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TOPICAL REPORT

Report No. BMI-270

Technology — Feed Materials,

on

DETERMINATION OF RARE EARTHS IN
REFINED URANIUM

to

U. S. ATOMIC ENERGY COMMISSION

by

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For The Atomic Energy Commission

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ABSTRACT

Rare-earth analyses were performed on several samples of refined uranium oxide and nitrate, using a modified cellulose column procedure. Rare earths were not detectable in samples of 0.03 shotgun or less. Detection limits were from 0.0005 to 0.015 ppm. Negligible quantities of low-cross-section rare earths were found in less pure uranium samples, obtained during start-up operations of the pilot plant at the Feed Materials Production Center, primarily those rare earths of greater than average abundance in nature. No correlation was found between rare-earth content and shotgun values. Rare earths do not appear to be contributing significantly to the neutron absorption of refined uranium.

A modified cellulose column procedure, which includes a preconcentration solvent-extraction step, is described. Radioactive tracer tests indicating rare-earth recoveries by the method of > 98 per cent are reported.

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INTRODUCTION

One of the essential properties of refined uranium is a minimum content of elements possessing a high neutron-absorption cross section. The five elements with the greatest cross sections are gadolinium, boron, samarium, europium, and cadmium. Two of these, boron and cadmium, can be determined by direct spectrographic analysis, even in a uranium matrix. Gadolinium, samarium and europium, like the other rare earths, have weak lines, and are difficult to determine by direct spectrographic methods, especially at the extremely low levels tolerable in refined uranium.

Acceptance specifications and refinery-plant control are based on the so-called "shotgun" test, an empirical determination which provides an over-all value for neutron absorption. The test involves reducing 10 kilograms of uranium to a button of about 10 g in weight, by repeated solvent extractions. It is a rather costly, laborious, and time-consuming procedure

Published data on the sources of high-neutron-absorption values in refined uranium are scant, particularly with respect to the rare earths. Boron is known to be one of the worst offenders. The more commonly found elements need to be present in macro quantities to have a serious effect on neutron absorption.

In view of the exceptionally high cross sections of several of the rare earths, notably gadolinium, an attempt was made to correlate rare-earth contents and shotgun values. Another objective was to determine rare-earth contents of samples of typical refined uranium products. The cellulose-column separation method, devised by the British,* had earlier been developed and modified at Battelle for the determination of rare earths in thorium.** This method appeared to provide the simplest and most reliable means of determining rare earths in uranium, at the 0.001 to 0.01 ppm level, although it appeared also that further development would be needed to attain this sensitivity.

If a correlation did exist, a simpler and more rapid method of plant control, primarily spectrographic, appeared to be possible, which would eliminate the need for the cumbersome shotgun test. The Feed Materials Production Center, now under construction at Fernald, Ohio, was interested in such a possibility and the study was made as a part of the work carried on for the National Lead Company of Ohio under AEC contract AT-(30-1)-Gen-228. Mallinckrodt Chemical Works also expressed interest in this

*Kember, CRL-AE-23 and Arden, CRL-AE-36

**BMI-260, Center, et al.

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study, having the same problem of refinery control, and supplied a number of the uranium samples with known shotgun values.

SUMMARY

The rare-earth content of typical production samples of refined virgin uranium has been found in the present study to be remarkably low, beyond limits of detection. Even poorly refined uranium, with high shotgun values, has been found to have rare-earth contents at undetectable or insignificant levels. The rare earths found most frequently were those of normally greatest abundance; cerium, lanthanum, and neodymium. Yttrium was also detected in a number of samples.

Rare earths do not appear to be contributing significantly to the shotgun values of refined uranium. No correlation appears, therefore, to exist between rare-earth contents and shotgun values; the source of high shotgun values must lie elsewhere.

A combination solvent-extraction cellulose-column method has been developed which permits spectrographic assay of refined uranium for rare earths, with detection limits varying from <0.0005 to <0.015 ppm, depending on the element. The method has been checked by radioactive tracer techniques, which have indicated that recoveries of rare earths in the final sample of 98 per cent or over should be readily realized. Loss of rare earths to the solvent in the solvent-extraction preconcentration step was negligible, of the order of 0.5 per cent. Losses to the cellulose and to the ether used to desorb the uranium were also negligible. The greatest losses appeared to arise from normal manipulations and absorption on glassware, etc.

EXPERIMENTAL DETAILS AND DISCUSSION OF RESULTS

Rare earths were determined in samples of refined uranium, initially using the cellulose-column procedure, substantially as described in BMI-260.* The maximum quantity of uranium conveniently handled by this procedure is about 20 to 25 grams. The results, described in detail later in the report, showed that increased sensitivity was desirable, particularly for the high-cross-section element, gadolinium.

* BMI-260, "The Determination of Rare Earths in Thorium".

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Preconcentration by Solvent Extraction

The results of the initial tests indicated that improved sensitivity could be most readily attained by increasing the size of the starting sample. However, to do so by simple expansion of the equipment size would result in an unwieldy procedure, and some other method was sought.

It had been shown earlier, by work at Battelle and elsewhere, that distribution coefficients of rare earths and other contaminants into tributyl phosphate, ether, etc., are quite low when the organic is saturated, or nearly so, with thorium or uranium. This fact is the basis of the manifold separations of the shotgun test in concentrating the impurities for the neutron-absorption determination. Application of this technique to pre-concentrate the rare earths prior to the final cellulose-column separation permits a much larger sample to be used.

Mixtures of tributyl phosphate and ether were examined first. Into a quantity of a TBP:ether mixture was poured sufficient molten uranyl nitrate hexahydrate just above the freezing point (~ 70 to 80°C) to approximately saturate both phases. After equilibration the phases were separated and the aqueous phase, containing the rare earths, was given a final separation by means of the cellulose column. Preliminary tracer tests with Eu 152-154 confirmed the negligible loss of rare earths to the organic phase, and indicated that preliminary separation of the bulk of the uranium by solvent extraction was a feasible procedure. This was reconfirmed by a more complete subsequent tracer test, with hexone solvent, described later in the report.

TBP has several serious disadvantages as a solvent for extracting the bulk of the uranium. Its viscosity is high, and clean phase separations are nearly impossible, even when the TBP is diluted 50:50 with ether. Further, any TBP reporting in the aqueous phase is carried through to the final sample for analysis, due to its high boiling point. It is extremely difficult to remove completely, and thus introduces some phosphate interference. Ether has a desirable viscosity, and any ether which may be carried over into the aqueous phase is readily volatilized during the final evaporation of the sample. However, it is rather difficult to handle during the addition of the molten UNH; the ether boils violently, and considerable volatilization occurs. Hexone (methyl isobutyl ketone) is another suitable solvent, less volatile and more viscous than ether. It too can be volatilized from the final sample before spectrographic analysis. Phase separations, though reasonably complete, are not so clean as with ether. Hexone was used for most of the test work.

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Modified Test Procedure

Along with the preconcentration by solvent extraction, other modifications of the procedure described in BMI-260 were made in the interests of easier manipulation. Only one cellulose-column separation was made; uranium removal was sufficiently complete to eliminate uranium interference in the spectrographic assay. Continuous flow of the nitrated ether was substituted for the repeated washings of the cellulose wad containing the uranium and rare earths. Two or three such washings, primarily to displace the water and improve the ease of handling the wad, are desirable, however. A 100-g sample of uranium[as $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] is taken to a boiling point of just above that of molten hexahydrate, about 118 to 120 C (to minimize the amount of aqueous phase). Two or three drops of 30 per cent H_2O_2 are added, to insure presence of the cerium in the cerous state, and the solution is boiled several minutes more to remove excess peroxide. The sample is cooled to just above the freezing point (~ 70 to 80 C) and 200 ml of hexone is added. The sample is transferred to a separatory funnel and shaken moderately, allowed to settle, and the phases separated. Two 1 per cent (2 ml) scrubs with distilled or de-ionized water are made, the aqueous phases being combined with the main aqueous phase. The final aqueous volume will be about 25 to 35 ml and will contain 20 to 25 g of the original 100 g of uranium.* The combined aqueous is soaked up in about 5 g of activated cellulose. This wad is preferably given two or three washings with 5 per cent HNO_3 :ether, the supernatant ether being decanted to the already prepared column containing about 8 g of activated cellulose. The wad is then transferred to the column. This can be done without the ether washes; however, the wad has a tendency to disintegrate when still wet with water, and packs poorly. Displacement of the water by the ether washes improves the handleability of the wad materially.

The column has previously been conditioned by passing 200 to 250 ml of 5 per cent HNO_3 :ether through it. After addition of the wad, 500 ml of 5 per cent HNO_3 :ether are passed through the column, at a flow rate of 2 to 5 ml/min, to remove the uranium. (Uranium removal is essentially complete after 250 ml have passed through; only a few milligrams are contained in the second 250 ml.)

The column is then eluted with 500 ml of 2N HNO_3 (prepared with de-ionized water) to remove the adsorbed rare earths. (The tracer test indicated that removal of rare earths is virtually quantitative after only 250 ml). Ether is removed from the eluate on a steam bath, and the eluate

* The original aqueous raffinate is near saturation and some UNH will crystallize out if cooled to room temperature.

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concentrated to ca. 1 ml for spectrographic analysis. Iron appears frequently as a major impurity, and it may be necessary to eliminate iron before spectrographic analysis.

If the initial uranium samples are impure, it may be advisable to further increase the sample size beyond 100 g in order to improve the sensitivity of the spectrographic analysis. In one such case, 400-g samples were taken. These were reduced to ca. 100 g. by extracting with 800 ml of hexone by the procedure described earlier. The aqueous raffinate plus scrub was reconcentrated to a boiling point of 118 to 120 C and re-extracted with the usual 200 ml of hexone. The final aqueous raffinates contained 19 to 22 g. of uranium. Due to the amount of salts in these samples, the minimum volume to which the final eluate from the cellulose column could be concentrated was 10 ml, at which concentration the spectrographic assay was made.

Increasing the sample size to 400 g also increases the sensitivity for high-purity uranium beyond that attainable with a 100-g sample.

Rare-Earth Tracer Test

The modified procedure used in determining rare earths in uranium was checked, using Eu 152-154 tracer. Freshly extracted uranium was used in order to minimize the counts of the tracer-free uranium blank; uranium in radioactive equilibrium had an undesirably high activity.

To 147 ml of UNH solution (100 g U) was added 100 λ of Eu 152-154 solution (equivalent to 500 micrograms of Eu). This solution and a companion tracer-free blank solution were carried through the modified procedure. Activities of the various phases were measured with a dipping-type Geiger solution counter, using the technique described in BMI-261*. A count rate meter was also used to obtain approximate scannings of the cellulose column at various stages in the procedure.

The tabulated results of this test are shown in Table 1. Summarized, the distribution of activity was as follows:

<u>Extraction</u>	<u>Per Cent</u>
Organic phase	0.5
Aqueous phase	99.5
	<u>100.0</u>

* BMI-261, "Distribution of Rare Earth Tracers between Aqueous and Tributyl Phosphate-Diluent Phases".

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TABLE 1. TRACER TEST WITH EUROPIUM 152-154

Procedure: 100 λ of Eu 152-154 solution ($\approx$ 500 micrograms of Eu) was added to 100 g of uranium [as $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] and carried through the modified cellulose-column procedure. A companion tracer-free uranium blank sample was similarly treated.

	Uranium Blank		Eu 152-154 Spike		Net Counts/Min for Eu 152-154
	ml	Total Counts/Min	ml	Total Counts/Min	
<u>Solvent Extraction</u>					
Organic	242	114, 000	243	137, 000	23, 000
Aqueous	22.5	204, 000	24.0	5, 280, 000	5, 076, 000
		318, 000		5, 417, 000	5, 099, 000
<u>Uranium Desorption</u>					
Fraction I	250	9, 900	250	12, 700	2, 800
Fraction II	250	3, 750	250	1, 250	-2, 500
		13, 650		13, 950	300
<u>2N HNO₃ Eluate</u>					
Fraction I	260	193, 500	260	5, 200, 000	5, 006, 500
Fraction II	250	600	250	2, 500	1, 900
Fraction III	100	150	100	550	400
		194, 250		5, 203, 050	5, 008, 800

Note: Solutions were counted with a thin-walled dipping-type Geiger counter, shielded with 2 inches of lead.

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<u>Cellulose Column</u>	<u>Per Cent</u>
Ether effluent (500 ml)	< 0.01
On column after elution	Negligible
Unaccounted for	1.3
Eluate - 1st 260 ml	98.2
2nd 250 ml	0.04
3rd 100 ml	< 0.01
	99.5+

Loss of europium to the organic phase of the initial solvent extraction was negligible, only 0.5 per cent, confirming the validity of this preconcentration step. Virtually no europium was removed during the elution of the uranium from the cellulose (<0.01 per cent). Elution with the 2N HNO₃ removed the europium essentially quantitatively, leaving virtually none on the cellulose. 98.2 per cent of the europium added was found in the eluate, of which nearly all was removed from the cellulose by the first 260 ml of 2N HNO₃.

The "unaccounted for" value was fairly low throughout the test, considering the number of extra manipulations and transfers performed. The normal procedure would have a minimum number of extra manipulations, and the recovery should be easily in excess of 99 per cent.

The columns were scanned with a count rate meter (Nuclear Instrument and Chemical Corporation) after passage of 250 ml and of 500 ml of nitrated ether. The counter-tube face was shielded by a 1/4-inch lead plate pierced with a 3/32-inch-diameter hole. The sides of the tube were not shielded and definition was consequently not very sharp. The presence of radiation from an outside source also contributed somewhat to the background.

The results, tabulated in Table 2, show that movement of the europium was slight during passage of the nitrated ether. The columns were also scanned during the elution with 2N HNO₃. After collection of 100 ml of eluate, counts above 3000/min were obtained only over the bottom 2 inches of the column. The columns were scanned again (in an adjacent room) after completion of the elution; counts ranged from 0 to 20/min over the entire length of the column for both the blank and the europium column, indicating essentially complete removal of the europium.

Loss of europium during the removal of iron from the concentrated eluate was also investigated. Iron was removed by the usual cold HCl-ether solvent-extraction method*. The nitrate eluate from the cellulose column

*Lundell and Hoffman, Outlines of Methods of Quantitative Analysis (1938) p 96.

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TABLE 2. SCANNING OF CELLULOSE COLUMNS

(8 g Original Cellulose + 5 g Cellulose Wad)

Inches From Top of Cellulose	Counts/Minute During Uranium Desorption Cycle			
	After 250 ml		After 500 ml	
	Uranium Blank	Eu 152-154 Spike	Uranium Blank	Eu 152-154 Spike
0		22,000		22,500
1		29,000		28,500
2		31,000		31,000
3	850-900	30,500	800-900	31,000
4		29,000		31,000
5		20,000		29,000
6		12,000		23,500
7		9,000		11,500
8	800-900	6,500	800-900	9,500
9		--		--
10		3,500		4,000
11	750-800	--	800-900	--
12		2,000		2,800
Background	800-900	1600-1800	800-900	2,100

Note: Counts/min as indicated by a count rate meter with tube face against the column. Face shielded with 1/4-inch lead plate; 3/32-inch diameter hole; no shielding on sides.

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was evaporated to ca. 10 ml, precipitated with NH_4OH , and filtered on No. 40 Whatman filter paper. The precipitate was dissolved with 1:1 HCl, to a total volume of 10 ml. This was chilled and extracted three times with 50 ml of cold HCl-saturated ether. The distribution of activity was as follows:

	<u>Per Cent</u>
In NH_4OH Filtrate	0.5
Adsorbed on Filter Paper	3.1
In Ether	< 0.01
Unaccounted for	1.9
Purified Sample	<u>94.5</u>
	100.0

The indications were that the precipitation and extraction, as such, introduced no serious loss of rare earth, but that adsorption by the filter paper did. Substitution of a fritted filter and centrifugation were tried as alternatives to the use of paper. The beaker used for precipitation and the fritted filter were boiled in dilute HCl to secure an estimate of these losses. The distribution of activity in this test was as follows:

	<u>Per Cent</u>
Loss to Beaker	0.7
Loss to NH_4OH Filtrate	0.3
Loss to Fritted Filter	0.07
Unaccounted for	Negligible
Purified Sample	<u>98.9</u>
	100.0

Centrifugation also provided 99 per cent recovery. Rinsing of the beaker used for precipitation with some of the 1:1 HCl used to dissolve the precipitate should minimize this small source of loss, without increasing contaminant pickup to any extent.

Unfortunately, the activity of the final sample precluded spectrographic analysis as a further check on recovery of the spike.

Results on Uranium Samples

Three samples of orange oxide (UO_3) of known shotgun assays were supplied by Mallinckrodt Chemical Works. These were identified as follows:

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<u>Code</u>	<u>Manufacturer</u>	<u>Shotgun</u>
51420	MCW	0.00
51766	MCW	0.03
52162	Harshaw	0.06

As mentioned earlier, these samples were initially analyzed for rare earths by the analytical division, using the procedure substantially as described in BMI-260. The principal departure from the described method was the substitution of 5 per cent HNO_3 :ether for the 12.5 per cent HNO_3 :ether required to desorb thorium from a cellulose column. Twenty-five-gram samples were taken; the eluates were concentrated to 1 ml and analyzed spectrographically. (Table 3).

Cerium, although found, was at the same level in a reagent blank also carried through the procedure, and the indications are that contamination accounted for the high level of cerium. The contamination appeared to have arisen from conducting the cellulose-column separations in the same laboratory in which routine ceric sulfate titrations of uranium are performed.

Other than cerium, the only rare earths detected were yttrium and ytterbium, in No. 52162, the 0.06 shotgun sample. However, it is reported that 11 ppm of ytterbium or 45 ppm of yttrium is required to produce a shotgun value of 0.01, so that the contribution of these elements to the 0.06 shotgun was negligible. On the other hand, it is reported that only 0.017 ppm of gadolinium is required to produce a 0.01 shotgun. (Gadolinium has the highest cross section of all of the elements.) The sensitivity of this test provided only a <0.04 ppm value for gadolinium. Since further concentration of the final 1-ml sample would not improve the sensitivity sufficiently, it was indicated that a larger sample was required.

After development of the modified procedure, these samples were analyzed again, using 100-g starting samples. Increasing the weight of uranium taken, from 25 g to 100 g, increased the sensitivity by a factor of from 2 to 10. Again, however, except for yttrium and ytterbium in No. 52162, all of the rare earths were still below limits of detection. (See Table 3.) The values for yttrium and ytterbium were in reasonable agreement with the original assay of No. 52162, though probably the better values.

In addition, No. 51420 and No. 51766 were composited to produce a 400 g starting sample (average shotgun = 0.015), which was similarly analyzed, using a double hexone extraction to reduce the sample size prior to the cellulose-column separation. This further increased the sensitivity, but the rare earths were still below the limits of detection. Gadolinium, for example, was determined to be present to the extent of only <0.002 ppm (equivalent to a shotgun value of only 0.002). It seems obvious from these

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TABLE 3. RARE-EARTH CONTENT OF SEVERAL
SAMPLES OF REFINED URANIUM

Rare Earth	ppm, Uranium Basis						400-g Sample
	25-g Samples			100-g Samples			51420 +
	51420	51766	52162	51420	51766	52162	51766 ⁽¹⁾
Y	< 0.005	< 0.005	0.42	< 0.002	< 0.002	0.30	< 0.0005
La	< 0.01	< 0.01	< 0.01	< 0.005	< 0.005	< 0.005	< 0.0025
Ce	1.5 ⁽²⁾	1.5 ⁽²⁾	1.5 ⁽²⁾	< 0.04	< 0.04	< 0.04	< 0.013
Pr	< 0.15	< 0.15	< 0.15	< 0.04	< 0.04	< 0.04	< 0.015
Nd	< 0.15	< 0.15	< 0.15	< 0.04	< 0.04	< 0.04	< 0.010
Sm	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.008
Eu	< 0.01	< 0.01	< 0.01	< 0.004	< 0.004	< 0.004	< 0.001
Gd	< 0.04	< 0.04	< 0.04	< 0.015	< 0.015	< 0.015	< 0.002
Dy	< 0.05	< 0.05	< 0.05	--	--	--	< 0.0025
Ho	< 0.03	< 0.03	< 0.03	< 0.005	< 0.005	< 0.005	< 0.001
Er	< 0.04	< 0.04	< 0.04	< 0.01	< 0.01	< 0.01	< 0.004
Tm	< 0.03	< 0.03	< 0.03	< 0.005	< 0.005	< 0.005	< 0.001
Yb	< 0.01	< 0.01	0.075	< 0.002	< 0.002	0.05	< 0.0005
Lu	< 0.01	< 0.01	< 0.01	< 0.002	< 0.002	< 0.002	< 0.001

Notes

- (1) Composite of 200 g each of No. 51420 and No. 51766
(2) Contaminated with cerium; see text

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analyses that the contribution of the rare earths to shotgun values in refined virgin uranium is negligible.

Direct spectrographic analyses were also made of these samples. These revealed the following boron and cadmium concentrations:

	ppm, Uranium Basis		
	<u>51420</u>	<u>51766</u>	<u>52162</u>
Boron	0.14	0.14	0.17
Cadmium	0.18	0.10	0.08

Based on the reported values of 0.05 ppm boron or 0.12 ppm cadmium for a 0.01 shotgun, the calculated shotguns for these samples would be from 0.036 to 0.042.

Four samples of UNH liquor were also supplied by the FMPC for rare-earth analysis. These were products of early pilot runs, made during the shake-down period, the objective of which was to determine capacities and operating characteristics of the pulse column. Degree of saturation of the organic was at a low level in these tests. Purity was not of primary concern, and consequently, the purity level of the samples was low. Shotguns were reported to range from 0.10 to 0.22. Contributing to the low purity was the fact that it had been necessary to use softened, rather than de-ionized, water for re-extraction. As a result, concentration of sodium in the liquors was high. The following direct spectrographic analyses were obtained on three of these samples:

<u>Element</u>	ppm, Uranium Basis		
	<u>Drum 2</u>	<u>Drum 63</u>	<u>Drum 69</u>
Na	100-1000	100-1000	100-1000
Ca	15	10	150
Mg	15	10	25
Ba	< 5	< 5	< 5
Fe	25	300	630
Al	< 10	< 10	< 10
Ni	8	18	30
Co	< 3	< 3	20
Cu	35	70	45
Pb	3	6	15
Cr	< 10	22	15
Mo	35	45	60
V	< 5	< 5	5
Zn	< 10	15	30
Mn	< 3	3	10
P	< 80	90	100
Cd	< 0.25	< 0.25	9.0
B	1.4	0.52	0.43

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In an attempt to increase the intensity of the rare-earth lines, 400-g samples were taken for the rare-earth analyses. However, even after an iron separation, it was possible to concentrate the eluates only to 10 ml for spectrographic analysis, due to the high content of soluble salts. Below this volume, crystallization occurred. Sensitivity was reduced because of the dilution and the high contaminant level, as indicated by the results, shown in Table 4. Also shown are spectrographic analyses of the 10-ml eluates for elements other than rare earths which were present in appreciable concentrations. The level of rare earths was higher than in the MCW samples; yttrium was detected in all four samples, and lanthanum, cerium, and neodymium were detected in Drum No. 63. Even so, none of these were at a level which would contribute significantly to the shotgun values for these samples, confirming the results on the MCW samples.

Greater sensitivity could have been attained by further purification of the 10-ml eluates before spectrographic analysis. Normally however, refined uranium will be of sufficient purity that this step will be unnecessary.

All spectrographic analyses made during this investigation were performed with an Applied Research Laboratory 1.5-meter grating spectrograph, using the techniques described in BMI-260.

The original data upon which this report was based are recorded in Laboratory Record Book 6440, on pages 17, 26-56, 66-69, 73-88, 95-98.

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TABLE 4. RARE-EARTH CONTENT OF SEVERAL SAMPLES OF URANIUM LIQUORS
FROM THE FMPC PILOT PLANT

Element	ppm, Uranium Basis				Element	ppm in 10-ml Eluate Sample			
	Drum 2	Drum 63	Drum 69	Run 4		Drum 2	Drum 63	Drum 69	Run 4
Y	0.027	0.037	0.19	0.005	Ca	100	80	500	100
La	<0.025	0.35	<0.025	<0.025	K	<200	<200	<200	<200
Ce	<0.15	0.37	<0.15	<0.15	Si	15	40	15	100
Pr	<0.15	<0.15	<0.15	<0.15	Mg	20	20	200	200
Nd	<0.10	0.10	<0.10	<0.10	Pb	150	40	400	20
Sm	<0.075	<0.075	<0.075	<0.075	Cr	20	200	40	40
Eu	<0.013	<0.013	<0.013	<0.013	Al	40	100	200	200
Gd	<0.025	<0.025	<0.025	<0.025	Mn	5	40	300	20
Ho	<0.025	<0.025	<0.025	<0.025	Sn	4	60	10	4
Er	<0.04	<0.04	<0.04	<0.04	Zn	5	30	150	40
Tm	<0.013	<0.013	<0.013	<0.013	V	8	15	40	100
Yb	<0.02	<0.02	<0.02	<0.02	P	100	300	400	100
Lu	<0.01	<0.01	<0.01	<0.01					

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RECOMMENDATIONS FOR FUTURE WORK

If further work is carried on with the objective of substituting spectrographic methods for the shotgun test, it is recommended that attention be turned toward other high-cross-section elements, i. e., boron, cadmium, etc. No further work on rare earths appears necessary; the indications are that these have little significance as far as shotgun values in refined uranium are concerned.

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