PROCESS FOR PREPARING STABLE PEROXIDE-CONTAINING BORATES

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In the preparation of peroxide-containing borates it has for a long time been the endeavor to make such products which have a comparatively high content in peroxide— which can also be expressed by the content of active oxygen—and which are particularly stable. The object has mainly been carried out by adding stabilizers to the perborates.

More recently the discovery has been utilized that products with high alkali content are unstable and therefore borates were made containing peroxide in which the ratio B$_2$O$_3$:Na$_2$O is about 1:0.5-0.9.

It has now been found quite unexpectedly that with a suitable method of preparation, stable peroxide-containing borates can be made in which the molar ratio

\[ B_2O_3:Na_2O = 1:1.1-2 \]

and which have a content in active oxygen of more than 15%. The preparation is done by making a solution from the conventional components, e.g. boric acid, NaOH, and hydrogen peroxide in the appropriate molar ratio; instead of the components named, equivalent amounts of similar starting materials may be used for making the solution which is prepared at low temperature and which is then passed over surfaces heated to 100–130°C.

Instead of using boric acid, NaOH and H$_2$O$_2$ the equivalent amount of, for instance, the several borates (such as metabolate, borax) may be used with NaOH and H$_2$O$_2$, instead of the components named it is also possible to use Na$_2$B$_4$O$_7$ and H$_2$O$_2$ either with boric acid and NaOH or with borate. The amount of H$_2$O$_2$ is preferably so taken that about 1.7–2.4 mol are calculated for 1 mol of Na$_2$O. In preparing the solutions from the components it is advantageous not to exceed temperatures of 26–30°C at the utmost. It has also proven advantageous to use heated rollers for the subsequent heat treatment of the solution. In this manner products are obtained having a content in active oxygen between 15 and 20%; these products have high stability without the addition of any stabilizer.

The products so obtained, which are by the way not crystalline, show a very much improved solubility amounting to many times the solubility of conventional perborate.

In spite of a similar analytical composition, the products according to the invention are therefore essentially different in their properties from mixtures of sodium perborate and Na$_2$O$_2$obtained by simple mixture. The products made according to the invention may be used in bleaching, laundering and cleansing agents per se or in admixture with known additives.

In some cases it is desirable to make the addition of substances which are useful for even liberation of oxygen in laundering and bleaching agents, already in the preparation of peroxide-containing borates. By processing in this manner, a better and more thorough mixing action is frequently obtained.

As such additions I may mention e.g. magnesium salts, more particularly magnesium silicate, waterglass, or mixtures thereof. These compounds are also known as stabilizers, in order to prevent peroxide decomposition during storage. However, an addition is not necessary for stabilizing purposes in the peroxide-containing borates when made according to the present invention, when the products are not intended for long-time storage.

The process according to the invention will now be more fully described in a number of examples, but it should be understood that these are given by way of illustration and not of limitation and that many changes can be made in the details without departing from the spirit of the invention.

Example 1

3760 g. boric acid and 3760 g. NaOH (molar ratio B$_2$O$_3$:Na$_2$O=1:1.55) are mixed in dry state and into the mixture 8.67 liters of 40% H$_2$O$_2$ are poured at a temperature of maximum 25°C, while stirring vigorously. After the H$_2$O$_2$ is all entered, a clear solution is formed which is sprayed in a thin layer on a roller drier and is evaporated to dryness at a temperature of 100–110°C.

Yield: 8490 g. dry powder with 18.9% active oxygen.

Analysis: 34.2% Na$_2$O; 24.9% B$_2$O$_3$; 40.0% H$_2$O$_2$; pH value in 1% solution=10.99.

Solubility 48 g. in 100 cc. H$_2$O at 35°C.

After 3 months storage at 25°C, the active oxygen had dropped to 18.75%.

Example 2

4270 g. boric acid are mixed with 4200 g. caustic soda (molar ratio B$_2$O$_3$:Na$_2$O=1:1.52) at 25°C and into the mixture 8.22 liters of 40% H$_2$O$_2$ are poured, whereas the solution is processed according to Example 1 on a roller drier at 110°C.

Yield: 9000 g. dry, amorphous powder with 17.4% active oxygen.

Analysis: 36.1% Na$_2$O; 26.6% B$_2$O$_3$; 37.0% H$_2$O$_2$; pH value in 1% solution=11.20.

Active oxygen after three months=17.30.

Example 3

2920 g. boric acid and 4300 g. caustic soda (molar ratio B$_2$O$_3$:Na$_2$O=1:1.63) are dissolved in 7.76 liters of 40% H$_2$O$_2$ and evaporated to dryness on a roller drier at 105°C.

Yield: 8630 g. with 16.9% active oxygen.

Analysis: 38.5% Na$_2$O; 25.6% B$_2$O$_3$; 35.9% H$_2$O$_2$; pH value in 1% solution=11.3.

Solubility 34 g. in 100 cc. H$_2$O at 35°C.

Oxygen content after 3 months at 25°C=16.75.

Example 4

4050 g. boric acid are mixed with 4000 g. caustic soda (molar ratio B$_2$O$_3$:Na$_2$O=1:1.54) and are slowly poured while stirring in 3000 cc. water. The temperature rises to 100°C, while a clear slightly syrupy solution is formed. The solution is cooled down to 70°C, care being taken that a premature solidification is avoided. The solution is then passed into 8.2 liters of 40% H$_2$O$_2$ cooled to 0–10°C. To the clear solution, 200 g. of magnesium silicate powder are added and the resulting product is hereafter quickly evaporated on a roller drier at 115°C.

8500 g. of a borate containing active oxygen are obtained, the active oxygen content (17.6%) being practically unchanged after 12 months.

Analysis: 35.9% Na$_2$O; 26.0% B$_2$O$_3$; 37.7% H$_2$O$_2$; pH value in 1% solution=11.30.

Example 5

1965 g. boric acid and 2155 g. caustic soda (molar ratio B$_2$O$_3$:Na$_2$O=1:71) are stirred with 1.5 liters water. The solution is slowly poured into 3.88 liters of 40% H$_2$O$_2$ at 10–20°C. To this solution are finally added 430 g. waterglass of 40° Bé. (8.83% Na$_2$O, 23.55% SiO$_2$, 61.82% H$_2$O) and is further processed as described in Example 4.
Yield: 4480 g. with 16.3% active oxygen.
Analysis: 37.3% Na₂O; 24.3% B₂O₃; 34.6% H₂O₂.
PH value=11.3.
Oxygen after 2 months=16.20.

What I claim is:

1. A process for preparing stable solid peroxide-containing borates, which comprises making an aqueous solution of a boron compound, selected from the group consisting of boric acid, borax and boronates, NaOH and H₂O₂ at temperatures of 0-30°C., in a molar ratio of B₂O₃, calculated on the boron compound present, to Na₂O of approximately 1.7-2.0:1, and conducting said solution over heated surfaces at a temperature ranging from 100-130°C., whereby a dry borate having an active oxygen content of more than 15 percent is obtained which will not decrease during storage, said borate being an effective laundering, cleansing and bleaching agent usable by itself without addition of other agents.

2. A process according to claim 1, wherein the solution is passed over rollers heated to 100-130°C.

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