Preparation of sodium thiosulphate pentahydrate

Sodium thiosulphate pentahydrate is a white ionic substance. It dehydrates spontaneously on air and at temperatures over 50 °C melts in its own hydration water. By heating over 100 °C it is fully dehydrated forming anhydrous thiosulphate. Sodium thiosulphate pentahydrate is very well soluble in water and the dissolving is very endothermic. Acids decompose sodium thiosulphate. Aqueous solutions of thiosulphate are often used for elimination of dangerous chlorine from gaseous mixtures. The mixture of sodium thiosulphate and sodium hydroxide is called "antichlorine".

$$4 \operatorname{Cl}_2(g) + \operatorname{Na}_2S_2O_3(aq) + 10 \operatorname{NaOH}(aq) \longrightarrow 2 \operatorname{Na}_2SO_4(aq) + 8 \operatorname{NaCl}(aq) + 5 \operatorname{H}_2O(l)$$

Sodium thiosulphate pentahydrate is commonly prepared by the reaction of powdered sulphur with boiling solution of sodium sulphite.

$$Na_2SO_3(aq) + S(s) \xrightarrow{\Delta T} Na_2S_2O_3(aq)$$

Work

Prepare sodium thiosulphate pentahydrate by the reaction of sulphur with 15,0 g of sodium sulphite heptahydrate.

Chemicals

- sodium sulphite heptahydrate, Na₂SO₃·7H₂O, white crystalline substance,
- powdered sulphur, S₈, yellow crystalline substance,
- ethanol, CH₃CH₂OH, colourless liquid.

Procedure

In a ground-glass flat-bottom flask dissolve the calculated amount of sodium sulphite heptahydrate in water enough to prepare 20,0 wt% sodium thiosulphate solution. Add finely powdered sulphur in 100 % excess. Fix a reflux condenser on the flask (Fig. 1). Let the reaction mixture boil softly for cca 2 hours. Then stop heating, filter the mixture from the flask through the fluted filter paper to an evaporating dish and let it evaporate to one half of its original volume.

During the evaporation, wash the unreacted sulphur on the filter with plenty of water and finally with ethanol. Transfer the wet sulphur to a watch glass and let it dry up on air. Once dry, weigh it. From the weight of unreacted sulphur, we can calculate the composition of the filtrate - the amount of prepared sodium thiosulphate as well as the unreacted sodium sulphite.

Weight the concentrated filtrate in the evaporating dish and put it to an ice bath.

Note: If there is some unreacted sodium sulphite in the filtrate, it crystalizes Fig. 1 Apparatus for the preferentially when cooled and contaminates the simultaneously crystalizing sodium thiosulphate. Therefore, the first excluded mixture of crystals should be filtered out and stored separately. Then throw a few crystals of pure sodium thiosulphate to the remaining filtrate to evoke next crystallization.

Filter out the crystals of sodium thiosulphate pentahydrate and dry them between two pieces of filter paper.



preparation of sodium thiosulphate.

Test tube experiment 1

In a test tube dissolve 1 g of sodium thiosulphate pentahydrate in 10 cm³ of water. Add small amount of diluted sulphuric acid. Acidifying the solutions of thiosulphates, a very weak thiosulphuric acid is excluded, which decomposes spontaneously to sulphur dioxide and sulphur – a beautiful example of heterogenic reaction with all four physical states:

$$H_2S_2O_3(aq) \longrightarrow H_2O(1) + SO_2(g) + S(s)$$

Escaping sulphur dioxide may be determined by a wet pH paper (must not touch the test tube). Write the chemical equation!

Test tube experiment 2

Pour *cca* 5 g of sodium thiosulphate pentahydrate into a test tube. Melt it by mild heating in a burner flame. After some time, thiosulphate fully dehydrates. Then continue heating in the strong flame. Thiosulphate decomposes, and a thin coating of yellow sulphur appears in the test tube.

4 Na₂S₂O₃(s) $\xrightarrow{\Delta T}$ 3 Na₂SO₄(s) + Na₂S(s) + 4 S(s)

Cool down the test tube and add a small portion of diluted hydrochloric acid.

 $Na_2S(s) + 2 HCl(aq) \longrightarrow 2 NaCl(aq) + H_2S(g)$

Escaping hydrogen sulphide has typical unpleasant odour and it may be determined by a wet pH paper (must not touch the test tube). Write the chemical equation!

Test tube experiment 3

Fill up a test tube to about three fourths with sodium thiosulphate pentahydrate. Put the test tube to a beaker with boiling water. Thiosulphate melts as the temperature reaches 60 °C. Continue heating in boiling water up to 100 °C. Then pull out the test tube from boiling water and put it to a beaker with iced water. Couple of minutes later the mixture cools down below its melting point but it remains still liquid. If a small crystal of thiosulphate is thrown into the test tube, very impressive quick crystallization occurs.

Safety instructions

<u>Sodium sulfite heptahydrate – Na₂SO₃ · 7H₂O</u>

| R22 | Harmful if swallowed. |
|------------|---|
| R31 | Contact with acids liberates toxic gas. |
| R36/38 | Irritating to eyes and skin. |
| S26 | In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. |
| S36/37 | Wear suitable protective clothing and gloves. |

<u>Sulfur – S</u>

| 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 advice. |
| S37/39 | Wear suitable gloves and eye/face protection. |

<u>Sodium thiosulfate pentahydrate – Na₂S₂O₃ · 5H₂O</u>

- **R36/38** Irritating to eyes and skin.
- S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.S37/39 Wear suitable gloves and eye/face protection.