## **Preparation of sodium sulphite heptahydrate** – *Procedure*

Sodium sulphite heptahydrate forms colourless monoclinic crystals. It is formed by crystallization of saturated aqueous solutions below a temperature of 33.4 °C. Above this temperature, it crystallizes from solution as anhydrous. Sodium sulphite heptahydrate turns into an anhydrous salt above a temperature of 150 °C. Wet crystals or an aqueous solution are easily oxidized even by atmospheric oxygen to sodium sulphate. Sodium sulphite heptahydrate dissolves well in water. The sulphate anion hydrolyses and therefore the aqueous solution of sodium sulphate is basic. It combines with sulphur dioxide in an aqueous solution to form sodium bisulphite. The sodium bisulphite solution is prepared by introducing sulphur dioxide into the sodium hydroxide solution

$$SO_2(aq) + NaOH(aq) \longrightarrow NaHSO_3(aq)$$
 (1)

Sodium sulphite is then prepared by the protolytic reaction of sodium bisulphite with sodium hydroxide

$$NaHSO_{3}(aq) + NaOH(aq) \longrightarrow Na_{2}SO_{3}(aq) + H_{2}O(l)$$
(2)

### Work

Pripravte heptahydrát siričitanu sodného reakciou oxidu siričitého s hydroxidom sodným.

## **Chemicals**

- sulphur dioxide, SO<sub>2</sub> use of SO<sub>2</sub> in the preparation of Na<sub>2</sub>SO<sub>3</sub> is  $\approx 40,0$  %.
- sodium hydroxide, NaOH, granulated

## **Procedure**

We will assemble the apparatus for the preparation of sulphur dioxide and sodium sulphite (Fig. 1). Sulphur dioxide is purified by bubbling through water 2. Liquefier 4 is used only if we also want to do experiments with liquefied sulphur dioxide. Otherwise, we can omit it in the apparatus.



Fig. 1 Apparatus for the preparation of sodium sulphite

1 - gas generator, 2 - filter, 3 - reactor, 4 - liquefier, 5 - absorber (15% NaOH), 6 - water pump.

We will prepare a solution of sodium hydroxide with such a composition that the resulting solution of sodium sulphite has the composition of a saturated solution at a temperature of 20 °C. Divide the prepared solution into two equal parts. Pour one part of the sodium hydroxide solution into reactor 3, where sulphur dioxide from developer 1 (reaction 1) is added to it. The reaction is strongly exothermic and the solution soon heats up to near boiling. That's why we cool the reactor in a beaker with cold water or ice. Sulphur dioxide is added to the sodium hydroxide solution until it is absorbed in the solution. We can monitor the course of the reaction using the released heat of reaction, i.e. the reactor is hot during the reaction and gradually cools down after the reaction is over. The bubbling of sulphur dioxide through the sodium hydroxide solution takes 30 to 50 minutes. After the end of the reaction, we <u>stop the evolution of sulphur dioxide</u> (by adding water to the fractionation flask) and let the apparatus cool down.

Pour the sodium bisulphite solution from the reactor into the crystallization dish with the second part of the sodium hydroxide solution set aside (reaction 2). Allow the resulting sodium sulphite solution to cool in

a mixture of ice and water. Filter the formed sodium sulphite heptahydrate crystals under reduced pressure through a frit funnel. We dry the crystals between sheets of filter paper and weigh them.

# **Preparation of sodium sulphite heptahydrate** – *Safety instructions*

## Sulfur dioxide – SO<sub>2</sub>

R23	Toxic by inhalation.
R34	Causes burns.
R44	Risk of explosion if heated under confinement.
<b>S26</b>	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
<b>S45</b>	In case of accident or if you feel unwell seek medical advice immediately (show the label where possible).
<b>S7/9</b>	Keep container tightly closed and in a well-ventilated place.
S36/37/39	Wear suitable protective clothing, gloves and eye/face protection.

### <u>Sodium hydroxide – NaOH</u>

R34	Causes burns.
<b>S26</b>	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
<b>S28</b>	After contact with skin, wash immediately with plenty of (to be specified by the manufacturer).
<b>S45</b>	In case of accident or if you feel unwell seek medical advice immediately (show the label where
	possible).
<b>S36/37/39</b>	Wear suitable protective clothing, gloves and eye/face protection.

# <u>Sodium sulfite heptahydrate – Na<sub>2</sub>SO<sub>3</sub> · 7H<sub>2</sub>O</u>

avant	Sugue	noptanyarate	114200
<b>R22</b>		Harmful if swa	llowed.

- **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.