Determination of Sulphate as Barium Sulphate

Introduction

The estimation method of Sulphate as Barium Sulphate is based on the slow addition of dried $BaCl_2$ barium chloride solution to the hot sulphate solution in an acidic media using HCl acid.

$$\mathbf{Ba^{+2} + SO_4^{-2} \longrightarrow BaSO_4}$$

The acid concentration was 0.05 N following **the below reasons :**

1- Large particles of the precipitate can be obtained
2- Precipitation particles are pure

In a solution of HCl (0.1 N), the solubility of barium sulphate is approximately 10 mg / L in (20°C), where as, in 1N solution, the solubility is approximately 87 mg / L .

The precipitation in an acidic medium of 0.05N with a moderate concentration of barium chloride will reduce the dissolution of barium sulfate to a large extent so that it can be ignored.

To dry barium sulphate precipitate, the precipitator should always be burned for at temperature (110-115°C) because the crystallization water is strongly attached to the crystalline network of the precipitation. This water can be disposed at a high temperature only. Therefore, barium sulphate should be burned at a temperature higher than (500°C) to precpitate of water as this salt is stable even at high temperatures.

$$BaSO_4.XH_2O \xrightarrow{115^0C} BaSO_4.XH_2O \xrightarrow{900^0C} BaSO_4 + X\,H_2O$$

The precipitation may be reduced at higher temperatures by using filter paper. In addition to the possible dissociation of salt into barium oxide and sulfur oxides**:**

$$BaSO_4 + 4C \longrightarrow BaS + 4CO$$

$$BaSO_4 \longrightarrow BaO + SO_3$$

But good results can be obtained when filter paper carbonization is at the lowest possible temperature without burning.

A slight disintegration of barium sulfide (BaS) can be obtained with precipitation cooling and crucible after burning, adding some drops of dilute H_2SO_4 and re-heating to remove the unused acid into the reaction in the form of vapors:

$$BaS + H_2SO_4 \longrightarrow BaSO_4 + H_2S$$

Materials

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1- **Sample sulphate salt.**
2- **Concentrated hydrochloric acid**
3- **Barium chloride solution (10%).**
4- **Sulfuric acid diluted (10%).**

Procedure

1- Carefully weigh 1.0 g of sulfur salt in a (400 ml) beaker equipped with a stirrer. Add 1 ml of concentrated hydrochloric acid and then dilute the solution to 100 ml as final volume.

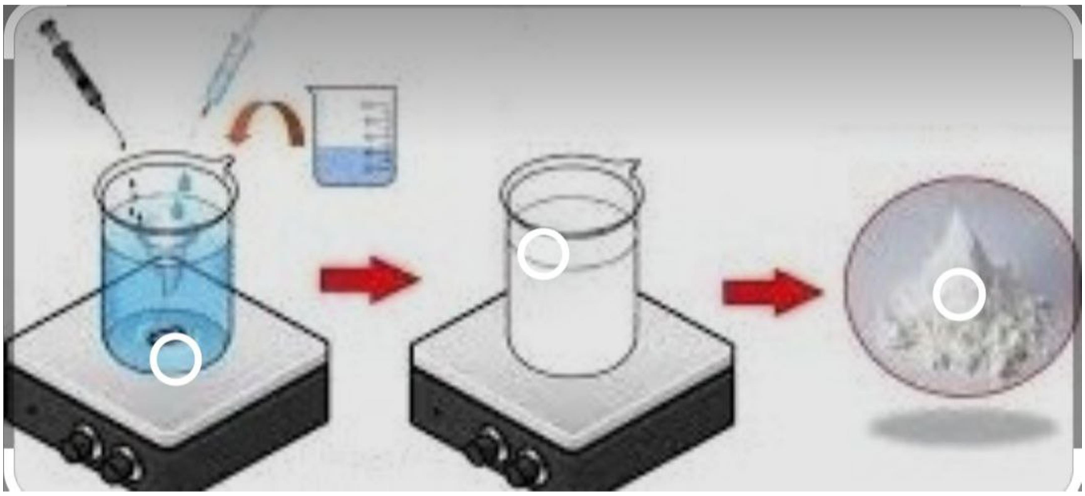
2- Heat the solution until boiling and add slowly with the continuous stirring (40 ml) of the barium chloride hot solution at a concentration of 10%. Heat the solution until it reaches boiling and (show the bubbles) and leaves it for 10 min.

3- Install the filtration apparatus and filter. Wash the precipitate with hot distilled water then let the precipitate dry up. Fold the filtration paper containing the precipitate and placed in a previously balanced crucible

4- Charcoal filter paper first slowly above the flame lamp and after it has been burned into white ash; two drops of concentrated sulfuric acid are added.

5- Place the crucible containing the burnt paper in a furnace muffle (900^0C) and then reheat slowly to remove the excess acid.

6- Transfer the crucible to a desiccator and weight the crucible and precipitate after it cools.



Calculation

Wt of Sample = 1.0 gm

Wt of precipitate $BaSO_4$= 0.5 gm

If you know A.wt of Ba= 137 , S= 32 , O= 16

Calculate the percentage of SO_4 in the sample ?

% SO_4 = Wt of SO_4 / Wt of Sample × 100

Wt of SO_4 = G.F × Wt of $BaSO_4$

G.F =MWT.(SO_4) / MWT. $BaSO_4$

=32+4X16 / 137+ 32+4X16 = 0.412

Wt of SO_4 = 0.412× 0.5 = 0.2gm

% SO_4 = 0.2 / 1.0 × 100 = 20.6 %

Discussion

1- sulphates are precipitate in an acidic medium (0.05 N)? why?
2- Why barium sulfate precipitate is burned to a temperature of about 900^0C?
3- Sulfuric acid is added to the crucible after burning the barium sulphate precipitate?
4- What are the main sources of barium sulphate precipitate contamination?