Preparation of potassium sulphate

Potassium sulphate is a white crystalline soluble in water. In common organic solvents, it is insoluble. Potassium sulphate can be prepared by the protolytic reaction of potassium hydroxide with sulphuric acid according to the chemical equation

$2 \operatorname{KOH}(\operatorname{aq}) + \operatorname{H}_2 \operatorname{SO}_4(\operatorname{aq}) \longrightarrow \operatorname{K}_2 \operatorname{SO}_4(\operatorname{aq}) + 2 \operatorname{H}_2 \operatorname{O}(\operatorname{I})$

Work

Prepare potassium sulphate by a protolytic reaction of KOH with H₂SO₄.

Chemicals

- potassium hydorxide, KOH, white crystalline, granules or flakes
- sulphuric acid, H_2SO_4 , concentrated water solution, w = 0.96
- ethanol, CH₃CH₂OH, denaturised spirit
- phenolphtaleine, enolic solution

Procedure

Weigh calculated amount of KOH and measure calculated volumes of 96% solution of H_2SO_4 and water necessary for preparation of a solution of potassium sulphate saturated at temperature 50 °C. Add three drops of an ethanolic solution of phenolphthalein to the concentrated sulphuric acid in a beaker. Observe the orange colour of indicator (phenolphthalein) in an extremely acidic solution. Divide calculated volume of water into two equal parts and pour them into two empty beakers. Dissolve solid potassium hydroxide in one portion of water. Using a glass rod carefully dissolve concentrated sulphuric acid in the other portion of water. Add in small portions the solution of potassium hydroxide to the solution of sulphuric acid and always stir the solution with a glass rod. If pure chemicals were used, the final solution should have pale pink colour. If the solution is still colourless, hydroxide used was probably not pure and another <u>small</u> portion of hydroxide must be carefully added to turn the colour to pale pink. Potassium hydroxide is very hygroscopic and it also react with carbon dioxide in air, what makes it impure:

$$2 \text{ KOH}(s) + \text{CO}_2(g) \longrightarrow \text{K}_2\text{CO}_3(s) + \text{H}_2\text{O}(l)$$

Heat the final solution to 70 - 80 °C and filter it through a fluted filter paper directly to an evaporation dish. Put the evaporating dish with the pale pink solution on a water bath and wait until crystallisation film is formed (saturated solution). Turn off a gas burner and measure the temperature of the solution. Pour the solution from the evaporating dish into a crystallization dish. Place the crystallization dish to the coolest part of the laboratory (or use an ice bath). After cooling to room temperature measure the temperature of the solution with crystals formed. Filter out the crystals on a Büchner funnel under vacuum and place wet crystals of the product on a watch glass into an oven heated over 100 °C to dry them up. Weigh the final dry white crystals of potassium sulphate.

If the dried product has still pink colour it could be purified by crystallization from a water-ethanolic solution. In this case dissolve the product in appropriate amount of water and add double amount of ethanol. Stir the solution rigorously and let it stand for 15 minutes. Since ethanol is miscible with water and potassium sulphate is not soluble in ethanol, potassium sulphate excludes from the solution in form of a fine white powder. Filter out the excluded product and dry it up on a watch glass in an oven again.

Safety instructions

<u>Potassium hydroxide – KOH</u>

R35	Causes severe burns.
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>Sulphuric acid – H_2SO_4 </u>

R23	Toxic by inhalation.
R34	Causes burns.
R49	May cause cancer by inhalation.
S23	Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer)
S45	In case of accident or if you feel unwell seek medical advice immediately (show the label where possible)
S36/37/39	Wear suitable protective clothing, gloves and eye/face protection.

<u>Potassium sulphate – K_2SO_4 </u>

R22 Harmful if swallowed.

S36 Wear suitable protective clothing

<u>Ethyl alcohol – C₂H₅OH</u>

- **R11** Highly flammable.
- **S7** Keep container tightly closed
- **S16** Keep away from sources of ignition No smoking