UNITED STATES PATENT OFFICE.

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PROCESS OF OBTAINING TUNGSTATES.

1,255,144. Specification of Letters Patent. Patented Feb. 5, 1918.

No Drawing. Application filed June 28, 1916. Serial No. 106,358.

To all whom it may concern:

Be it known that we, JOHN B. EKELEY and WILLIAM B. STODDARD, of Boulder, in the county of Boulder, and in the State of Colorado, have invented a certain new and useful Improvement in Processes of Obtaining Tungstates, and do hereby declare that the following is a full, clear, and exact description thereof.

Our invention relates particularly to the treatment of ores containing heavy acid forming elements but particularly tungsten, so as to separate the impurities from the same and obtain the tungsten in the form of a salt of tungstic acid. It relates however also in a similar way to the treatment of ores containing chromium, molybdenum, vanadium, or uranium, which are also heavy acid-forming elements.

The object of our invention is to obtain tungstic acid or a salt thereof, or a similar compound of any of the other heavy acid-forming elements, by the treatment of various ores containing the same in such a manner as to separate out in a very complete manner the impurities contained in the ores and so as to obtain a compound of such element, as for example, a salt of tungstic acid, in a very pure form. A further object is to carry out this process by the aid of fusion in such a manner that the impurities contained in the ore will become separated from the compound of the heavy acid-forming element by sharp lines of demarcation in the fused bath and in the resulting solid obtained by the cooling of the fused materials. Further objects of our invention will appear from the detailed description thereof appearing hereinafter.

While our invention is capable of being carried out in many different ways, for the purpose of illustration we shall describe only one example of the several ways in which our invention may be followed.

As one example of the way in which our invention may be carried out we may proceed as follows: A tungsten ore, as for example, wolframite, hübnerite, ferberite, scheelite, tungstite, etc., is crushed, sampled, and ground while dry in any suitable manner, by any one of the well-known methods, to a suitable degree of fineness. The degree of fineness may vary to some extent with the nature of the ore, as for example, the facility with which it subjects itself to the succeeding treatments in the process. The ground ore is then roasted in any suitable furnace so as to drive off the sulfur and arsenic contained in the ore as far as this is possible. The roasted ore is then mixed with sodium chlorid, sodium carbonate, and if found desirable, some silica. An amount of sodium chlorid is added, preferably from two-fifths to the same in weight as the weight of the ore. An amount of the sodium carbonate is added sufficient to combine with all of the tungstic acid and leave in addition an excess of the sodium carbonate. Sufficient silica is ordinarily added to combine with the excess of sodium carbonate so as to produce an insoluble silicate preferably containing sodium oxid and silicon dioxid in the proportions of one to four. Some ores, however, do not require the addition of any silica for this purpose, and in working with such ores the silica is omitted. If desired, sodium nitrate or sodium chlorate may be added to the mixture which provide oxidizing agents in the bath when fused. Instead of sodium chlorid, we may use sodium fluorid or calcium chlorid or fluorid. The charge is then fused in a suitable furnace. After the bath of fused materials has been allowed to cool, it will be found that the solid materials will have become separated into a water soluble portion and a water insoluble portion, the tungstic acid being contained in the form of sodium tungstate in the water soluble portion. The solid mass is crushed and then extracted with water and filtered. The filtrate is a clear solution of sodium tungstate which is precipitated as calcium tungstate by the addition of calcium chlorid in a sufficient quantity to replace all of the sodium by calcium in the sodium tungstate contained in the solution.

The sodium chlorid performs the function of producing sharp lines of demarcation between the two portions contained in the fused bath and in the resulting solid when cooled, the said two portions comprising a water soluble portion containing sodium tungstate, sodium carbonate and sodium chlorid, and a water insoluble portion comprising silicates and other materials derived from the gangue of the ores treated. The sodium chlorid, furthermore, acts to disintegrate and dissolve the tungsten ore with remarkable rapidity. The sodium chlorid also acts as a solvent and diluent for the sodium carbonate. It also plays a part in

almost entirely preventing the formation of soluble silicates, phosphates, etc. The sodium chlorid also produces a crystalline structure with marked cleavage to the water soluble portion of the melt when solidified so that it is very friable and very easily crushed. When sodium chlorid is used, the portion of the melt containing the sodium tungstate, carries comparatively little undecomposed sodium carbonate, but large quantities of sodium chlorid, which makes it very easy to dissolve the water soluble portion in water as compared with a corresponding melt made with sodium carbonate alone. Furthermore, the presence of sodium chlorid and sodium carbonate permits the mass to be more readily melted than when the sodium chlorid is omitted, and the melted mass has a low viscosity so that the reacting fluid comes into contact with the particles of ore very readily. The extreme fluidity of the sodium carbonate and sodium chlorid when fused together renders it possible also to use less of the sodium carbonate than when the sodium chlorid is omitted. For the same reason it is possible to treat a coarser material in a shorter time than if the sodium chlorid were omitted. The sodium carbonate is provided for combining with the tungstic acid so as to produce sodium tungstate, and in the presence of the sodium chlorid the sodium carbonate also exerts a selective action on the tungstic acid in the ore by causing the sodium oxid contained in the same to combine with the tungstic acid in preference to the silica or phosphorus pentoxid, or other acid ingredients of the ore. Instead of treating a tungsten ore as above, an ore of another heavy acid-forming element may be similarly treated, as for example, an ore of chromium, molybdenum, vanadium or uranium, as for example, chromite, molybdenite or carnotite.

By the term "fusing" herein, and in the claims, we mean maintaining in a state of fusion and not necessarily passing from the solid to the liquid state. In other words, by the term "fusing" in the claims, we mean fusing one or more of the ingredients separately and mixing them together while fused or heating all of the ingredients together until fused.

While we have described our invention above in detail, we wish it to be understood that many changes may be made therein without departing from the spirit of our invention.

We claim:

1. The process which comprises fusing a tungsten ore in a bath containing an alkali and a compound of an alkali-forming element, that is a solvent of the alkali when fused.

2. The process which comprises fusing a tungsten ore in a bath containing sodium carbonate and a compound of an alkali-forming element, that is a solvent of the sodium carbonate when fused.

3. The process which comprises fusing a tungsten ore in a bath containing a halid of an alkali-forming metal, and a basic compound of an alkali-forming metal.

4. The process which comprises fusing a tungsten ore in a bath containing chlorid of sodium, and a basic compound of an alkali-forming metal.

5. The process which comprises fusing a tungsten ore in a bath containing a halid of an alkali-forming metal and sodium carbonate.

6. The process which comprises fusing a tungsten ore in a bath containing chlorid of sodium and sodium carbonate.

7. The process which comprises fusing a tungsten ore in a bath containing a halid of an alkali forming metal and an alkali and silica.

8. The process which comprises fusing a tungsten ore in a bath containing chlorid of sodium, an alkali and silica.

9. The process which comprises fusing a tungsten ore in a bath containing a halid of an alkali forming metal, sodium carbonate and silica.

10. The process which comprises fusing a tungsten ore in a bath containing chlorid of sodium, sodium carbonate and silica.

11. The process which comprises fusing a tungsten ore in a bath containing a halid of an alkali-forming metal, an alkali and an oxidizing salt.

12. The process which comprises fusing a tungsten ore in a bath containing a halid of an alkali-forming metal, an alkali and sodium nitrate.

13. The process which comprises grinding and roasting a tungsten ore, fusing the tungsten ore in a bath containing a halid of an alkali-forming metal, an alkali and an oxidizing salt.

14. The process which comprises grinding and roasting a tungsten ore, fusing the tungsten ore in a bath containing a halid of an alkali-forming metal, an alkali and sodium nitrate.

In testimony that we claim the foregoing we have hereunto set our hands.

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