

UNITED STATES PATENT OFFICE.

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METHOD OF MANUFACTURING THORIUM NITRATE.

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Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that I, HERBERT N. MCCOY, a citizen of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented a new and useful Improvement in Methods of Manufacturing Thorium Nitrate, of which the following is a specification.

The present invention relates to processes for the purification of thorium compounds and for the production of a purified thorium nitrate therefrom.

This process comprises a plurality of steps which are directed to the treatment of naturally occurring thorium-containing compounds for the concentration of the thorium content therein and its final purification. It is described in the following specification as applied to the treatment of an India monazite sand from which the greater part of the ilmenite content has already been removed by a process of magnetic separation. This product contains approximately 92% of monazite sand.

In accordance with this process, the monazite sand thus obtained is treated in batches containing approximately 460 pounds. The batch of monazite sand is first decomposed in such manner as to convert the thorium into an insoluble compound readily separable from other constituents of the monazite. This is effected in the following manner:

A batch of approximately 460 pounds of the monazite sand, already slightly concentrated by a magnetic separation process, is charged into a cast iron pot with 475 pounds of concentrated sulfuric acid (specific gravity 1.84). The pot containing this batch is then heated by means of a low gas flame, a slow heating being maintained until the material begins to stiffen, for example, for a period of about four hours. At this stage, the temperature of the material is about 180° to 200° C. The flame is then shut off and the material is allowed to cool to about 160° C., being stirred during this time. At this temperature the batch again becomes thin. This cooling process will require about four hours. The pot containing the batch is then subjected to a renewed heating for a period of from 8 to 10 hours, at the end of which time the material becomes very stiff and shows a yellow color. The heating during this period is more vigorous, a maxi-

mum temperature of approximately 300° being obtained. At this stage the heating is stopped, the contents of the pot are stirred, permitted to cool for from 4 to 6 hours and the pot is then opened and dumped. The average weight of the material dumped from a pot is about 825 pounds.

The product of four batches treated as indicated in the previous paragraph, and constituting about 3300 pounds, is then run into a tank containing 3200 gallons of cold water and is stirred for about one hour. The filtrate of a portion of this mixture will be found to have an acidity of .6 to .8 normal, using methyl-orange as an indicator. At the end of this period of stirring, a sample is taken from the tank, filtered, and tested to insure the absence of thorium in the filtrate. The contents of the tank are now decanted and filtered, preferably by means of a wooden filter press, and washed for three-quarters of an hour, or until the filtrate is no longer acid to the tongue. The filter cake thus obtained, which is designated as "the first gray mud" is a needle-like crystalline material, as viewed under a microscope and has an average weight of 1360 pounds. The thorium is contained therein as an insoluble phosphatic compound, probably thorium meta-phosphate sulfate, and is admixed with a certain percentage of rare earths and other impurities. The dried product contains about 75% thorium, calculated as thorium oxid.

The insoluble thorium phosphates of the gray mud are directly converted to insoluble thorium hydroxid as follows: 400 gallons of cold water is run into a 2000 gallon iron tank and 1000 pounds of caustic soda is added. The gray mud prepared as above described, and of the amount of 1360 pounds, representing the product of four pot-runs, is then dumped into the tank and the mixture is stirred and boiled by means of a closed steam coil for one hour. The tank is then filled with hot water, steamed to boiling and allowed to settle for two hours, at the end of which time about 800 gallons of clear liquid may be drawn off from the top of the tank. It is again filled with hot water and the material filtered through an iron press. The filter cake is thoroughly washed with hot water (65° C.) and then air-dried.