

UNITED STATES PATENT OFFICE.

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ART OF MANUFACTURING ZIRCONIUM COMPOUNDS.

No Drawing.

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To all whom it may concern:

Be it known that I, LONNIE W. RYAN, 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 Manufacturing Zirconium Compounds, of which the following is a specification.

The present invention relates to improvements in the art of manufacturing zirconium compounds, and will be fully understood from the following description, in which certain specific processes exemplifying the present invention are set forth in detail.

In accordance with the present invention, a suitable zirconium ore, such as Brazilian zirkite, is decomposed by treatment with sulfuric acid. Although it has hitherto been known to decompose such silicious zirconium ores with upwards of three times their weight of sulfuric acid, in accordance with my invention, acid in amount not more than twice the weight of ore is employed. The sulfuric acid used is preferably 100% acid. 100 parts of zirkite are charged into a cast iron pot with, say, 150 parts of sulfuric acid and the mixture is heated to 200 and 250° C. and maintained at such temperature (preferably about 225° C.) for about ten hours, preferably with continuous stirring. At the end of this period, the temperature is raised to 350 to 400° C. and this temperature is maintained for about two hours additional. The material (pot-cake) is permitted to cool at least to some extent, and is then added to from 700 to 800 parts of water, with which it is stirred, and, if desired, may be heated to the boiling point. The liquid is then filtered, the residue amounting to about 40 parts. About two-thirds of the zirconium contents of the ore goes into the filtrate, the remainder, with about an equal part of silica, forming the bulk of the residue. The further treatment of this residuum will be described hereinafter.

The filtrate, which contains excess sulfuric acid, is then neutralized with any suitable alkaline material, preferably one which forms an insoluble sulfate. As the neutralizing medium, I prefer to employ such materials as lime, calcium hydroxide, barium hydroxide etc., as the reaction

products are substantially eliminated by precipitation from the zirconium-containing liquid. Too great an amount may not be employed, however, or part of the zirconium content of the liquid will be precipitated. The precipitate filtered out is a substantially pure sulfate, which is practically insoluble in the filtrate.

The zirconium may be immediately precipitated from the filtrate in the manner hereinafter set forth, although preferably the latter is subjected prior to precipitation to a reduction in order to convert any iron present into the ferrous condition. For example, to the sulfate, as above set forth, a sufficient quantity of a suitable reducing agent, for example, fourteen parts of sodium thiosulfate, may be added to effect this reduction.

In carrying out the precipitation in accordance with the present invention, the zirconium is thrown out in a granular form as a basic sulfate from an aqueous zirconium sulfate solution. In carrying out the precipitation, a suitable alkaline material is added to the solution until the zirconium is substantially completely precipitated as the basic sulfate, this addition of alkali taking place in the presence of certain auxiliary ions of strong electrical character, such as the strongly electro-positive potassium ion or the strongly electro-negative chloride ion.

In carrying out the precipitation, the auxiliary ion which is provided in accordance with the present invention and which causes the formation of a granular precipitate, may be added in proper proportion prior to the addition of the alkali which effects the precipitation of the zirconium, or it may be added simultaneously therewith. For example, in case of the use of a potassium compound, there may be added to the solution of the zirconium compound a suitable potassium salt, such as potassium sulfate, and this addition may be followed by an alkali such as sodium carbonate or caustic soda, the latter being added until the zirconium is substantially completely precipitated. The amount of the potassium compound employed should be sufficient to provide at least one-twentieth ($\frac{1}{20}$) mole for each mole of zirconium oxide (ZrO_2). Preferably sufficient to provide about one-eighth