## United States Patent Office.

GEORGE T. HOLLOWAY AND HARRY W. LAKE, OF LONDON, ENGLAND.

## PROCESS OF MAKING TUNGSTATES.

SPECIFICATION forming part of Letters Patent No. 667,705, dated February 12, 1901.

Application filed December 30, 1899. Serial No. 742,131. (No specimens.)

To all whom it may concern:

Be it known that we, GEORGE THOMAS HOL-LOWAY, residing at 57 Chancery Lane, and HARRY WILLIAM LAKE, residing at 3 Great 5 Winchester street, London, England, subjects of the Queen of Great Britain and Ireland, have invented certain new and useful Improvements in Processes of Preparing Tungstates and other Tungsten Compounds, (for 10 which we have filed an application for Letters Patent in Great Britain, No. 11,907, dated June 8, 1899,) of which the following is a specification.

Our invention relates to the preparation of 15 tungstate of sodium or tungstate of potassium.

Our invention consists in forming sodium or potassium tungstate by fusion of an ore of tungsten with a silicious flux, to which is added or which is combined with or contains 20 a salt of oxid of sodium or potassium, the silicious flux being in the bath in excess of the quantity required to combine with all the iron and manganese contained in the one under treatment, so that pure tungstate of 25 soda may be produced, which will not require further purification.

In carrying our invention into effect we fuse in a crucible or reverberatory or other furnace a mixture of wolfram or other suit-30 able tungsten ore with a flux consisting of sodium silicate. In place of the sodium silicate a suitable mixture, consisting of silica (in the form of sand, powdered quartz, flints, or other variety of silica) and carbonate, sulfate 35 or chlorid of sodium may be employed, so as to produce sodium silicate when fused in the furnace. The sodium silicate or the mixture forming the silicate may be replaced by other fusible silicates, such as powdered glass; but to we prefer to employ the variety of sodium silicate known as "water-glass," which can be used as such or can be prepared first in the ordinary way in the crucible or furnace in which the preparation of the sodium tung-15 state is to take place. The tungsten ore, preferably in the form of powder, is mixed with the sodium silicate or either of the silicates above mentioned or with the mixture which forms sodium silicate and charged into the 50 crucible or furnace, which is heated to a high temperature, so as to thoroughly fuse the silicious flux. The quantity of ore in each |

charge should be such that the silica present in the flux will be in excess of that required to combine with the iron and manganese. We 55 prefer that the flux be first fused and the ore added to the molten flux. If the ore is not sufficiently finely divided, it may be necessary to stir the fused mass from time to time, so as to insure complete reaction of the flux 60 with the ore. During the reaction the oxids of manganese and iron in the wolfram ore dissolve in the silicious flux, while the tungstic anhydrid, (known as "tungstic acid,") combined with the said oxids in the ore, unites 65 with the sodium oxid, which exists in the flux as sodium silicate, or which is added in the form of a sodium compound, as hereinafter described, and forms sodium tungstate. The sodium tungstate being heavier than the flux 70 sinks to the bottom of the crucible or furnace and may be drawn off or otherwise removed, as desired. As this treatment removes the sodium oxid from the flux and as the flux may be used continuously until it has dis- 75 solved as much of the oxids of iron and manganese as it will take up or has otherwise lost its efficiency, it is necessary to replenish from time to time the charge in the furnace with a sodium compound capable of forming 80 sodium tungstate. The sodium compound is added in the form of sodium carbonate or other suitable compound of sodium with the charge of ore, or a quantity of sodium carbonate or other suitable sodium compound 85 may be added initially with the charge of sodium silicate and ore. The proportion of sodium compound should be such as will prevent the flux from being so difficultly fusible as to prevent the sodium tungstate or the ore 90 from sinking through it. When the silicate flux employed contains no sodium or an insufficient quantity to form sodium tungstate with the ore, the addition of this sodium compound is of course essential.

By varying the amount of sodium present in the flux or added with the charge we may produce either the ordinary soluble tungstate, (Na<sub>2</sub>WO<sub>4</sub>,) which may be sold as such without further treatment, or may be crystallized from 100 a solution in water, or we may produce a variety of sodium tungstate which contains more tungsten and less sodium than the ordinary tungstate and which is far less soluble.

Potassium tungstate is prepared in a similar manner to the sodium salt, the only change in the process being the use of a potassium compound in the silicious flux, the silicious flux being composed of potassium silicate.

Having now particularly described and ascertained the nature of our said invention, we declare that what we claim is—

The process for the extraction of tungsten compounds from tungsten ores, consisting in adding the said ore, in a finely-divided state, to a fused bath of silicate of an alkaline metal,

capable of combining freely with the iron and manganese or lime in the ore, whereby practically pure tungstate of the alkaline metal is produced at one operation, substantially as described.

In witness whereof we have hereunto set our hands in presence of two witnesses.

GEORGE T. HOLLOWAY. HARRY W. LAKE.

Witnesses:

DUGALD CLERK, ALBERT E. PARKER.