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Report A-131a

Purification of Uranium Oxide

by

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I. Introduction

Great difficulty has been experienced in the purification of uranium oxide, especially in the removal of small amounts of the rare earth elements. It was found that 90 g of uranyl nitrate, $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, can be dissolved in 100 ml of ether. If a little water is added and the mixture is vigorously shaken, any rare earths present will pass into the water extract. By shaking a mixture of 2.6 mg of rare earth oxides, 90 g of uranyl nitrate (equivalent to 50 g of U_3O_8), and 5 ml of water with 100 ml of ether, 2.6 mg of rare earth oxides were recovered in the combined water extracts obtained by 3 successive extractions with 5-ml portions of water. In a similar experiment with 0.0262 g of rare earth oxides, 0.0258 g was recovered. In both experiments, the ether extract contained about four fifths of the uranium, and the water extract about one fifth. Practically all other impurities, excepting possibly molybdenum, follow the rare earths. This forms the basis of a simple method of purifying crude uranium oxide, and of extracting some uranium from carnotite.

8603-1

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II. Procedure

1. Method for purification of uranium oxide.

Transfer 50.0 g of U_3O_8 to a 600-ml beaker, add 75 ml of diluted nitric acid (1-1)¹, and digest on the steambath until all action ceases.

¹ This denotes 1 volume of concentrated nitric acid, sp.gr.1.41 diluted with 1 volume of water. This system of designating diluted acids is used throughout this paper.

Remove the insoluble matter by filtration, and wash the residue on the paper 4 or 5 times with diluted nitric acid (5 + 95). Evaporate the filtrate containing the uranyl nitrate to dryness on the steambath. Cool the dried residue, add 100 ml of ether and 5 ml of water, and stir or swirl until all the uranyl nitrate is dissolved. This may require one half to one hour depending on the size of the crystals and on the impurities in the material. Large quantities of impurities may cause the water in the bottom of the beaker to have the appearance of an emulsion. This does not interfere in the separation because the ether can be poured into a separatory funnel without mixing with the water layer.

Transfer the ether to a separatory funnel, wash the beaker 3 times with 5-ml portions of ether, and transfer these portions of ether to the separatory funnel. Stopper the funnel with a cork, and shake vigorously for 1/2 to 1 minute. Allow the liquid to stand for a few minutes, and, if a small portion of water layer appears at the bottom of the separatory funnel, drain it into the beaker which originally contained the ether solution. Now add 5 ml of water to the solution in the funnel, shake vigorously, allow the solution to separate into 2 layers, and again drain the water layer into the beaker which originally contained the ether

solution. Repeat the extraction with another 5-ml portion of water. Drain the ether into a suitable dish (platinum or silica), evaporate the ether, gradually heat and finally ignite the residue at 1000 C.

III. Results obtained in the purification of uranium oxide.

The efficacy of this method of purification is shown in Table 1.

IV. Discussion

The purification of crude uranium oxide by partition of uranyl nitrate between water and ether has commercial possibilities because practically all impurities are removed in a single operation. The reagents, namely: nitric acid, ether, and water are inexpensive when it is realized that the ether can be recovered and used again. The fire hazard must be considered, but it is no greater than in many other commercial processes. In the purification of U_3O_8 the yield is about 80 percent, but the uranium passing into the water extracts is not permanently lost because it can all be recovered by reworking these residues.

Evaporations of ether from the uranyl nitrate should be done at a low temperature under reduced pressure if large amounts are involved because there is a possibility of rather violent reaction between uranyl nitrate and ether if the temperature is too high at the time when nearly all the ether is gone.

The recovery of uranium from pitchblende and carnotite is described in Report A-131b.

Table 1.

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A-131a

Results obtained in the purification of uranium oxide or its recovery from ores by dissolving uranyl nitrate in ether and extracting with water. (Spectrochemical analysis^a)

Element	U ₃ O ₈ # 15-5			U ₃ O ₈ # 181			Carnotite Ore	
	Original Oxide	Purified Oxide	Water extract ignited to oxide	Original Oxide	Purified Oxide	Water extract ignited to oxide	Original Ore	U ₃ O ₈ obtained by procedure 2
Ag	T	T	W	VW	T	W	?	T
Al	VW?	VW?	W	VW	VW	W	S	-
As	W	-	M	W	-	W	-	-
Au	-	-	T	VW	-	VW	T	-
B	T	-	VW	T	-	-	-	T
Ba	-	-	-	-	-	VW	M	-
Be	-	-	-	-	-	-	-	-
Bi	VW	-	W	VW	-	W	-	-
C	-	-	-	-	-	-	-	-
Ca	VW	-?	W	W	T	M	VS	T
Cb	-	-	-	-	-	-	-	-
Cd	-	-	-	-	-	-	-	-
Ce	-	-	-	-	-	-	-	-
Co	-	-	-	W	-	M	W	-
Cr	-	-	VW	-	-	VW	-	-
Cs	?	?	?	?	?	?	-	-
Cu	VW	T	M	W	T	L	T	T
Dy	-	-	-	-	-	-	-	-
Er	-	-	-	-	-	-	-	-
Eu	-	-	-	-	-	-	-	-
Fe	W	VW?	M	W	VW?	M	S	M
Ga	-	-	?	-	-	-	?	-
Gd	-	-	-	-	-	-	-	-
Ge	-	-	-	-	-	-	-	-
Hf	-	-	-	-	-	-	-	-
Hg	-	-	-	-	-	-	-	-
Ho	-	-	-	-	-	-	-	-
In	-	-	-	-	-	VW	-	-
Ir	-	-	-	-	-	-	-	-
K	T	-	W	W	-	M	-	-
La	-	-	-	-	-	-	-	-
Li	-	-	T	-	-	VW	-	-
Lu	-	-	-	-	-	-	-	-
Mg	W	T	W	W	VW	W	M	T
Mn	VW	-	W	W	-	W	W	-
Mo	W	W?	M	W	-	M	-	-
Na	W	T	M	W	T	S	-	-
Nd	-	-	-	-	-	-	-	-
Ni	-	-	VW	VW	-	W	-	-
Os	-	-	-	-	-	-	-	-
P	-	-	-	-	-	-	-	-
Pb	VW	-	W	W	-	M	VW	-
Pd	-	-	-	-	-	-	-	-
Pr	-	-	-	-	-	-	-	-
Pt	-	-	-	-	-	-	-	-
Ra	-	-	-	-	-	-	-	-
Rb	-	-	-	-	-	-	-	-
Re	-	-	-	-	-	-	-	-
Rh	-	-	-	-	-	-	-	-
Ru	-	-	-	-	-	-	-	-
Sb	W	-	M	W	-	L	-	-
Sc	-	-	-	-	-	-	-	-
Si	L	VW	L	L	W	W	S	VW
Sm	-	-	-	-	-	-	-	-
Sn	-	-	VW	-	-	-	-	-
Sr	-	-	-	-	-	VW	W	-
Ta	-	-	-	-	-	-	-	-
Tb	-	-	-	-	-	-	-	-
Te	-	-	-	-	-	-	-	-
Th	-	-	-	-	-	-	-	-
Ti	-	-	-	-	-	-	-	-
Tl	-	-	-	-	-	-	-	-
Tm	-	-	-	-	-	-	-	-
U	VS	VS	VS	-	-	-	-	-
V	VW	-	W	-	-	-	VS	-
W	-	-	-	-	-	-	-	-
Y	-	-	-	-	-	-	-	-
Yb	-	-	-	-	-	-	-	-
Zn	-	-	VW	-	-	VW	-	-
Zr	-	-	-	-	-	-	-	-

a - Spectrochemical analysis by

B. F. Scribner and H. R. Mullin. It should

be noted that, owing to the complexity of

the uranium spectrum, many impurities actually present in the original material were not detected until they were extracted chemically and further concentrated.

b - In addition to the elements designated

in this column, the following rare

earth elements were found in the

water extract, named in order of de-

creasing magnitude: Y, Dy, Eu, Gd, Tb,

Ho, Er, Yb, Tm, Ce, Pr, Nd, Sm,

Sc, Lu and La.

In general, the designations VS and S correspond to major constituents (greater than 1%), M and W to minor constituents (1 to 0.01%), and VW, T, and FT to trace constituents (less than 0.01%).

The absence of a designation indicates that a test was not made for that element.

8608-4