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PURIFICATION OF METAL POWDERS.

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This invention relates to a method for purifying materials with a new type of solvent and more particularly it relates to a method for the purification of metals which are obtained in the powdered state by reduction and separation and are contaminated by impurities during these processes.

An object of this invention is the provision of a new type of solvent.

Another object of this invention is the provision of a method whereby metal powders may be obtained in a high state of purity.

A further object of the invention is the provision of a method whereby metallic powders may be produced free from metallic hydroxides, oxides or other impurities.

A special object of the invention is the provision of a method whereby uranium may be obtained in a state of purity sufficient to allow its working into a coherent mass.

Other objects will be apparent from a further reading of the specification.

It is known that there are many processes practiced for obtaining metals from their compounds by which the metals are obtained in a powdered condition contaminated with certain impurities such as the by-products of the reaction and with hydroxides and oxides resulting from the process which is practiced to isolate the metallic powder from its by-products and resulting also from exposure to the atmosphere. The formation of hydroxides and, subsequently, of oxides is partly due to the fact that the isolation process, usually known as washing, is done with an aqueous solution, whereby at least part of the metal powder is converted into hydroxide, which, upon heating, is converted into an oxide. The formation of these impurities is due to the fact that particles of metal powder have such large surface areas in proportion to their volume that they are very active chemically and therefore combine readily with impurities such, for example, as oxygen and hydrogen or combinations of the same.

By following the process outlined in this invention, metals prepared by a process which yields a metallic powder in an impure condition may be obtained in a high state of purity. It is therefore apparent that the present invention is very valuable when isolating easily-oxidizable metals such, for example, as uranium, thorium and zirconium, from the by-products of the reaction which effects their reduction. It has been found that this invention, as hereinafter described, is especially valuable and even necessary when it is desired to work easily-oxidizable metals from the powdered condition into a coherent mass.

According to my invention, metals may be obtained in a pure state and may be prevented from accumulating impurities by washing the reaction products with a solution comprising a non-aqueous liquid in which is dissolved a non-aqueous or anhydrous solvent of the impurities or by-products which contaminate the metal desired. Preferably, the non-aqueous liquid is an alcohol and the solvent is an acid and, more particularly, the alcohol may be methyl alcohol and the acid may be hydrogen chloride, although, in general, the non-aqueous liquid may be any liquid in which may be dissolved an anhydrous solvent which is necessary to dissolve the impurities which are admixed or combined with the metal desired. It will be appreciated that the use of a non-aqueous medium will avoid the hydration or oxidation of the metal being purified when carrying out the process of isolating the metal from the impurities admixed or combined with it.

Inasmuch as the present invention is particularly useful for the production of easily-oxidizable metals in a state of purity, I will illustrate the procedure employed in washing uranium, which is metal of the character mentioned.

In the reduction of uranium oxide by means of calcium chloride and sodium, as performed by heating these compounds in a closed bomb, the resultant products are largely uranium metal in powdered form, sodium chloride and lime. When the sodium chloride and the lime are washed out with water and dilute acid, which has been the usual practice, a brownish-black product results, the color indicating the presence of a considerable percentage of uranium oxide. This oxide, when agglomerated, will not conduct the electric current and, therefore, will not give satisfactory results if an attempt is made to sinter or fuse the same by high-frequency induction currents in a vacuum furnace, which has been found to be the preferred method for changing this metal from the powdered or agglomerated

state into a coherent mass. By practicing the present process, however, the oxide may be prevented or removed so that the metal powder, when agglomerated, may be sintered or fused by induction currents and, furthermore, contains very little hydroxide and occluded gases so that no trouble is experienced in eliminating these impurities when the pressed body of powder is treated in a vacuum furnace for the purpose of producing a coherent mass.

The procedure of washing the uranium powder, after the latter has been reduced from its oxide in a heated bomb or other closed container by means of calcium chloride and sodium, may be effected by washing the reaction products with a dilute acid suitable for the solution of calcium oxide, sodium chloride and other impurities without substantial action on the uranium as metal, subsequently decanting the supernatant liquid after the undissolved material has settled and finally filtering the undesired material. During the filtration process, the material on the filter, which is largely metallic uranium, should be kept covered with alcohol, thoroughly washed with the same and may be finally washed with ether.

This step in the process, however, which employs an aqueous solution in which is dissolved a solvent material, is not a necessary step in the purification of the powder, as my process, as hereinafter described, is sufficient in itself to free the metal from impurities. In other words, the reaction products resulting from the reduction of metal in the bomb may be completely purified by means of a non-aqueous liquid in which is dissolved an anhydrous solvent for the impurities which are to be separated from the metal desired.

In order to further purify the uranium according to my invention, the metal may be washed with a solution of dry hydrogen chloride in practically anhydrous alcohol. This solution may be made by slowly dropping concentrated hydrochloric acid into concentrated sulphuric acid, passing the hydrogen chloride thus liberated through drying agents such, for example, as calcium chloride, and subsequently passing the dried hydrogen chloride into practically anhydrous, say 99%, alcohol. In effecting the washing process with the solution thus made, the uranium powder may first be placed in a shallow dish and covered with practically pure methyl alcohol, 99% purity being sufficient for this purpose. A solution of hydrogen chloride in alcohol may then be added gradually and the mixture thoroughly agitated. The addition of the acid-alcohol mixture may be continued until a small amount of hydrogen is evolved. The evolution of the hydrogen as bubbles arising from the surface of the alcohol is an indication that the acid has been made as strong as possible without causing too much of the uranium to go into solution. The addition of the acid-alcohol solution should be stopped at this point.

The resulting mixture comprising uranium powder and the acid-alcohol wash is filtered from the liquid which is in contact therewith, the uranium being kept covered by means of alcohol during filtration. While still wet with alcohol, the uranium powder may be scraped or brushed from the filter into a closed container. In introducing uranium into such a container, which may be a mold, care must be exercised to have it still moist with alcohol so as to protect it from oxidation but dry enough so that, when subsequently exposed to pressure in the mold, the metal may not be forced out through the fissure formed by the junction of the two elements of the mold which will now be described.

The mold may be a closed mold consisting of two members having, for example, semi-cylindrical grooves cut in one face of each of the members, these members being so disposed in relation to each other that the two semi-cylindrical grooves form a cylindrical hole, the two members being bolted together and having the ends of the cylindrical hole closed by two plungers, at least one of which extends from one end of the mold, after the uranium powder has been placed in the hole thereof. The latter may then be placed in a press and pressure applied to the plungers, whereby the uranium powder may be agglomerated by gradually increasing pressure until the particles thereof cohere sufficiently so that the agglomerated mass of uranium, when withdrawn from the mold, may not disintegrate with careful handling. The mass of uranium may then be treated, if desired, by sintering or fusing the same, so as to preserve it in a form in which it is not subjected to the action of the atmosphere to the extent that it will be totally converted into the oxide.

While I have described in detail a process for preparing one metal, modifications may be necessary for the proper preparation of other metals but these modifications, such, for example, as the concentration of the acid used, the duration of time necessary for the purification of the powder, etc. may be readily devised by one skilled in the art.

It should be understood that my invention embraces not only the method for the purification of metal powders but of other materials and it also includes the discovery of a new solvent which comprises a non-aqueous liquid containing a dissolved substance, the combination being regarded as a new kind of solution or reagent for solvent purposes. It will, of course, be understood

that a plurality of non-aqueous solutions may be combined in which one or more substances may be dissolved, all of which, taken together, may find valuable uses in the chemical art. Furthermore, it should be understood that the non-aqueous liquid itself, as well as the dissolved substance, may be a solvent and may be used as such.

What is claimed is:

1. The method of purifying a readily oxidizable metallic powder such as thorium, uranium, etc. which comprises treating said powder with a non-aqueous liquid which does not react with the oxides of said powder and which is capable of preventing further oxidation of said powders, and combining with said liquid an acid which will interact with the impurities with which said powder is contaminated to form compounds which are readily soluble in said liquid.

2. The method of purifying a rare metal powder which consists in protecting said powder from oxidation by a non-aqueous liquid which is incapable of reacting with said powder or any impurity therein, said liquid having dissolved therein, an acid which is capable of reacting with said impurities and which will not appreciably react with said metal powders to form compounds which are soluble in said liquid.

3. The method of purifying a rare metal powder such as uranium which consists in washing the same with alcohol in which is dissolved an acid capable of reacting with the impurities in said powder to form compounds which are soluble in said alcohol.

4. The method of purifying a rare metal powder which consists in washing the same with alcohol in which hydrogen chloride is dissolved.

5. The method of purifying rare metal powders, which comprises washing the same with methyl alcohol in which hydrogen chloride is dissolved.

6. The method of purifying uranium powder which consists in washing it with a non-aqueous liquid in which is dissolved a solvent for the impurities with which said metal powder is contaminated.

7. The method of purifying uranium powder which consists in washing it with a non-aqueous liquid in which is dissolved an acid for the solution of the impurities which contaminate said metal powder.

8. The method of purifying uranium powder which consists in washing it with alcohol in which is dissolved an acid by means of which the impurities in said metal powder may be dissolved.

9. The method of purifying uranium powder which consists in washing it with an alcohol in which is dissolved hydrogen chloride.

10. The method of purifying uranium powder which consists in washing it with methyl alcohol in which is dissolved hydrogen chloride.

In testimony whereof, I have hereunto subscribed my name this 19th day of August, 1922.

JOHN WESLEY MARDEN.