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DISTRIBUTION OF IMPURITIES IN SEP-II

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DISTRIBUTION OF IMPURITIES IN STEP II

I. INTRODUCTION

When uranium is purified by the ether extraction method, black oxide is dissolved in nitric acid and ether is added; an ether layer containing uranium and an aqueous layer containing uranium and impurities are obtained. The distribution of some impurities between the two phases and the effect of such impurities on the extraction process were studied.

II. DISCUSSION

The processing required in the preparation of pure uranyl nitrate might be decreased if, instead of uranosic oxide, sodium diuranate were used as a source of uranium. If it is assumed that uranosic oxide contains the same amounts of impurities as sodium diuranate, with the exception of soda, the extraction of soda by the wash of an ether solution of nitrate prepared from diuranate would be the criterion of suitability of sodium diuranate. The distribution and effect of sodium in the extraction process were therefore investigated, and the results are described in this report.

For various of the impurities, the distribution in the process and the effect on uranium holdup in the insoluble cake were investigated. Particular attention was given to the behavior of boron, vanadium, chromium, and molybdenum.

III. EXPERIMENTAL

Starting with a sodium diurante containing 10% Na_2O and 85% U_3O_8, a solution was made by dissolving in nitric acid and evaporating to the hexahydrate. A saturated solution of the resulting solid in ether was washed several times with distilled water (in the proportion 150 cc. ether layer to 5 cc. water). Analysis of the ether layer showed the Na_2O content to be .05% (based on U_3O_8). This amount of sodium was very soluble in ether and could not be removed by washing. Since the sodium could have been held up by organic matter, from the wood tanks used in previous processing, three runs were made using untreated and calcined sodium diuranate and a synthetic mixture of $NaNO_3$ and uranyl nitrate. These were then treated as in the previous run. The following table gives the content of Na_2O in uranyl nitrate obtained from the ether layer after various degrees of purification.

Material	Wash 0	1	2	3	4	5	6	7
Blend 2A Untreated	0.03%	0.03%	0.03%	0.04%	0.05%	0.04%	0.03%	0.01%
Blend 2A Untreated	0.015	0.012	-	0.0025	-	0.018	-	0.0016
Blend 2A Calcined	0.0072	0.0009	-	0.002	-	nil	-	nil
Blend 2A Calcined	-	0.002	-	nil	-	-	-	-
$UO_2(NO_3)_2 \cdot 6H_2O/NaNO_3$	0.021	0.004	-	nil	-	nil	-	0.001

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It can be concluded that some impurity in the sodium diuranate prevents complete removal of sodium with the limited number of washes which can be used in the washing step. If this contamination of 20 to 30 ppm. of soda (in a 53% uranium material) is above specification for soda, it would be necessary to calcine the sodium diuranate before dissolving in nitric acid.

Several small scale ether extractions were made in the Proving Laboratory with mixtures of uranyl nitrate hexahydrate and sodium nitrate, corresponding to the sodium-uranium ratios in various grades of orange and yellow varieties of sodium diuranate. No difficulty was experienced with diuranates containing up to 10% Na_2O. However, some solid sodium nitrate remained when higher soda types were used. The nitrate crystals adhered to the walls of the vessel and interfered with the separation and washing of the ether layer.

Since the purification of uranyl nitrate depends on the extraction of impurities from an ethereal solution of uranyl nitrate, it is essential that the impurities be insoluble in ether or have a distribution coefficient higher than uranyl nitrate. The purification was studied with particular reference to boron, vanadium, chromium, and molybdenum, because they might be introduced by the equipment or ore. Both vanadium and chromium had very high distribution coefficients, since none could be found in the ether layer when 1% (based on uranyl nitrate) was added. Attempts to determine the numerical value of the distribution coefficient of boron were unsuccessful, but it appears that boron has a higher distribution coefficient than uranium, and can, therefore, be extracted by water washes. When sodium molybdate equivalent to 1% of the uranium present was added to a saturated ether layer, it was not possible to remove all the molybdenum by washing the ether layer with water, but it was possible when a concentrated uranyl nitrate solution was used instead of water. However, when uranium compounds containing 1% molybdenum were carried through the processing steps which are used in the Ceramics Plant and finally converted to uranyl nitrate, which was then dissolved in ether, the molybdenum was removed from the ether layer by several washes with water. The difference is due to the filtration of the uranyl nitrate hexahydrate just before ether purification. An insoluble uranyl molybdate is formed which is filtered off, thus removing the bulk of the molybdenum.

When black oxide is dissolved in nitric acid, varying amounts of uranium are retained in the nitric acid-insoluble portion. A correlation between the uranium holdup and assay of the black oxide was noted. To check which impurities cause this holdup, several black oxides were prepared by calcining ammonium diuranate, which had been precipitated from a solution containing uranyl nitrate and the impurity to be studied. The blacks were dissolved in nitric acid according to the procedure used in the Ceramics Plant, and the solution filtered. The following table gives a correlation of data and calculations:

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Impurity	Composition of Black		Percent Holdup in Cake		U_3O_8/ Impurity
	U_3O_8	Impurity	U_3O_8	Impurity	in Cake
Fe_2O_3	87.4%	2.62%	0.16%	44.6%	0.005
SiO_2	93.6	4.51	0.34	97.5	0.07
WO_3	94.0	4.62	1.49	92.2	0.3[illegible]
P_2O_5	93.9	5.01	15.9	47.4	6.3
MoO_3	93.6	4.95	5.5	73.5	1.4
Sb_2O_3	95.8	3.17	6.24	100	1.7
TeO_2	95.1	3.02	6.35	100	1.8
V_2O_5	95.1	3.01	4.9	100	1.5

Calcium, arsenic and boron were also tried but gave no insoluble cake.

The ratios of uranium to impurities in the insoluble cakes indicate that phosphorus causes the greatest holdup of uranium, and, therefore, is the impurity which should be held to the lowest limits to prevent excess uranium retention in the acid-insoluble cake.

IV. SUMMARY

Sodium di-uranate was successfully purified by the ether extraction process. High concentrations of sodium caused mechanical difficulties through the precipitation of sodium nitrate in the water layer. Processing and extraction of the sodium salts, as produced in the process for the treatment of domestic ores, was not successful, because the sodium was not completely removed in the ether extraction. This was apparently due to the formation of organic salts.

A number of impurities were tested for their effect on uranium holdup. It was established that the presence of P_2O_5 in the uranium oxide led to unusually high uranium losses.