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UNITED STATES ATOMIC ENERGY COMMISSION
THE DISTRIBUTION OF PRECIOUS METALS IN
VARIOUS RESIDUES OBTAINED IN THE
PREPARATION OF URANYL NITRATE FROM
PITCHBLEND.

By
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THE DISTRIBUTION OF PRECIOUS METALS IN VARIOUS RESIDUES
OBTAINED IN THE PREPARATION OF URANYL NITRATE
FROM PITCHBLLENDE

Introduction

In a process involving the extraction of uranium from a pitchblende ore and in the subsequent purification of the extracted uranyl nitrate several different residues are obtained. Since the pitchblende is known to contain appreciable quantities of the precious metals, namely gold, platinum, and palladium, a sampling program was arranged to determine how these metals distributed themselves in these residues.

Samples of the ore, the residues, and liquors at various points in the process were taken and sent to the National Bureau of Standards for analyses.

Discussion

The essential processing steps which give the residues that were studied for their precious metals content are:

1. The ore is digested in nitric acid with sulfuric acid added to precipitate lead and radium and other insoluble sulfates; the slurry is filtered giving gangue lead cake and gangue lead filtrate (a solution of uranyl nitrate).
2. Barium carbonate is used to precipitate the excess sulfate and this slurry is filtered to give barium sulfate cake and

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barium filtrate.

3. The barium filtrate is concentrated in an evaporator--which brings down some solids--diluted, and filtered giving boil-down cake (principally BaSO_4) and boildown filtrate.

A modification of steps two and three just described involves precipitating barium sulfate and concentrating the slurry before making a single filtration instead of a separate barium sulfate filtration and a separate boildown filtration. This single filtration scheme was followed in two of the three batches which were sampled for a precious metals study.

After salting the boildown filtrate with calcium nitrate and checking and adjusting the solution's acidity the liquor is described as column feed.

4. The column feed is sent into an ether extraction column--the ether extracting almost exclusively the uranyl nitrate--and the water layer containing the calcium nitrate and impurities is drained off as the raffinate.

The raffinate is de-etherized, concentrated, neutralized with calcium hydroxide and filtered to give neutralized raffinate cake and neutralized raffinate filtrate. The latter solution consisting of primarily calcium nitrate is crystallized; a certain portion of the salt in the aqueous mother liquor is discarded to the sewer.

Thus, the precious metals enter the process in the ore and leave the process in any or all of the following materials:

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- (1) the gangue lead cake
 - (2) the barium sulfate cake
 - (3) the beddown cake
 - (4) the raffinate cake
- or (5) with the purified uranyl nitrate in the ether layer.

In order to establish the distribution of the precious metals in the residues, representative samples were taken at each step of the process and submitted to the National Bureau of Standards for determination of gold, platinum and palladium. A spectrographic check was made at Wallinckrodt for silver in ten different representative ore samples, with less than 2 ppm. detected. The pilot plant procedure used at the time the samples were taken represented as closely as possible the conditions which we expected the large production plant would follow; no part of the process was intended to be a treatment designed to handle the precious metals.

Included at the end of this report are three tables giving the results obtained on the three different sets of samples submitted for analysis; the three sets represent three batches of ore processed in our pilot plants.

I. Gold

Two of the three ore samples gave practically check results of 33 and 36 ppm. gold based on the uranium content (all values

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given in this report are based on parts precious metals per million parts uranium). The third sample-set value of 13 ppm. gold may be real and different because of difference in the ores or it may be a low result, since summing the quantities found in the various residues for each set indicates a gold content of about 25 ppm.

Because the results of liquors and cakes fail to balance within a given set of samples, it is difficult to determine the exact distribution of the gold to the various residues. However, by giving considerable credence to the amounts found in the residues it appears that between 80 to 95% of the gold originally present in the ore goes out in the gangue lead cake, and that the remaining 5 to 20% leaves in the barium sulfate cake with perhaps a trace leaving in the OK liquor, and with none detected in the raffinate.

II. Platinum

The ore results on platinum are similar to the just discussed results on gold in that they appear to be low when compared to the value obtained by totaling the individual residue values. Thus, on this basis, the amount of platinum originally present in the ore is in the order of 10 to 20 ppm.

A survey of the distribution of the platinum in the residues shows an extreme spread of results—10 to 80% of the platinum originally present remaining in the gangue lead cake, 0 to 30% in the barium sulfate cake, and 20 to 80% leaving in the raffinate

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cake. It is difficult to say what further sampling of other batches would show since the metal amounts dealt with are so small (in the part per million magnitude) that the spread is close to the limit of accuracy of the analytical determination.

III. Palladium

The amount of palladium present in the pitchblende ore is negligible for all practical purposes. The three ore samples checked with 0, 1, and 2 ppm. palladium detected. It should be noted that in each of the three sets of samples, the gangue lead cake contained all of the palladium originally present, and as in the cases of gold and platinum the amounts found in the gangue lead cake indicate that the ore analyses was low in its palladium answer.

Summary

A study was made of the distribution of four precious metals—silver, gold, platinum, and palladium—in the residues from a process whereby purified uranyl nitrate is prepared from a pitchblende ore. Samples were taken at various points in the process and analyzed for the metals mentioned.

The conclusions are as follows:

- (1) Neither silver nor palladium is present in the ore in significant quantity.
- (2) There is approximately 30 ppm. of gold (based on uranium

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For each 1,000,000 of uranium in the pitchblende ore which "Allinckrodt will receive and process there will be the following amounts (in pounds) of precious metals in the various solutions and residues:

<u>ilot Plant catch 15</u>				
<u>Flow</u>	<u>Material</u>	<u>Au</u>	<u>Pt</u> ^{pounds}	<u>Pd</u>
In	Ore	33	19	2
Out	Gangue Cake	31	17	7
Out	Barium Sulfate Cake	1	0	0
Out	Boil-down Cake	0	0	0
	Boil-down Filtrate	1	4	Not detected

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TABLE II

Pilot Plant Batch K110-52A106

<u>Flow</u>	<u>Material</u>	<u>Au</u>	<u>pounds</u> <u>Pt</u>	<u>Pd</u>
In	Ore	36	5	0
Out	Gangue - Lead Cake	30	8	2
	Gangue - Lead Filtrate	1	14	Not detected
Out	Barium Sulfate Cake (+ Boil-down Cake)	1	2	0
	* Barium Filtrate	2	15	Not detected
	* Column Feed	5	6	0
Out	OK Liquor	1	1	Not detected
	Raffinate	0	5	Not detected
Out	Neutralized Raffinate Cake	Not de- tected	1	—
Out	Neutralized Raffinate Filtrate	0	6	0

* These values should be practically identical.

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