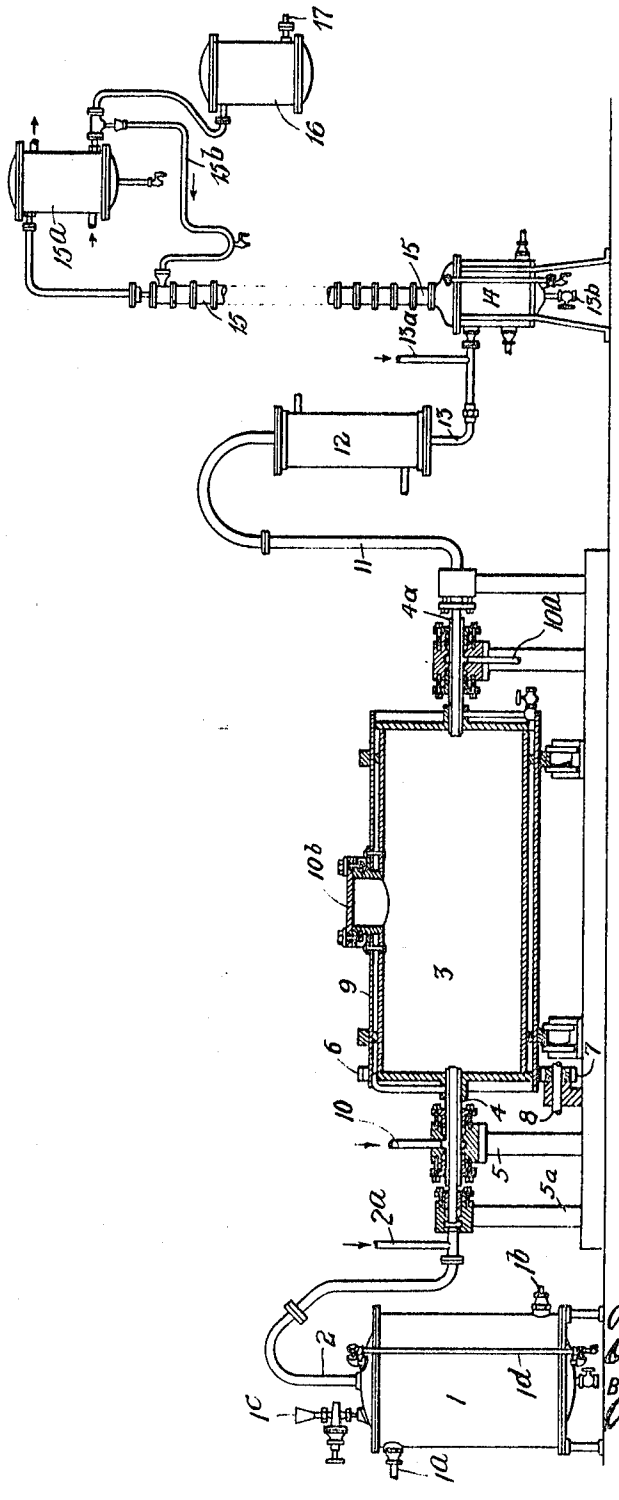
R. P. CALVERT AND O. L. THOMAS.

PROCESS OF OBTAINING BORIC ACID FROM MIXTURES CONTAINING BORATES.

APPLICATION FILED NOV. 5, 1917.

1,308,577. Patented July 1, 1919.



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PROCESS OF OBTAINING BORIC ACID FROM MIXTURES CONTAINING BORATES.

1,308,577. Specification of Letters Patent. Patented July 1, 1919.

Application filed November 5, 1917. Serial No. 200,439.

To all whom it may concern:

Be it known that we, ROBERT P. CALVERT and OTHO L. THOMAS, of Wilmington, in the county of New Castle, and in the State of Delaware, have invented a certain new and useful Improvement in Processes of Obtaining Boric Acid from Mixtures Containing Borates, and do hereby declare that the following is a full, clear, and exact description thereof.

Our invention relates to a process of obtaining boric acid from mixtures containing borates, and relates particularly to a process in which boric acid is combined with an alcohol and the resulting alkyl borate is volatilized.

The object of our invention is to provide a process for obtaining boric acid from mixtures containing borates, and particularly mixtures of soluble salts containing the same, so that said salts will be freed from the borates and the borates may be used in any desired manner for the production of compounds of boron.

Further objects of our invention will appear from the detailed description thereof contained hereinafter.

While our invention is capable of being carried out in many different ways, we shall describe herein only certain ways of carrying out the same, and while it is capable of being used in connection with many different types of apparatus, we shall describe only one type of apparatus for use in connection therewith and in the accompanying drawings, in which—

The figure is a diagrammatic representation of an apparatus which may be used in connection with our invention.

In the drawings, we have shown a jacketed still 1, having a steam inlet 1^a and a steam outlet 1^b for heating the still. The still is further provided with a valved filling pipe 1^c for methyl alcohol, a gage glass 1^d and a vapor outlet pipe 2 which has an inlet pipe 2^a for air or steam, said pipe 2 being arranged to deliver the vapor therefrom into a rotary drum 3, having hollow trunnions 4 and 4^a, carried in suitable supports 5 and 5^a. The drum 3 is adapted to be rotated by means of peripheral gears 6, meshing with a gear 7 on a shaft 8, passing through the supports 5, and adapted to be driven from any suitable source of power. The drum 3 has a heating jacket 9, adapted to be supplied with steam through the hollow trunnion 4, which connects with a steam supply pipe 10, while the condensed steam is carried off through a pipe 10^a. A manhole 10^b in the drum 3 permits the entry and exit of the solid materials for the same. The vapors from the drum 3 pass out through a pipe 11, which is connected to a condenser 12, having an outlet pipe 13, leading to a fractionating still 14. The still 14 has a water inlet pipe 13^a, connected to the pipe 13, a draw-off pipe 13^b, and a vapor outlet pipe 15 leading to a primary condenser 15^a having a trap return pipe 15^b leading to the vapor outlet pipe 15, and thence to a condenser 16, from which the condensed materials are drawn off through an outlet pipe 17.

For example, our invention may be carried out as follows: A quantity of a material, which may be a mixture of a number of compounds, for example 70% of potassium chlorid, 12% of sodium chlorid, 1% of sodium carbonate, 2% of sodium sulfate, 11% of borax and 4% of water, is mixed with a quantity of hydrochloric acid, or some other acid, such for example as sulfuric acid, in the drum 3. For this purpose, 100 parts of said material may be mixed with 5.6 parts of hydrochloric acid having a strength of 23° Bé. The mixture is slightly warmed, while the drum 3 is rotated, in the presence of an alcohol, for example methyl alcohol, which has been distilled over by introducing 40 parts of methyl alcohol into the still 1 and applying heat thereto. This converts the borates into boric acid, and the methyl alcohol combines with the boric acid to give methyl borate, which is thereafter volatilized in the drum 3 and distilled off, together with the remaining methyl alcohol and unchanged boric acid, and condensed in the condenser 12, and subsequently conveyed into the still 14.

If desired, the removal of the volatile materials from the drum 3 may be assisted by

the injection of a current of air or steam by means of the pipe 2^a.

Thereupon, 8 parts of water are introduced to the still 14 through the pipe 13^a, in order to hydrolyze the methyl borate and liberate methyl alcohol and boric acid, after which the methyl alcohol, together with any undecomposed methyl borate, is distilled off through the pipe 15 into the condenser 16 by the application of heat to the still 14, so that the distillate may be recovered from the pipe 17. The aqueous solution of the boric acid in the still 14 may be drawn off by the pipe 13^b and allowed to cool, so as to deposit the boric acid crystals. The amount of water added to the still 14 is usually in the proportion of 25 gallons of water to 100 gallons of the distillate contained therein, although the amount of water may be varied somewhat according to the concentration of the boron compounds in said distillate.

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 removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, and volatilizing the compound formed by the alcohol and the boric acid.

2. The process which comprises removing the borate from a mixture of sodium and potassium salts containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, and volatilizing the compound formed by the alcohol and the boric acid.

3. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding methyl alcohol thereto, and volatilizing the compound formed by the alcohol and the boric acid.

4. The process which comprises removing the borate from a mixture of sodium and potassium salts containing the same by liberating the boric acid from the borate, adding methyl alcohol thereto, and volatilizing the compound formed by the alcohol and the boric acid.

5. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, and hydrolyzing the distillate.

6. The process which comprises removing the borate from a mixture of sodium and potassium salts containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, and hydrolyzing the distillate.

7. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding methyl alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, and hydrolyzing the distillate.

8. The process which comprises removing the borate from a mixture of sodium and potassium salts containing the same by liberating the boric acid from the borate, adding methyl alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, and hydrolyzing the distillate.

9. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, hydrolyzing the distillate, and distilling off the alcohol.

10. The process which comprises removing the borate from a mixture of sodium and potassium salts containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, hydrolyzing the distillate, and distilling off the alcohol.

11. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding methyl alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, hydrolyzing the distillate, and distilling off the alcohol.

12. The process which comprises removing the borate from a mixture of sodium and potassium salts containing the same by liberating the boric acid from the borate, adding methyl alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, hydrolyzing the distillate, and distilling off the alcohol.

13. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate, adding a lower monohydric alcohol thereto, volatilizing the compound formed by the alcohol and the boric acid, condensing the vapors thus produced, hydrolyzing the distillate, distilling off the alcohol, and crystallizing out the boric acid from the residue.

14. The process which comprises removing the borate from a mixture containing the same by liberating the boric acid from the borate by means of the addition of another acid and adding a lower monohydric alcohol thereto, and volatilizing the compound formed by the alcohol and boric acid.

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

ROBERT P. CALVERT.
OTHO L. THOMAS.

Witnesses:
G. D. HOPKINS,
S. L. ABRAMS.

Copies of this patent may be obtained for five cents each, by addressing the "Commissioner of Patents, Washington, D. C."