5.1×10^{13}
Th concentration	1,000 g/liter
U ²³³ concentration	5 g/liter
Pa ²³³ concentration	1 g/liter

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insolubles may complicate reactor operation, and it is desirable to purge them from the core.

4. Finally, the fission products constitute a health hazard in the event of a catastrophe, and there is incentive for removing them from the reactor system and isolating them in a form such that they cannot be released to the environment.

The objectives of thermal breeder reactor processing are then: first, to remove bred material from the blanket and to return it to the core; second, to maintain good neutron economy by removing fission and corrosion product poisons from the core and blanket; third, to improve reactor operability by removing insolubles; and fourth, to reduce the biological hazard associated with the reactor by continuously removing fission products. In the discussion which follows the principles of thermal breeder reactor processing will be presented, flowsheets for a three-reactor power station described, and the economics of chemical processing reviewed.

THERMAL BREEDER REACTOR PROCESSING PRINCIPLES

Core Processing

Poisoning^{3,4}

Neutron losses to extraneous materials in a reactor fuel or blanket are conveniently described by the term:

$$\% \text{ poison} = (100) \frac{\Sigma_p}{\Sigma_f}$$

where Σ_p = total macroscopic cross section of poison per unit volume (cm^2)

Σ_f = total macroscopic fission cross section of the U^{233} per unit volume (cm^2)

If a reactor operates for an extended period of time, the percent poison, which is a measure of neutron, and hence fissionable material, losses, increases as indicated in Figure 1, with the rare earths and the corrosion products contributing heavily.

Behavior of Fission and Corrosion Products under Reactor Conditions

The poisons encountered in a homogeneous reactor core may be conveniently divided into the following categories: the insolubles; the gases; and the solubles. Table 2 shows the solubility of those materials which have limited solubility. The rare earths, iron and chromium, all of which are important poisons exhibit very low solubility and may be removed from the reactor by a hydroclone.⁵

The rare gases, xenon and krypton, will be removed from the reactor with radiolytic deuterium and oxygen which is sent to the recombination system. If copper is employed as an internal catalyst for complete radiolytic gas recombination, an alternate method for removing Kr, Xe, and I must be used. A process for removing iodine, which is a significant poison and the leading radiological hazard in the reactor, is under development. Figure 2 summarizes the chemistry of iodine under reactor conditions. Design studies of a process for removing iodine from the fuel by stripping with oxygen at reactor temperature and pressure, followed by back-stripping into water indicate that it is attractive.

Soluble poisons--cesium, nickel, and manganese--will be removed from the core system in the hydroclone underflow receiver with the solids in a manner which has already been described.⁵

If the TBR core is processed on a daily cycle for removal of insolubles and on a 190-day cycle for removal of soluble poisons, a poison level of

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Fig. 1 Total Poison Effects in Core of Two Region Thermal Breeder.

Table 2

Elements of Limited Solubility in TBR Fuel

	<u>Solubility (mg/kg D₂O at 280°C)</u>
La ₂ (SO ₄) ₃ , Ce ₂ (SO ₄) ₃ , Pr ₂ (SO ₄) ₃ , Nd ₂ (SO ₄) ₃ , Y ₂ (SO ₄) ₃	85
SrSO ₄ , BaSO ₄	10
ZrO ₂ , Nb ₂ O ₅ , Fe(OH) ₃ , Cr(OH) ₃	5
Ru, Rh, Pd	5 ⁽¹⁾

(1) The chemical specie of the insoluble compound of this group is not known with certainty.

Table 3

TBR Core Poisons⁽¹⁾

	<u>% Poison</u>
<u>Limited Solubility</u>	
Nd	0.8
Ce	0.2
Sm, Eu	0.8
Ba, Sr	0.1
<u>Gases</u>	
Kr, Xe	0.5
I	0.1
<u>Solubles</u>	
Cs	1.3
Ni, Mn	0.6
Tc	<u>1.2</u>
<u>Total</u>	5.6

(1) Daily processing cycle through hydroclones and 190-day processing cycle through Thorex.

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$$\text{IO}_3^- \rightleftharpoons \text{I}^0 \rightleftharpoons \text{I}^0_{(g)}$$

In 0.02 m UO_2SO_4 , 0,005 m H_2SO_4

$$\frac{[I^{\circ}_{(l)}]}{[IO_3^{-}_{(l)}]} = 7$$

$$\frac{[I^{\circ}(g)]}{[I^{\circ}(l)]} = 7$$

In D_2O at pH 4 to 9

$$\frac{[I^{\circ}(g)]}{[I^{\circ}(l)]} = 0.2$$

Fig. 2

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5.6% is maintained. Table 3 summarizes TBR poisoning when such a processing scheme is employed. Achieving this poison level, without daily solids removal, by withdrawing an aliquot of fuel for purification would necessitate processing the core on a 55-day cycle.

Hazards Reduction⁶

Continuous processing of TBR fuel results in a substantial reduction in the radiological hazard associated with the system. In evaluating the hazard reduction, the term "relative hazard" may be used. The relative hazard for each radioisotope is arbitrarily defined as the exposure contributed by the isotope in a reactor accident divided by its permissible biological tolerance. Table 4 shows the relative hazards of several radioisotopes. It is first assumed that the reactor has operated for 80 days without processing before an accident, and secondly, that the reactor is processed on a one-day cycle for removal of fission products. Approximately one-third of the total relative hazard is attributable to iodine. With the exception of tellurium and molybdenum, the balance of the hazard is due to fission products which are insoluble under reactor conditions. If the reactor is processed on a one-day cycle, the total relative hazard may be reduced 18.8 fold.

Blanket Processing by Leaching of Mass 233 and Contaminants from Thorium Oxide

An ideal blanket processing scheme would consist of leaching either protactinium or uranium-233 together with fission and corrosion products from the thorium oxide. The thorium oxide could then be returned directly to the reactor. Limited success has been obtained in leaching U^{233} from thorium oxide by reacting the oxide with 8 M H_2SO_4 , to convert the uranium

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Table 4

Biological Hazards Associated with Reactors under Two Operating Conditions
and the Hazard Reduction Obtained by Continuous Removal of Fission Products

Isotope	Relative Hazard for 80-day Operation, $\times 10^{-4}$	Relative Hazard for 1-day Processing Cycle, $\times 10^{-4}$	Reduction in Hazard by Continuous Processing
I ¹³¹	375	32.5	11.5
Ba ¹⁴⁰ -La ¹⁴⁰	165 (110 to bone)	8.94 (6.0 to bone)	18.5
Sr ⁸⁹	145	3.01	48
Ce ¹⁴⁴ -Pr ¹⁴⁴	140	1.86	75
Y ⁹¹	85.5	1.68	51
Nb ⁹⁵	36.4	2.22	16.4
La ¹⁴⁰	33.0	1.79	18.5
Pr ¹⁴³	30.5	1.53	20
Te ¹²⁹	25.0	0.67	38
Sr ⁹⁰ -Y ⁹⁰	15.0	0.19	80
Ce ¹⁴¹	13.4	0.35	39
Mo ⁹⁹	10.3	2.2	4.7
Total	1,070.0	57.0	18.8

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to soluble sulfate which is then removed by water washing the solids. The procedure is not practical, however, since 10 liters of 8 M H_2SO_4 leach are required per kilogram of ThO_2 , and the sulfate contamination introduced into the thorium oxide is eliminated only with great difficulty.

THERMAL BREEDER REACTOR CHEMICAL PROCESSING FLOWSHEET

The principles which have been outlined may be incorporated into a flowsheet (Fig. 3) for chemical processing of a three-reactor power station. For the TBR under consideration the core will be processed daily through hydroclones for solids removal and on a 190-day cycle through a solvent extraction (Thorex) plant by withdrawing 120 liters daily from the underflow receiver. This volume will contain 149 g U^{233} and 10,300 curies of soluble and insoluble fission product poisons. After heavy water recovery by evaporation to 1 M UO_2SO_4 , the underflow receiver contents will be stored 50 days for decay of short-lived radioactivity before being blended with blanket material.

The blanket of the TBR will be processed on a 320-day cycle through the Thorex plant in order to maintain 3% poison. Sixty-seven kilograms of thorium, containing 338 g U^{233} and 67 g of Pa^{233} will be withdrawn daily in 67 liters of heavy water. This will be blended with the concentrated underflow receiver contents, which have previously been cooled for 50 days, and sent to the heavy water recovery step.

In this study it is assumed that core and blanket material from three 450 MW reactors would be processed in common chemical plant facilities, beginning with the D_2O recovery step, in equipment having a capacity of 200 kg of thorium per day.

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Fig. 3. TBR Chemical Processing Plant.

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D₂O Recovery

The thorium oxide slurry from the blanket will be combined with 50-day cooled underflow receiver contents from the core and added to the tray dryer where heavy water will be removed by heating to 300°C. Heavy water losses in the uranyl sulfate and thorium oxide will be less than 0.5% and 0.2% by weight, respectively.

ThO₂ Dissolution

The solids consisting of thorium oxide, and the sulfate and oxides of fission products, corrosion products and uranium will be slurried out of the evaporator with light water and sent to the dissolver.

Thorium oxide is readily soluble in nitric acid containing 0.04 M fluoride ion as catalyst. Dissolution rates increase with increasing excess of nitric acid, and decrease with increasing calcination temperature of thorium oxide. The results of typical dissolution experiments are summarized in Table 5.

Table 5

Dissolution of ThO₂ in Nitric Acid Containing 0.04 M HF

<u>HNO₃</u> Molarity % Excess		ThO ₂ Calcination Temperature (°C)	Dissolution Time (Minutes)
8	30	1000	225
		800	150
		650	60
13	115	1000	38
		800	29
		650	28

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Decay for Cooling^{7,8}

Decay of core and blanket material prior to solvent extraction is desirable for three reasons: (1) it permits less thick shielding in the chemical plant, since the controlling element, 40 h La^{140} , the daughter of 12.4 d Ba^{140} , has decayed appreciably; (2) radiation damage to the tributyl phosphate solvent is decreased; and (3) the thorium oxide preparation steps are more easily effected with cooled thorium which contains a lower Th^{234} and fission product background.

Although the solvent extraction plant processing is simplified by handling long-cooled material, inventory charges which accrue on uranium and thorium during cooling dictate that the shortest possible decay periods be employed. It now appears that radiation damage to tributyl phosphate and its diluent will determine the minimum cooling time. Table 6 summarizes, as a function of decay time, the activity levels expected in the underflow receiver contents and thorium oxide slurry, and the solvent exposure corresponding to each decay time.

Exposure of tributyl phosphate to radiation results in its degradation to mono- and dibutyl phosphate, while the diluent forms organic peroxides. Exposure of 42% TBP--58% Naptha No. 1 diluent to an irradiation of one watt-hour per liter results in a two-fold decrease in the separation of thorium and uranium from fission products in the solvent extraction step. Increasing the exposure to 6 watt-hours per liter of solvent does not result in a further decrease in decontamination. However a dose of 6 watt-hours per liter produces sufficient mono- and dibutyl phosphates to result in serious emulsion formation in the solvent extraction processing.

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Table 6

Contributions of Core and Blanket Material to Solvent Exposure in the
Thorex Process

Core processing period: daily through hydroclones; 190 days
through Thorex

Blanket processing period: 320 days

Solvent exposure: 10 min.

Cooling Time (days)	Exposure Contribution by: (Beta-watt hours/liter of Solvent)	
	Core Material	Blanket Material
5	3.66	0.82
10	2.63	0.72
20	1.67	0.58
30	1.17	0.55
40	0.89	0.36
50	0.70	0.29
60	0.56	0.23
70	0.49	0.18
80	0.40	0.14
90	0.34	0.11
100	0.29	0.09
110	0.25	0.07
120	0.22	0.06

Tributyl phosphate extraction processes have been successfully demonstrated at exposure levels of 0.5 watt-hour per liter of solvent. This study is therefore based on that dose. Cooling times of 110 days for the underflow receiver contents, and 60 days for blanket material are therefore required. It is, however, likely that a core and blanket cooling time as short as 5 days and entailing a solvent exposure of 3.4 watt-hours per liter would be feasible, although it remains to be experimentally verified.

Thorex Processing⁹

After cooling, the nitric acid solution of the fuel and blanket will be sent to the feed adjustment step where the solvent extraction process feed will be prepared (Fig. 4).

In the Thorex process solvent extraction column, uranium and thorium are co-extracted away from fission products and protactinium with 42.5% tributyl phosphate. In a second partitioning tower, thorium is preferentially stripped from the solvent into a 0.4 M nitric acid solution. The thorium nitrate product is then passed to thorium oxalate precipitation and calcination operations for refabrication of blanket material. The tributyl phosphate leaving the thorium partitioning tower passes to a third tower where uranium is stripped from the solvent with 0.008 N nitric acid.

Uranium-233 is adsorbed from the resulting solution by the cation exchange resin and subsequently eluted with sulfuric acid yielding a uranyl sulfate solution which may be returned to the reactor core as fuel.

A 200-kg-of-thorium-per-day pilot plant has demonstrated Thorex process operability over the past year. Thorium containing up to 3,000 g of U²³³ per ton has been processed successfully after six weeks' cooling.

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CONTAMINANT	DECONTAMINATION FACTOR	
	URANIUM PRODUCT	THORIUM PRODUCT
Gross γ	2×10^5	9×10^3
Pa	4×10^6	1×10^4
Ru	4×10^3	3×10^3
Nb - Zr	9×10^5	1×10^4
TRE	1×10^6	8×10^4

Fig. 4

URANIUM AND THORIUM PURIFICATION FROM
FISSION PRODUCTS IN THE THOREX PROCESS

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Recoveries of both uranium and thorium are approximately 99.9%. Table 7 shows typical separations of uranium and thorium from fission products which are obtained in the process. It may be concluded from these results that the Thorex process will yield uranium and thorium products of adequate purity for return to the reactor, although remote handling will be required.

The following photographs (Figs. 5-8) show typical views of the Thorex pilot plant. The Thorex pilot plant is housed in concrete-shielded cells, and is controlled by a central instrument panel. All indicating, recording and controlling instruments are located on this panel as well as switches for operating pumps, spargers and agitators in the cell and makeup areas.

A heavily shielded sampler gallery is required in the Thorex pilot plant for obtaining samples of highly radioactive solutions. Shown are three sample blisters, each of which shields and contains 10 sampling points. Sample bottles are loaded into one of three carts attached to a shielded conveyor chain; each cart is automatically positioned at its correct blister, reducing the probability of incorrect sampling. The carts are returned to the loading station in the same manner and the sample bottles are secured to shielded carriers for delivery to the analytical laboratory.

Table 7

Uranium and Thorium Purification from Fission Products in the Thorex Process

Contaminant	Decontamination Factor	
	Uranium Product	Thorium Product
Gross γ	2×10^5	9×10^3
Pa	4×10^6	1×10^4
Ru	4×10^3	3×10^3
Nb-Zr	9×10^5	1×10^4
TRE	1×10^6	8×10^4

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Fig. 5. Thorex Pilot Plant Building 3019.

Fig. 6. Thorex Instrument Panel.

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Fig. 7. Thorex Sampler Gallery.

Fig. 8. Thorex Sampler in Sampling Position.

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Pa²³³ Handling

If 60 days' cooling for blanket material is employed prior to solvent extraction, approximately 4% of the mass 233 will be present as Pa²³³. The protactinium will remain with fission products in the waste from the first solvent extraction tower. This waste will be evaporated to 2 M aluminum nitrate and stored to permit decay of protactinium to U²³³. The resulting U²³³ will be recovered by solvent extraction with 7.5% tributyl phosphate.

ThO₂ Blanket Refabrication¹¹

Thorium oxide which is suitable for blanket use has been prepared at the rate of 750 pounds per week in the Thorex pilot plant. Thorium oxalate is precipitated by the addition of 15-16% excess oxalic acid to a 0.9 M solution of thorium nitrate at 40°C. Figure 9 shows the precipitator employed for this step. The resulting slurry is passed to a batch vacuum filter (Fig. 10) which employs a cotton duck filter cloth, and the mother liquor filtered off. The thorium oxalate cake is washed three times with hot water, removed from the filter and placed in a Vitreosil calcination tray. The tray is loaded in a 60 kw calcination furnace (Fig. 11) which has a capacity of 160 pounds of thorium oxide. The furnace heat is gradually increased until the oxide is fired at a final temperature of 800°C.

U²³³O₂SO₄ Refabrication

A cation exchange, U²³³ isolation process is employed in the Thorex pilot plant (Fig. 12). It consists of two columns filled with Dowex-50 ion exchange resin at the right, the waste catch tank in the center, and

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Fig. 9. Precipitator.

Fig. 10. Vacuum Filter.

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Fig. 11. 60 kw Calcination Furnace.

Fig. 12. Cation Exchange, U^{233} Isolation Process.

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the uranium product catch tank at the left. The uranyl nitrate product from solvent extraction flows down through the small column which serves to pick up residual thorium and then passes down through the large column where the uranium is adsorbed. $U^{233}O_2SO_4$ core fuel will be prepared by eluting the large column with sulfuric acid.

THERMAL BREEDER REACTOR CHEMICAL PLANT CONCEPTUAL DESIGN¹

The flowsheet previously described was used as a basis for preparing a conceptual design of a three-reactor-station chemical plant. The arrangement of process cells, analytical laboratory, and operating building is shown in Fig. 13. This plant is designed to process fuel from a power station in which three large reactor cells are arranged in line and for which a tentative building arrangement has been proposed.¹ The three hydroclone cells are buttressed against the wall of the concrete building and the remainder of the process canyon is an extension of the hydroclone cells, perpendicular to the reactor building. Immediately adjacent to the hydroclone cells are three cells for: heavy water recovery, decay storage, and blanket makeup. These cells, together with the hydroclones, constitute that part of the chemical processing system involving heavy water solution and slurry handling, and are separated from the Thorex process canyon which is designed for underwater maintenance.

The Thorex canyon is divided into two main areas for hot and warm equipment. The hot cell contains dissolution, feed adjustment, acid recovery and Thorex "A" column equipment. The warm area contains the thorium partitioning column, the uranium strip column, and solvent recovery column. Both areas are operated dry and would be flooded for underwater maintenance.

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Fig. 13

TWO-REGION REACTOR STATION WITH PROPOSED CHEMICAL PLANT

PROCESS ECONOMICS

Annual operating costs for the chemical plant associated with a three-reactor station are shown in Table 8. The investment in hydroclone facilities for the three reactors was estimated to be \$191,050, and the annual operating cost for core processing equipment was estimated to be \$40,000.

The balance of the chemical processing plant including facilities for heavy water recovery, reactor feed makeup, a 0.2 ton-per-day Thorex plant, and radiochemical and isolation laboratories entail an investment of \$5,798,300. The annual operating cost of this part of the plant was estimated to be \$1,290,100.

The total annual operating charge for the chemical plant for processing a three-reactor station was estimated to be \$2,228,800, assuming amortization of the facilities at the rate of 15% per year. This annual charge does not include process losses and inventory charges which amount to \$360,000 and \$265,000 annually.

Figure 14 shows cost distribution of chemical process costs for the power station. Total fuel cost for the station is 2.19 mills/kwh. Slightly more than half of the fuel costs is attributable to inventory charges, most of which are incurred in the reactor system. The chemical processing cost, 1.06 mills/kwh, constitutes 48.2% of the total fuel costs with the blanket processing comprising the largest portion.

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TBR ANNUAL CHEMICAL PROCESSING COSTS⁽¹⁾

BASIS: THREE, TWO-REGION, 450 Mw REACTORS

CORE PROCESSING – TOTAL INVESTMENT – \$191,050

AMORTIZATION AT 15%	\$ 28,700
OPERATING COST	40,000

BLANKET PROCESSING – TOTAL INVESTMENT – \$5,798,300

AMORTIZATION AT 15%	870,000
OPERATING COST	1,290,100

TOTAL	\$2,228,800
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(1) OTHER COSTS ARE: LOSSES – \$360,000,
INVENTORY CHARGES – \$265,000

Table 8

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DISTRIBUTION OF TOTAL FUEL COST

TOTAL COST : 2.19 mills/kwh

INVENTORY CHARGE : 12%

FIXED CHARGE ON INVESTMENT: 15%

HEAVY WATER LOSSES: 5% (assumed)

DISTRIBUTION OF CHEMICAL PROCESSING COST (48.2% of Total Fuel Cost)

TOTAL COST : 1.06 mills/kwh

THOREX PLANT

PLANT SIZE: 200 kg/day-Thorium

FIXED INVESTMENT: \$ 5,798,300

ANNUAL OPERATING COST: \$ 1,290,000

Fig. 14

COST DISTRIBUTION OF 1350-MW REACTOR POWER STATION

BASIS: THREE TWO-REGION 450 Mw REACTORS

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