UNITED STATES PATENT OFFICE.

OTTO N. BERNDT, OF CHICAGO ILLINOIS, ASSIGNOR TO LINDSAY LIGHT COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF ILLINOIS.

ART OF RECOVERING THORIUM FROM MONAZITE SANDS.

1,323,735. Specification of Letters Patent. Patented Dec. 2, 1919.

No Drawing. Application filed June 27, 1917. Serial No. 177,285.

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 from Monazite Sands, of which the following is a specification.

This invention relates to the art of recovering thorium from monazite sands and to a new thorium compound produced during such recovery.

It has heretofore been proposed to effect the recovery of thorium from such sands by a two-step process, involving, first the heating of the sand with excess of sulfuric acid, whereby the thorium, as well as the rare earths, and some other constituents thereof, were rendered soluble in dilute acids and subsequently effecting a selective precipitation from this solution by the addition of various reagents, by which means the thorium was thrown down in a more or less pure state.

It is found that by conducting the first stage of the process under the conditions which will later be described, it is possible to render the rare earths soluble in water or dilute acids leaving the thorium in the form of an insoluble compound, thus allowing by treatment of the product of the first stage of this process with water and filtration a simple and direct concentration of the thorium, without the necessity of first converting the thorium into a soluble form and then dissolving and precipitating it.

In general, the operation of the present process may be regarded as taking place as follows:

As is well known, monazite is essentially a phosphate of rare earths and thorium, in which the percentage of thorium varies as a rule from one to ten per cent. The rare earths are chiefly cerium, lanthanum and didymium. Monazite usually also contains small amounts of titanium, zirconium and iron.

The chemical changes occurring during the heating of monazite with concentrated sulfuric acid are probably complex on account of the large number of elements present in the monazite.

It seems probable that the first action of sufficient hot concentrated sulfuric acid on monazite gives rise to a mixture of normal or acid sulfates and phosphates of the bases present. If this product is treated with water, the larger part, including much of the thorium, will dissolve. But, if the product of the first action of hot concentrated sulfuric acid on monazite be further heated for several hours at about 280° to 300° C. water and sulfuric acid are driven off and the thorium compound is slowly converted into a phosphatic compound of thorium insoluble in water and dilute acids.

As a result of investigation of the nature of the reaction and the chemical composition of the product obtained, it is believed that the insoluble crystalline compound resulting from the process is a double salt of thorium containing metaphosphate and sulfate radicals and probably being of the formula, $Th(PO_3)_2SO_4$. Thus the results of an analysis of the compound obtained as compared with the calculated percentages of its components on the basis of the above formula, gives the following results:

	Found.	Calculated.
ThO_2	54%	54.25%
P_2O_5	28.5	29.21
SO_3	16.3	16.46

With regard to the mechanism of the process, it is believed that the first heating of the monazite sand with the concentrated sulfuric acid results in the formation of a sulfate of thorium, soluble in water. On raising the temperature, it is believed that there is a gradual rearrangement of the composition of the compound, with partial elimination of its sulfate content and substitution of the metaphosphate radical.

This compound has been made in substantially pure state in the following manner:

12 grams of thorium sulfate crystals $(Th(SO_4)_2.8H_2O$ were dissolved in 5 cc. of hot 80% syrupy orthophosphoric acid to a clear solution. This was heated several hours at a constant temperature of 280° C. During the heating water and fumes of sulfuric acid were evolved and at the end of ten hours heating the product was a nearly solid mass of minute, acicular white crystals. The mass was mixed with 15 parts by weight of 3% sulfuric acid and stirred two hours. Practically none of the thorium had dissolved in the acid solution which was separated from the white, insoluble residue by