325
9/10/64

REPROCESSING OF COATED OMRE FUEL

MASTER

L. A. Decker and M. E. Jacobson

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION

**NATIONAL REACTOR TESTING STATION
US ATOMIC ENERGY COMMISSION**

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

PRICE \$.50

AVAILABLE FROM THE
OFFICE OF TECHNICAL SERVICES
U. S. DEPARTMENT OF COMMERCE
WASHINGTON 25, D. C.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

PRINTED IN USA

IDO-14641
AEC Research and Development Report
Chemical Separations Processes
for Plutonium and Uranium
TID-4500 (30th Ed.)
Issued: July 1964

REPROCESSING OF COATED OMRE FUEL

L. A. Decker

M. E. Jacobson

PHILLIPS
PETROLEUM
COMPANY

Atomic Energy Division

Contract AT(10-1)-205

Idaho Operations Office

U. S. ATOMIC ENERGY COMMISSION

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

REPROCESSING OF COATED OMRE FUEL

L. A. Decker

M. E. Jacobson

A B S T R A C T

As a result of a comprehensive review of earlier work and a laboratory-scale demonstration with irradiated fuel, it is believed that stainless steel clad OMRE fuel coated with degraded terphenyl can be processed satisfactorily without prior cleaning. Either electrolytic dissolution in nitric acid or chemical dissolution in sulfuric acid may be used. The fine particles of coating material present in the dissolver effluent from either process should present no problem in conventional solvent extraction.

REPROCESSING OF COATED OMRE FUEL

L. A. Decker

M. E. Jacobson

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT.	iii
I. SUMMARY	1
II. INTRODUCTION.	1
III. INITIAL EXPERIENCE WITH PROBLEM	2
1. Post-Irradiation Examination	2
2. OMRE Core I Sample Cleaning.	3
3. Plant Experience	3
IV. RECENT EXPERIMENTAL APPROACHES TO PROBLEM	3
1. OMRE Core II Sample Cleaning and Dissolution	3
2. Synthetic Coating Removal.	4
3. Removal of Coating by Burning.	6
4. Demonstration of Processing Without Prior Film Removal.	6
5. Characterization of Solids	9
V. CONCLUSIONS	11
VI. LITERATURE CITED.	12
Appendix A Pyrolytic Film Forming Apparatus	13
Appendix B Details of Treatment of Pyrolytic Coatings	14
Appendix C Procedure for Preparation of TBP Solution	16

LIST OF TABLES

<u>Table</u>	<u>Page</u>
I Description and Results of Cleaning of Pyrolytically Fouled Heat Exchanger Surfaces.	5
II Dissolution Rates for OMRE Fuel in 6.5M Sulfuric Acid . . .	8

LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
1 Coated OMRE fuel plate sample	4
2 Electrolytic dissolver.	7
3 Flowsheet used for process demonstration.	10
4 Pyrolytic film forming apparatus.	13

REPROCESSING OF COATED OMRE FUEL

L. A. Decker

M. E. Jacobson

I. SUMMARY

Several earlier investigations of the processing problems caused by the film of degraded terphenyl coolant on OMRE fuel elements had resulted in a tentative recommendation that the fuel be cleaned prior to reprocessing. Recent demonstrations performed in the Multicurie Cell at the Idaho Chemical Processing Plant (ICPP), however, permit reversing the earlier recommendation. Dissolution tests have been performed satisfactorily both chemically using 6.5M sulfuric acid and electrolytically with 8.0M nitric acid; both dissolution processes result in an effluent containing suspended fine particles of the coating material. Dissolver effluent from each process was successfully processed through a pulsed extraction column, and virtually all the particles appeared in the aqueous raffinate solution.

Based on these studies, it is believed that OMRE fuel can be processed without removal of the organic film coating.

II. INTRODUCTION

The fuel elements received at the ICPP from the Organic Moderated Reactor Experiment (OMRE) are coated with a hard film of organic material produced as a degradation product from the organic moderating fluid (terphenyl) under the influence of heat and radiation. The fuel from the OMRE has generally been listed as one requiring special pretreatment before chemical reprocessing to recover the unused uranium⁽¹⁾. This listing had been based on the hypothesis that the coating would impede dissolution, plug lines, and hinder extraction. A special pretreatment was given to the fuel from OMRE Core I before reprocessing; this consisted of immersion in boiling Turco 4502, a commercial basic permanganate cleaning preparation. While this treatment was intended to remove the carbonaceous degradation product which deposited on the fuel plates, the actual behavior of the film during reprocessing operations was uncertain⁽²⁾. Therefore, several investigations were made to determine the optimum method of dealing with the film coating for subsequent cores.

The OMRE fuel element consists of a Type 304 stainless steel box which holds 16 fuel plates having a core consisting of fully enriched uranium dioxide particles uniformly dispersed in a matrix of stainless steel. The core is 20 mils thick and is clad with five mils of stainless steel. The overall dimensions of each fuel plate are 0.03 inch by 2.8 inches by 37 inches⁽³⁾.

The nature of the hard film* on the fuel plates varied from thin (on the order of 0.1 mil) and tenacious to thick (on the order of one mil) and brittle. Early investigations showed that the film was resistant to all common laboratory reagents, although there were indications Turco 4502 would remove it.

After the start of construction of the Experimental Organic Cooled Reactor (EOCR), further attempts were made to remove and identify the coating which forms on the irradiated fuel plates. Synthetic coatings made in heat exchangers and a Linac irradiator were exposed to Turco 4502. Neither those films nor films from OMRE Core II fuel pieces were removed after several hours exposure to the boiling solution.

These indications that Turco 4502 does not remove all forms of the film cast doubt on the usefulness of the Turco pretreatment in processing the OMRE fuel. This created additional incentive to process the remaining core of OMRE fuel without attempting to remove the film. Demonstrations of effective procedures by which this could be accomplished were carried out in the course of the work reported herein.

III. INITIAL EXPERIENCE WITH PROBLEM

The OMRE was brought to criticality in September, 1957, and by June, 1958, sufficient operating time had been experienced to warrant removal of an element for examination. Since that time, several experimental programs have been conducted in an effort to characterize and remove the film coating from the OMRE fuel elements.

1. Post-Irradiation Examination

A post-irradiation examination⁽⁴⁾ of an OMRE fuel assembly by Battelle Memorial Institute was apparently the first effort to remove and characterize the film coating from the fuel element surfaces. One fuel element was disassembled and examined after it had received a uranium burnup of about two percent. It was found that the coating would flake off in some places from handling manipulations; however, most of the coated areas resisted flaking and the film was described as tenacious. Attempts were made to clean the film coating from the fuel plate surfaces by using such solvents as trichloroethylene, carbon

* As contrasted to the soft film of terphenyl which is on the fuel as it is withdrawn from the core. This soft film may be drained off or washed off with an organic solvent.

tetrachloride, acetone, benzene, trichlorobenzene, butyl alcohol, sodium hydroxide, 50-percent nitric acid, and ortho-xylene. None of these solvents were successful in removing or loosening the film.

Attempts were also made to get the film coating to crack or chip away by bending the metal, buffing with a fiber brush, and immersing the plate in liquid nitrogen. The only successful method evolved for removing the film was buffing the plates with a stainless steel wire wheel.

Optical examination of the cut fuel plate showed that the average thickness of the coating on that fuel element was 0.25 mil.

2. OMRE Core I Sample Cleaning

Before processing Core I from OMRE, a search was made for a chemical reagent that would remove the film from the fuel⁽⁵⁾. Samples of end box material and fueled portions of a fully irradiated plate were treated with Turco 4502 and other reagents. Only hot Turco 4502 was found to remove the film. Reagents that were tried with no success were 6M sulfuric acid, 1M hydrogen peroxide with and without sulfuric acid, and kerosene.

3. Plant Experience

The fuel elements from Core I of OMRE were processed at the ICPP⁽⁶⁾. The elements were cleaned with Turco 4502 and dissolved one at a time in the dissolver designed to process the Submarine Intermediate Reactor (SIR) fuel. Sulfuric acid was used to dissolve the stainless steel, after which nitric acid was added to dissolve the uranium dioxide. The uranium was then extracted with 10 percent TBP in kerosene.

A metallic residue containing silicon and iron as major constituents remained in the dissolver vessel after dissolution in the sulfuric acid. Later experiments indicated that the crystalline material was silica and that iron sulfate was also present. This residue would be present even if the fuel were not coated with an organic film. The solids present in the dissolver effluent initially caused plugging of the sample and liquid transfer lines during reprocessing. Following plant modifications which permitted agitation of the dissolver solution by sparging during dissolution, no further plugging was encountered.

IV. RECENT EXPERIMENTAL APPROACHES TO PROBLEM

1. OMRE Core II Sample Cleaning and Dissolution

Further studies⁽⁷⁾ of coating removal by aqueous solutions of Turco 4502 were carried out using samples cut from a fuel plate from one of the fuel elements of Core II of the OMRE. After removal from the

reactor, these samples were stored first in terphenyl coolant, then in a dry cask.

Samples with three different coating thicknesses were used, light (~ 0.1 mil), medium (~ 1 mil), and heavy (> 1 mil). Figure 1 shows one of the medium coated samples used in this study. One sample of each was heated at 80°C for two hours in Turco 4502 solution (two pounds per gallon). The lightly coated sample was apparently cleaned but the medium and heavily coated samples were not. Another Turco product, Smut-Go #2, had no apparent effect on a medium coated sample.

The coating was shown to contain iron and was identified by X-ray diffraction as iron-carbide, Fe_{20}C_9 . No iron was found in the cleaning solution after filtering; therefore, little of the coating had dissolved.

Fig. 1 Coated OMRE fuel plate sample. Coating thickness is approximately one mil

Attempts were made to show that certain dissolution systems might adequately penetrate the coating and propagate dissolution of the fuel. A few preliminary experiments were performed on some of the OMRE Core II fuel samples to determine the effect of the coating on the dissolution and the effect of the resulting solids in the solvent extraction system. These are described in Sections 4.1 and 4.2.

2. Synthetic Coating Removal

Synthetic carbonaceous coating residues were formed on heated surfaces of tubes or wires exposed to used OMRE coolant at three separate laboratories: Phillips Petroleum Company at Bartlesville, Oklahoma; California Research Corporation, San Francisco, California; and Atomics International, Canoga Park, California. After heating, the tubes were split lengthwise to expose the coating on the inside surface. A pyrolytic film forming apparatus similar to that used by Phillips Petroleum Company at Bartlesville, is shown in Appendix A.

Attempts were made to remove the coatings from these heat exchanger surfaces with Turco 4502 and by heating to 500°C. Table I gives a summary of the results of these efforts. In all cases the lightly coated specimens became clean after 2-3 hours in boiling Turco 4502 solution. The medium and heavily coated specimens were still visibly fouled after six hours of boiling in the same solution. These

Table I

DESCRIPTION AND RESULTS OF CLEANING OF PYROLYTICALLY FOULED HEAT EXCHANGER SURFACES

Sample Description			Fouling Conditions			Cleaning Results	
Type ^(a)	Specimens	Description	Surface Temperature °F	Time of Exposure Hr	Fouling Coat	Turco 4502	Followed by 500°C in Air
1	11	Split tubes 1/2-in. diam x 8 in.	900-1050	6-8	light	Clean in 2-3 hr.	-
2	5	Wires 0.1-in. diam x 1 in.	1050	10-67	heavy	Still dirty after 6 hr.	Clean in 2 hr.
3	15	Split tubes 1/2-in. diam x 1 in.	825-1011	173-3760	heavy	Still dirty after 6 hr.	Clean in 2 hr.

(a) Type 1 samples from Phillips Petroleum Company

Type 2 samples from California Research Corporation

Type 3 samples from Atomic International

results together with those of heating the coated specimens, are shown in detail in Appendix B.

3. Removal of Coating by Heating

An alternate, non-aqueous method of removing the coating consisted of heating the fuel plate to 500°C for two hours which converted the black coating to a light brown powder which easily wiped off and was soluble in sulfuric acid. Air, passing through the furnace at about 2.4 cc/min, became contaminated with about 0.8 cpm/cc by carrying with it some of the particulate material from the coating. Heating the synthetically coated specimens at 260°C had no effect on any of the coatings but heating to 500°C oxidized all but some of the thickest portions of coating.

Since no plant facilities are available for heating irradiated fuel or for filtering the air from such a furnace, this method of cleaning would involve considerable capital expense and was not considered further.

4. Demonstration of Processing Without Prior Film Removal

Fuel samples with the coating intact were used in experiments to gain information on the behavior of the suspended coating particles during dissolution, solution transfer, and solvent extraction. Some of the samples of fuel that were dissolved had been stored only in protective, non-aqueous surroundings; the rest of the fuel used in these studies, including those used to demonstrate solvent extraction, had been stored in water at the ICPP basin water under normal fuel handling conditions.

4.1 Sulfuric Acid Dissolution: In early exploratory experiments the stainless steel in a medium coated sample was dissolved in 6.5M sulfuric acid followed by dissolution of the uranium dioxide by adding nitric acid to the solution. The dissolver product solution was adjusted to about 3.2M nitrate, 1.3M sulfate, 4.8M acid, 20.4 g/l stainless steel, and 2.0 g/l uranium. The coalescence time of this solution was about 65 seconds, suggesting that no emulsion problem would be created due to the solids.

During the process demonstration the dissolution rate for the OMRE fuel in sulfuric acid was determined by dissolving a piece of the fuel in 6.5M acid and analyzing periodic samples for iron to determine the rate of dissolution. One of the fuel pieces which had been cleaned by heating was dissolved and the rate of dissolution was compared with that from the dissolution of a coated, untreated fuel piece. Each dissolution proceeded rapidly until the acid concentration became depleted, even when only half enough acid for complete dissolution was present initially (corresponding to plant "two cut" dissolution)⁽⁶⁾. (See Table II.) Dissolution was initiated on the coated surface as well as on the cut surfaces. There was no discernable difference in the

processing rate for the treated or the untreated fuel samples. The decrease in iron concentration sometime after 75 minutes indicated that there was precipitation of excess iron sulfate in the concentrated solution.

Results of the determination of coalescence time by the standard method⁽⁸⁾ were normal in each case, no films nor threads formed in the dispersion, and the organic phase was clear after coalescence. These results indicate that there would be no abnormal extraction column operation with feed from fuel dissolved with the coating intact.

4.2 Electrolytic Dissolution: In preliminary studies a heavily coated sample was dissolved electrolytically in 8M nitric acid at four volts direct current and a current density of 1 amp/cm². The dissolver used in this and in subsequent electrolytic dissolutions is shown in Figure 2. Anodic electrical contact was made with the fuel piece by clamping it in a niobium clamp. The stainless steel cathode was cooled by passing water through the tubing which was used for the electrical lead.

The product solution which resulted from the dissolution had an acid content of 6.3M and contained a finely divided (~ 0.1 mm) residue from the coating. The solution was contacted with 10 percent TBP extractant in an emulsion tester⁽⁸⁾. The coalescence time was about 40 seconds which indicated that excessive emulsification would not occur during column operation.

In further work, one of the cleaned samples and one with the coating intact were dissolved with a current density of about 1.0 amp/cm² and a potential of seven volts. The coating did

CPP-S-2897

Fig. 2 Electrolytic dissolver

Table II

DISSOLUTION RATES FOR OMRE FUEL IN 6.5M SULFURIC ACID

Fuel Description	Sample Number	Time After Acid Addition min	Time to Initiation of Reaction min	Iron Concentration g/l	Approximate Initial Dissolution Rate mg/cm ² min
Cleaned by burning	1	22	2	19.2	3.6
	2	44		56.7	
	3	75		75.2	
	4	174		63.1	
Coating intact	1	22	10	16.3	3.6
	2	44		49.2	
	3	75		67.0	
	4	200		56.5	
Coating intact(a)	1	37	27	34.7	4.4
	2	55		64.5	
	3	75		62.6	
	4	95		58.7	
	5(b)				

(a) One half the volume of acid used as for other two dissolutions.

(b) Fifth sample taken after making solution about three molar nitrate and allowing to stand overnight: sample had 36.4 g/l iron which would correspond to about 109 g/l iron in the undiluted sulfuric acid dissolver product solution.

not interfere with establishing initial electrical contacts. The dissolution rate for each sample proceeded at approximately the theoretical rate as determined by the ampere-hours used in the dissolution. There was no discernable difference between the behavior of the coated sample and the uncoated sample.

A process demonstration dissolution was carried out by dissolving 500 grams of the fuel samples electrolytically in 8M nitric acid in 19 individual batch operations. The dissolver which was used for these dissolutions is shown diagrammatically in Figure 2.

If desired, the coating could be removed electrolytically before dissolution, since it was observed that, as the dissolution initiated, the film coating flaked off. Immersion in 2M nitric acid at one amp/cm² for 2.3 minutes almost completely removed the film from the fuel surface. The solution had 0.55 mg/l uranium and 2.75 g/l iron after a total of 5.8 minutes at 100 amps after which the fuel surfaces were almost entirely clean. The uranium almost certainly came from the cut edges. In an intact fuel plate there would be no uranium exposed to the solution during the short period of time necessary to clean off the film. In a similar experiment with 0.2M nitric acid the specimen was clean and shiny after 12.5 minutes immersion. During that time the current density decreased from 0.4 amp/cm² at 10 volts to 0.1 amp/cm² at five volts. The resulting 700 ml of solution had 0.22 mg/l uranium and 0.32 g/l iron.

4.3 Extraction Demonstration: The solution resulting from the sulfuric acid dissolution was contacted with a 10 percent TBP in kerosene solution (after adjusting to about 3M nitric acid and a volume of four liters) in a four foot tall, one-inch diameter pulsed extraction column. Figure 3 shows a schematic diagram of the system used, Preparation of the TBP solution is described in Appendix C.

The solutions resulting from the electrolytic dissolutions were run through the pulsed extraction column in three four-liter batches after adjusting to a concentration of about 75 g/l stainless steel. A cumulative operating time of 110 minutes was logged during processing of the 12 liters of solution.

The extraction column operated satisfactorily with no plugging; dispersion and coalescence in the column were normal, and the transfer lines showed no tendency to plug. The 1/8-inch holes in the plates of the column remained clean and free from plugging. The residue from the film on the fuel plates appeared in the aqueous raffinate stream, while no part of that residue appeared in the organic product.

5. Characterization of Solids

In addition to the above evidence that the solids would readily pass into raffinate in a pulsed column, it was desirable to establish the nature of the contamination on the solids, the rate of settling in stagnant solution, and the properties pertinent to the behavior of the material in process vessels.

Fig. 3 Flowsheet used for process demonstration

CPP-S-2896

Analysis by emission spectroscopy and by X-ray diffraction showed that the solids that were collected on the filter papers were mostly inorganic materials, consisting mostly of silicon and iron. Minor constituents present in the solids were chromium, nickel, manganese, and zirconium.

The coating particles followed the aqueous stream through the pulsed column in the demonstration. The density of the agglomerated particles was determined to be about 1.8 by buoyancy measurements. A simple sedimentation test indicated that the bulk of the solids would remain suspended in the sulfuric acid dissolver solution for 1.5 hours.

Turco 4502 dissolved most of a portion of the particles from the coating that had passed through the extraction column after boiling for two hours. This indicates that, even though Turco 4502 will not completely remove the intact film from the fuel plate surfaces, it will successfully decontaminate equipment which has come in contact with the film coating particles.

A gamma scan made on the coating material after it had passed through the column showed that the total gamma activity came from the following isotopes: Ce^{144} -4.6 percent, Cs^{137} -9.3 percent, Sb^{125} -15.5 percent, and Ru^{106} -70.6 percent. Gamma scans of the particulate matter from the Turco treatments of the coated fuel showed the activity to be about 60 percent Zr^{95} - Nb^{95} , and 40 percent Ce^{144} . The Turco treatment, though unable to remove the coating from the fuel, was able to leach the zirconium-niobium and most of the cerium activities from the coating. The ruthenium activity was apparently very tightly adsorbed by the coating.

V. CONCLUSIONS

The coating on the OMRE fuel elements should no longer be considered to be a serious hindrance to successful chemical reprocessing. Recent studies indicate that this fuel can be processed with the coating intact either by electrolytic dissolution or by sulfuric acid-nitric acid two-step dissolution followed by conventional solvent extraction.

The dissolution rate was not impeded in either process by the presence of the film. Scratches on the surfaces of the fuel element from handling allow contact of the dissolving reagents with the metal surface through the coating. The coating is undercut rapidly enough that the overall dissolution rate is not diminished. The coating breaks up into small enough particles that it can be transferred through liquid lines without plugging. Although Turco 4502 is not effective in all cases in removing the film from the fuel element surfaces, it is apparently effective in removing particles of coating from other surfaces on which they have redeposited. Decontamination of equipment can therefore be accomplished with Turco 4502.

VI. LITERATURE CITED

1. Culler, F. L., Jr., et al, "Proposed Aqueous Processes for Power Reactor Fuels", Nucleonics, 20, No. 8, p. 124-127, (1962).
2. Ayers, A. L. and G. J. Raab, Reprocessing Uranium from the First OMRE Core, IDO-14552, p. 5, (1961).
3. Armenoff, C. T. and M. H. Binstock, Fuel Elements for the Organic Moderated Reactor Experiment, NAA-SR-1934, (1957).
4. Burian, Richard J. and John E. Gates, Postirradiation Examination and Evaluation of an OMRE Fuel Assembly, BMI-1319, (1959).
5. Stevenson, C. E., Technical Progress Report for April through June, 1959, Idaho Chemical Processing Plant, IDO-14494, (1960).
6. Ayers, A. L. and G. J. Raab, Reprocessing Uranium from the First OMRE Core, IDO-14552, (1961).
7. Bower, J. R., Editor, Chemical Processing Technology Quarterly Progress Report, July-September, 1961, IDO-14574, (1962).
8. Parrett, O. W., The Determination of Excessive Emulsification by Coalescence Behavior Measurements, IDO-14486, (1954).

APPENDIX A

PYROLYTIC FILM FORMING APPARATUS

A pressure vessel for preparing synthetic films by heating terphenyl and a metal surface in contact with the terphenyl is shown in Figure 4. It was designed for operation at 1000°C and 1000 psig. In operation, the vessel is filled about three quarters full of the terphenyl being tested and then closed. The coating forms on the hair pin shaped resistance heater after about 60 hours at 500°C heater surface temperature and about 425°C bulk terphenyl temperature.

This is similar to the apparatus used at Bartlesville for the formation of pyrolytic film on heat exchanger surfaces as described in "Organic Coolant Program Quarterly", IDO-16713; however, increased utility is obtained by use of the union joint which allows faster assembly and disassembly.

Fig. 4 Pyrolytic film forming apparatus

APPENDIX B

DETAILS OF TREATMENT OF PYROLYTIC COATINGS

The samples of pyrolytic coatings were subjected to severe conditions in attempts to remove the coating and expose the bare metal. The following table gives the conditions and the results of exposure to those conditions.

	Sample	Coolant ^(a)	Sample Wall Temp., °F	Hours Exposure	Appearance	Treatment Turco 4502 Hrs.	Weight Loss, mg	Treatment 500°C Hrs.	Total ^(b) Weight Loss mg
PPCo.	CF 4	Core II	900	13	Heavy	3			7
	CF 5	Core II	900	13	Heavy	3		2	335
	CF 6	OMP Terphenyl	1050	1.5	Heavy	3			46
	CF 7	OMP Terphenyl	900	12	Medium	2			32
	CF 9	Core II	900	4.5	Light	2 clean			68
	CF 14	Core II	930	6	Very Light	2 clean			39
	CF 17	Core II + High Boiler	930	6	Light	3 clean			37
	CF 18	Core II + 482 EPM Ferricene	930	6	Heavy	3			159
	CF 19	Core II	944	6	Light	3 clear.			39
	CF 20	Core II	940	6	Light	3 clear.			26
	Run 6 Electron Loop	OMP Terphenyl	900	18	Light	3 clear.			> .0
AI	2-4	Core I 30% HB	825	244	Dull	2		2	43
	2-6	Core I 30% HB	825	244	Dull	2		2	53
	2-12	Core I 30% HB	850	244	Dull	2		2	53

APPENDIX B (Continued)

Sample	Coolant (a)	Sample Wall Temp., °F	Hours Exposure	Appearance	Treatment Turco 4502 Hrs.	Weight Loss, mg	Treatment 500°C. Hrs.	Total (b) Weight Loss mg
2-13	Core I 30% HB	850	244	Dull	2		2	45
3-5	Core I 30% HB	900	304	Very Heavy	5	29	2	49
3-6	Core I 30% HB	900	304	Dull	3	32	2	53
3-11	Core I 30% HB	925	304	Shiny	5	25	3	143
3-13	Core I 30% HB	925	304	Shiny	5	34	2	82
3-14	Core I	925	304	Dull	3	28	2	77
5-5	Core I 30% HB	987	173	Shiny	3	27	2	171
6-5	Core I 30% HB	960	165	Dull	2	33	2	153
6-7	Core I 27% HB 16% Diphenyl	827	3760	Dull	2	27	2	101
6-12	Core I 27% HB 16% Diphenyl	874	3760	Dull	2	21	2	118
6-13	Core I 30% HB	1011	165	Dull	2	25	2	235
58-6	-	-	-	Shiny	3	52	2	195
CRC								
1		1050	10 $\frac{1}{2}$	Heavy	6 $\frac{1}{2}$	28	2	128
2		1050	66 $\frac{5}{6}$	Heavy	6 $\frac{1}{2}$	115		
3		1050	24 $\frac{1}{2}$	Heavy	6 $\frac{1}{2}$	163		
4		1050	5 $\frac{5}{6}$	Heavy	6 $\frac{1}{2}$	24		
5		1100	22	Heavy	6 $\frac{1}{2}$	97	2	175

NOTES

- a) Coolants labeled Core I, Core II were from OMRE reactor.
High Boiler (HB) is residue from high temperature vacuum distillation of coolant.
- b) All pieces were clean after 500°C treatment.

APPENDIX C

PROCEDURE FOR PREPARATION OF TBP SOLUTION

The tributyl phosphate (TBP) that was used in the extraction demonstrations was purified before use by washing three times:

First wash - 0.5M sodium carbonate

Second wash - 0.01M nitric acid

Third wash - distilled water

The kerosene used was purified by passing through silica gel beds. This procedure removed a redish brown impurity. The TBP solution was then made by adding 10 percent TBP by volume to kerosene.

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION