

# UNITED STATES PATENT OFFICE.

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## ART OF RECOVERING THORIUM.

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*To all whom it may concern:*

Be it known that I, OTTO N. BERNDT, 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 the Art of Recovering Thorium, of which the following is a specification.

The present invention relates to the art of recovering thorium from its natural mineral sources and more particularly from monazite sand.

In accordance with the present invention a concentration of the thorium content of the mineral, such as monazite sand, is effected by the use of fuming sulfuric acid. It is preferred that the action of the acid upon the monazite sands takes place under controlled conditions as to relative proportions, temperatures, and time, for the maximum efficiency and economy of the process. As a result of continued action of fuming sulfuric acid upon the sands an insoluble compound of thorium is formed whereas the compounds formed by the rare earth metals and by other constituents of the sands are largely soluble in water or dilute acid. On treatment of the mass resulting from the action of the fuming sulfuric acid upon monazite sand with water in suitable proportions, and separation of the soluble constituents of the mass, the insoluble residue will contain the thorium in greatly concentrated form.

The treatment of the monazite sand or other phosphatic compound of thorium with the fuming sulfuric acid is carried out in suitable pots, preferably provided with stirring apparatus and with the application of heat, which results in a gradual rise in temperature at a rate depending upon the heat applied. On attaining a temperature between 200° and 230° C. the temperature rises more rapidly to between 325 and 350° C., at which the completion of the reaction is indicated by a change of color of the reaction mass to a greenish yellow color. This mass, as above stated, contains substantially all of the thorium content of the sands in an insoluble form.

In carrying out the process of my invention I have found that a great economy of materials and of heat and a high efficiency of equipment may be attained by the use of fairly definite proportions of the reacting materials and by a suitable control of the

temperature of treatment. Thus, in practicing the invention I prefer the following procedure:

500 pounds of monazite sand, which has been concentrated by means of a magnetic separator to remove ilmenite and other magnetic constituents and contains about 9% thorium oxid, are treated with more than 425 pounds of fuming sulfuric acid stronger than 66° Bé., containing preferably about 5% excess sulfur trioxid. In general 475 pounds of such acid is a suitable amount. The mixture is heated for a period sufficient to decompose the sands and to convert the thorium into an insoluble compound, the heating preferably being at such a rate that a temperature of 200 to 230° C. is reached in between 3 to 4 hours. The rate of heating, however, may be such that this temperature is reached in from three to six hours. At this point the rise in temperature becomes more rapid, due probably to heat evolved by the reacting mass in substantial amounts, attaining a temperature of 300 to 350° C. in from one half to one hour. The reaction mass becomes greenish yellow in color and is in stiff plastic state, capable of being further stirred. At this point external heating is stopped and the mass is permitted to cool for several hours while being stirred and is removed from the baking pot or digester. The product is a thick pasty mass, greenish yellow in color, and weighing about 950 pounds, but should weigh not more than 1000 pounds and not less than 900 pounds.

The mass removed from the digesters is treated in suitable tanks with a sufficient quantity of water, preferably about 900 gallons for the above amount. A slight agitation of the solution which has a slight acidity will keep the insoluble thorium compound in suspension while the heavier silicious residues will settle to the bottom. The suspension of the thorium compound may be decanted and filtered, the thorium compound remaining as a residue on the filter in a greatly concentrated state and suitable for further purification.

It is readily apparent that the proportions of the reacting materials may be varied in accordance with the proportional amounts of thorium and impurities in the sand and the SO<sub>3</sub> (sulfur trioxid) content of the fuming acid. In the specific example above