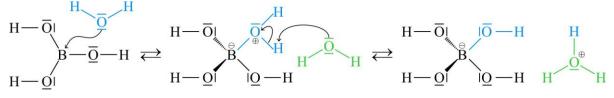
## Preparation of boric acid

Boric acid H<sub>3</sub>BO<sub>3</sub> is white crystalline forming small flakes. It is only slightly soluble in water, but its solubility sharply increases with increasing temperature. In crystalline form are the planar molecules of H<sub>3</sub>BO<sub>3</sub> ordered to layers (Fig. 1) with weak hydrogen bridges. Boric acid is very weak acid ( $K_a = 5.8 \cdot 10^{-10}$ ). It is not Brønsted acid (proton donor), but Lewis acid (acceptor of lone electron pair). Therefore, the oxonium cations in acidic solution originate from water.



 $H_{3}BO_{3}(aq) + 2 H_{2}O(1) \iff [B(H_{2}O)(OH)_{3}](aq) + H_{2}O(1) \iff [B(OH)_{4}]^{-}(aq) + H_{3}O^{+}(aq)$ 

When heated above 169 °C, boric acid dehydrates forming metaboric acid HBO<sub>2</sub> Further heating above 250 °C leads up to hygroscopic diboron trioxide.

$$2 \operatorname{H_3BO_3(s)} \xrightarrow{169 \,^\circ \mathrm{C}} 2 \operatorname{HBO_2(s)} + 2 \operatorname{H_2O(g)} \xrightarrow{250 \,^\circ \mathrm{C}} B_2 O_3(s) + 3 \operatorname{H_2O(g)}$$

Boric acid can be prepared by the protolytic reaction of tetrahydroxido-pentaoxidotetraborate octahydrate (borax) with hydrochloric acid.

$$Na_{2}[B_{4}O_{5}(OH)_{4}](aq) + 2 HCl(aq) + 3 H_{2}O(l) \longrightarrow 4 H_{3}BO_{3}(aq) + 2 NaCl(aq)$$

#### Work

Prepare 0,100 mol of boric acid by the reaction of borax with hydrochloric acid.

#### **Chemicals**

- sodium tetrahydroxido-pentaoxidotetraborate octahydrate (borax),  $Na_2[B_4O_5(OH)_4] \cdot 8 H_2O$ , white crystalline
- hydrochloric acid, HCl, concentrated, w(HCl) = 0.36

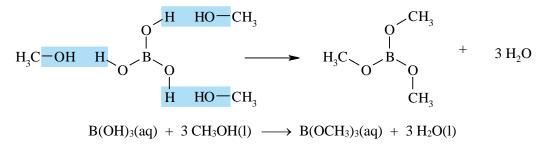
### **Procedure**

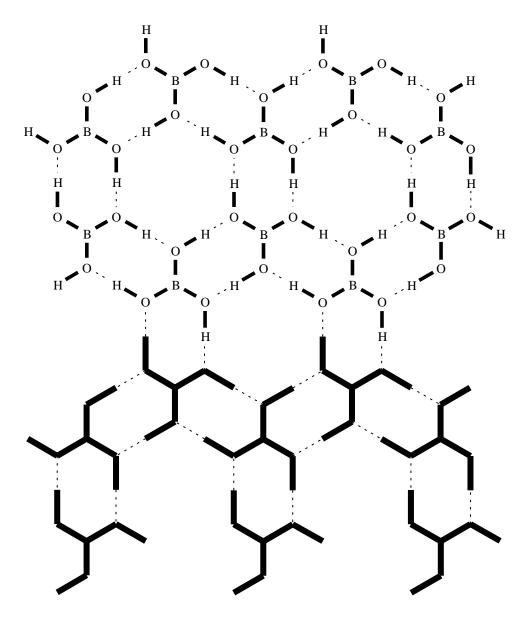
Dissolve the calculated amount of borax in warm water in a beaker. Use calculated volume of water to prepare the saturated solution of boric acid at 80 °C. Add hydrochloric acid in small portions using a glass rod and always stir the solution. After adding entire volume of hydrochloric acid, check the pH of the final solution. If the solution is not sufficiently acidic (pH < 3), add another small portions (dropwise) to tune the pH.

After cooling to room temperature filter out the shiny white crystals of  $H_3BO_3$  on the Büchner funnel, wash them with icy water directly on the funnel and dry them between clean filter papers until dry. Do not put the crystals to an oven, because boric acid could be decomposed!

### Preparation of methyl ester of boric acid

Great ability of boric acid to form esters by the reaction with alcohols is often used for its qualitative proof. Put small amount of just prepared and dried boric acid into a crucible and add few drops of methanol. Using a glass rod stir the mixture and then light it with wooden rod or a safety match in tweezers. Volatile fumes of methyl ester (trimethyl borate) change the colour of flame to green, what indicates the presence of boron compound.





**Fig. 1** Structure of crystalline boric acid.  $\ell$ (B–O) = 1,36 Å,  $\ell$ (O–H–O) = 2,71 Å, distance between layers d = 3,18 Å.

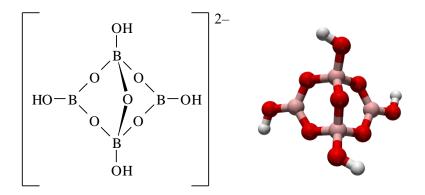


Fig. 2 Structure of tetrahydroxido-penta( $\mu$ -oxido)tetraborate anion.

# Safety instructions

Sodium tetrahydroxido-pentaoxidotetraborate octahydrate –  $Na_2[B_4O_5(OH)_4] \cdot 8H_2O_5(OH)_4$ 

- **R22** Harmful if swallowed.
- **R36/37/38** Irritating to eyes, respiratory system and skin.
- S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical adviceS36 Wear suitable protective clothing

## <u>Hydrochloric acid – HCl</u>

- R34 Causes burns.
- **R37** Irritating to respiratory system.
- S2 Keep out of the reach of children
- S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice

## <u>Boric acid – H<sub>3</sub>BO3</u>

- **R20/21/22** Harmful by inhalation, in contact with skin and if swallowed.
- **R36/37/38** Irritating to eyes, respiratory system and skin.
- **R40** Limited evidence of a carcinogenic effect.
- S22 Do not breathe dust
- S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
- **S36** Wear suitable protective clothing

## <u>Methyl alcohol – CH<sub>3</sub>OH</u>

- **R11** Highly flammable.
- **R23/25** Toxic by inhalation and if swallowed.
- S2 Keep out of the reach of children
- **S7** Keep container tightly closed
- **S16** Keep away from sources of ignition No smoking
- S24 Avoid contact with skin