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CENTRAL INTELLIGENCE AGENCY

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50X1-HUM

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50X1-HUM

1. Using a process developed in July 1947, a test installation is said to have produced a total of several hundred kilograms of metallic rhenium, making this metal available for a close study of its properties, its alloys and its uses as a catalytic agent. Previously, only small amounts of rhenium had been available at high cost through a small-scale production by Gebr. Borchers/Oker which used Mansfeld pig iron as a source. With the development of the new process, rhenium production by Gebr. Borchers has been discontinued and that firm has turned to the separation of the molybdenum, nickel, cobalt and copper which are also present in the Mansfeld pig.

50X1-HUM

the Mansfelder Kupferschieferbergbau A. G. is producing rhenium at the rate of 120 kg. per month.

50X1-HUM

2. Sometime during 1946, the Russians ordered W.Feit, an employee of the Vereinigte Kaliwerke in Aschersleben, to examine the Mansfeld intermediary products, especially those which appear to be rich in rhenium, with the object of possible direct extraction of water-soluble rhenium compounds. It also appeared that intermediary lead oxides produced at the Hettstedt Lead Works (Bleihütte) during a differential rolling process were relatively rich in rhenium. The copper division in the laboratory attached to the Kupferkammer and the Bleihütte and the Central Laboratory at Eisleben also became concerned with a technological process for obtaining rhenium, using as a source a fine lead dust recovered at the Bleihütte. This dust currently contains up to 90 grams of water-soluble rhenium per ton. It is considered advisable to assume an average of 60 grams per ton, of which 50 grams can be extracted.

3. The following process, which describes the production of crude rhenium sulphide, was developed to recover the rhenium in the lead dust in the form of potassium perrhenate and later as pure metallic powder. It was finally tested with good results in an experiment involving the use of 30 tons of basic material. The further processing of crude rhenium sulphide to obtain the above-mentioned end-product is comparatively simple and can be handled, in view of the small quantities, in a laboratory. On the basis of this, the Kupferkammer-Bleihütte in Hettstedt planned an installation described below.

Assuming a daily yield of 7 tons of lead oxide, which can be considered as maximum production for the near future, the process appears schematically as follows:

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50X1-HUM

7 tons of lead oxide, soaked with lye (laugen) and filtered.	
8 tons of lixiviated lead-slick (goes to the furnace)	8 cubic meters weak solution d 1.017 concentrated by evaporation to d 1.2
100 kg. of gypsum (damp)	1 cubic meter concentrated solution d 1.2 to be treated (zementieren) with 9 kg. zinc dust (Zinkstaub).
15 kg. filtrate (Cement-Cd) (damp)	1.1 m^3 of purified solution to be precipitated by 7 kg. Na_2S + 140 kg. H_2SO_4
waste lye	3.8 kg. of damp Re_2S_7. Dissolved in 5 kg HNO_3 and $KReO_4$ crystallized out by adding K_2CO_3.

4. Method of conveying seven tons of lead oxide

The lead oxide could be conveyed most efficiently and dust free if it is immediately wetted down in the filter house in a small mixer and if it is pumped from there for treatment with lye.

5. Lixiviating seven tons of lead oxide with nine cubic meters of water

The lead oxide slick is pumped into two containers each of ten cubic meter capacity where it is aerated for several hours by mechanical means. While one container is being filled the content of the other is being filtered.

6. Filtration

a. An experiment with a rotary filter with a 2.5 sq. meter filter surface showed that lead oxide slick can easily be filtered by such an apparatus. Performance per hour was 220 kg. of mud cake containing 27% moisture, equalling 180 kg. of dry slick or about 190 kg. lead oxide (sic).

b. A filter with a surface of 2.5 sq. meters operated for 6 hours per shift could process 1.14 tons of lead oxide. An existing 7 sq. meter rotary filter, which was used formerly to obtain cadmium and which is out of operation now, would be able to produce 3.2 tons of lead oxide. This means it would be capable of handling the lead oxide yield in three shifts per day.

c. During this test, lead oxide containing 86 grams of rhenium per ton was used. Of this amount 75 grams per ton were dissolved while 11 grams per ton were lost, i.e., 12.9% remained as water-soluble rhenium in the lead oxide slick. It may be possible to obtain better results by a stronger washing process. The resulting slick mass, about 8 tons, is loaded into carts and taken to the lead plant while the 9 to 10 cubic meters of filtrate and cleaning water have to be evaporated down to one cubic meter.

7. a. Tests have shown that evaporation cannot be carried out in a simple iron pan heated by coal because the chloride-containing lye attacks the iron too strongly. When evaporation of 70 liters of solution was carried out in an iron vessel during the experiment, seven grams of iron were dissolved. As a result, this process has to be carried out in lead equipment. The lead lined vaporizer of the zincvitriol installation, with a daily vaporization capacity of 15 cubic meters of water, was found to be suitable. It is a 30 cubic meter wooden cabinet measuring 4 x 3 x 2.5 meters, lined with lead and equipped with three lead-coated iron heating coils 35 meters in length. The steam consumption amounts to about 10 tons per day.

b. Since the vaporization decreases greatly with decreasing content it may be advisable to carry out the evaporation in two stages. By this process, a large pre-vaporizer would condense the solution down to about one quarter of its volume (from d 1.017 to d 1.085) and thereby reduce the quantity to be handled daily from ten cubic meters to two cubic meters. A small vaporizer could then evaporate the solution to one cubic meter of concentrate d 1.17 - 1.20.

8. When the concentrated solution (1 m3/day) is cooled, 100 kg. of gypsum are separated out. This can be carried out by feeding the solution alternately into two available lead-lined wooden cabinets of three cubic meters capacity each. These cabinets are equipped with water-cooled coils and the solution is cooled off in three days. The gypsum is collected on the bottom of the vat while the clear solution can be decanted. From time to time, the gypsum is washed with hot water in order to recover the rhenium content of the lye which adheres to the gypsum. This solution is also evaporated while the gypsum is shoveled out of the cooling cabinet.

9. The decanted clear lye (about one cubic meter per day) is pumped to the cadmium precipitator.* For this purpose a six cubic meter container with mixer is available and for the final filtering a 25 section, 50 x 50 cm. filter press, with pump, is used. The cadmium precipitation as well as the following rhenium precipitation is carried out every five days, whenever 5 cubic meters of lye have been collected.

10. The rhenium precipitation is carried out in a lead-lined six cubic meter vat which has a good drainage outlet, decanting taps, and pipes for air and steam. The Na_2S solution needed for the precipitation is prepared and filtered in a box of 0.5 cubic meter capacity. A vat having a capacity of one tank car would be needed for the sulphuric acid.**

11. For filtering the rhenium sulphide, two earthenware suction filters of about 75 cm diameter are sufficient. The acid filtrate is pumped to the waste dump (Halde). For processing the sulphide to $KReO_4$, 4 earthenware containers of 50 liters each are needed, one earthenware suction filter of 50 cm. diameter and five evaporation pans of 50 liters capacity. Reduction of the potassium perrhenate into metallic rhenium was accomplished in an electric pipe furnace in an atmosphere of hydrogen.

* [redacted] Comment: Apparently cadmium is also present in the raw material. 50X1-HUM

** [redacted] Comment: The word used is Schwefelsäure. The schematic outline of the process indicates that HNO_3 is added at this point.