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RECOVERY OF URANIUM FROM CONTAMINATED PRECIPITON OIL

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ABSTRACT

The distillation and residue extraction recovery procedures developed for the recovery of uranium from Kinney and diffusion pump oil, were applied to contaminated precipitron oil. The effectiveness and advisability of using the method, in whole or in part, is discussed, and various recommendations for processing this particular type of oil are presented. Additional contaminating substances present in this type oil added to the complexity of the problem, but a satisfactory recovery procedure was, nevertheless, successfully developed.

INTRODUCTION

A quantity of precipitron oil, contaminated with uranium, was subjected to the uranium recovery procedures of vacuum distillation followed by nitric acid extraction of the distillation residue dissolved in trichlorethylene.¹ The purpose of the test was to determine whether or not the process, previously applied successfully to recovery of uranium from Kinney and diffusion pump oils, would be equally effective when applied to this particular type of oil.

Because of other contaminating substances found to be present in this oil, considerable difficulty was experienced with both the distillation and extraction phases of the process. The oil required

an excessively long outgassing period prior to high vacuum distillation and sublimable solids were vaporized during the period that oil was being distilled. Severe emulsification occurred during extraction of the distillation residua when trichlorethylene was used as residua diluent, a condition which had not been previously encountered when extracting the distillation residua of Kinney and diffusion pump oils. Alteration or probably an elimination of the distillation procedure became apparent and a variety of residua or oil diluents were tested as possible substitutes for trichlorethylene.

EXPERIMENTAL

Vacuum Distillation

The distillation equipment consisted of the usual arrangement of boiler, fractionating column, condenser and receiver, plus the necessary pumps, cold trap and instrumentation (see Figure 1). The still was fabricated of 1-inch pyrex pipe. The boiler was a 2-liter flask heated by a Glascol mantle. The fractionating column was simply a 2-foot length of unpacked, 1-inch pyrex pipe, heated by an externally-wound nichrome ribbon, element. Vacuum was produced by a 1-1/2 hp. Cenco "Hypervac 20" forepump serving a MC-200 oil diffusion pump. The pressure attained with this system was less than one micron (McLeod gauge).

A 1-liter sample was placed in the distillation flask and the heating and evacuation started. It was at this stage that the

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DISTILLATION APPARATUS

A DISTILLATION FLASK
B REFLUX COLUMN
C THERMOWELL

D CONDENSER
E VALVE TO VACUUM PUMP
F RECEIVER

FIG. 1

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first difficulty became apparent. The quantity of dissolved gas, water and other substances present in the oil, believed to be mostly ammonium salts, was so great that it was possible to complete the pumpdown only after four hours of carefully regulated operations. Normally, this would have required only 15 to 30 minutes. Distillation was eventually accomplished, however, and 90 percent of the material was distilled without further difficulty. A deposit of what was apparently the ammonium salts mentioned above, sublimed on the cold sections of the system in a thin film which was partially washed off by the distillate and carried down into the receiver.

The pumpdown difficulties encountered with this distillation could not be tolerated for processing any extensive quantity of oil. This trouble could probably be overcome, or at least substantially reduced, by the incorporation of some type of jet injection which would facilitate the rapid outgassing of an oil change. Although sufficient additional material was not available for further tests, oil of another type and in similar condition was tried successfully in such an injection unit.² There is no apparent reason why such a unit could not be employed if it should become necessary to distill appreciable quantities of precipitron oil.

Extraction of Distillation Residue

The residue from the distillation of one liter of precipitron oil was diluted with five parts of trichlorethylene and extracted by washing with 5 N nitric acid.¹ Troublesome emulsions formed during the extraction, probably caused by a variety of foreign contaminating materials originally present in the used oil, but the extraction was

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eventually completed after prolonged treatment. Analysis of the extract solution indicated that 4.8 mg. of uranium was recovered, or 4.8 micrograms per gram of oil when calculated as the concentration of uranium in the original oil.

Extraction of Original Oil

In view of the difficulties encountered with both the distillation of the oil and the emulsions formed during the extraction of the distillation residua, it was decided to attempt a direct extraction of the oil without first distilling off the bulk of the oil. Accordingly, three 10-gram samples of precipitron oil were diluted with five parts of trichlorethylene and extracted with 3 N nitric acid as had been done with the distillation residua. In these cases, formation of emulsions occurred to some extent but the extractions were eventually completed successfully. The results of these tests are as follows:

TABLE I
URANIUM EXTRACTED

<u>Sample</u>	<u>Milligrams Total</u>	<u>Micrograms per Gram Oil</u>
A	0.062	6.2
B	0.054	5.4
C	0.051	5.1

Several other solvents were tested as oil diluents in order to determine whether or not the tendency to form emulsions during extraction of the oil, could be eliminated by using some diluent other than trichlorethylene. The solvents tested were, diethyl ether,

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dibutoxy tetra ethyl glycol (penta-ether), dibutyl carbitol, carbon tetrachloride, and chloroform. The degree of emulsification exhibited by the various solvents differed widely. The observations of each test were as follows:

TABLE II
EMULSION TESTS

<u>Diluent Solvent</u>	<u>Discussion of Results</u>
Diethyl ether	Very little emulsion
dibutoxy tetra ethyl glycol (penta-ether)	Very little emulsion
Dibutyl carbitol	Fair - probably suitable for general use
Carbon tetrachloride	Highly emulsified
Chloroform	Highly emulsified
Trichlorethylene	Highly emulsified

Since diethyl ether, as a diluent, showed the least tendency to form emulsions, an extraction test was made with a portion of the original sample of precipitron oil to determine how effective the extraction with this diluent would be as compared to similar extractions with trichlorethylene. Three 10-gram samples of oil were diluted with 50 ml. of diethyl ether and washed three times with 2.5 N nitric acid. Following this, the aqueous phases were analyzed separately for uranium. The results of these tests are shown in Table III.

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TABLE IIIURANIUM RECOVERED BY NITRIC ACID EXTRACTION

<u>Pass Number</u>	<u>micrograms</u>		
	<u>Sample A</u>	<u>Sample B</u>	<u>Sample C</u>
1	32.0	43.0	38.0
2	4.6	7.2	6.0
3	2.2	2.7	2.9
Total	28.8	52.9	46.9

Although these results are not wholly in agreement with those found with the similar samples of oil when diluted with trichlorethylene, it is reasonable to believe that successful extraction can be accomplished with ether as the diluent.

SUMMARY

Recovery of uranium from contaminated precipitron oil may be accomplished by application of the methods described herein. The fact that used precipitron oil contains other substances, resulting from the particular use to which this type of oil is put, makes it difficult to employ the uranium recovery procedures originally developed for Kinney and diffusion pump oils. Vacuum distillation could be eliminated, if economically expedient, and uranium extracted directly from the oil dissolved in diethyl ether. The use of ether represents another departure from the usual practice in which trichlorethylene is used as the oil diluent. The substitution is apparently necessary to avoid formation of emulsions during the extraction.

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The number of tests possible in this investigation was limited considerably by the comparatively small amount of material available for the tests. Satisfactory recovery of uranium from precipitron oil is apparently possible by dissolving the oil in five parts of diethyl ether and washing this solution with 2.5 - 3.0 N nitric acid. The uranium dissolves completely into the acid solution from which it may be recovered by conventional means.

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