

## Lab - 8

### Sulphate

#### Gravimetric Method for Sulphate Determination:

##### Theory:

Nearly all sulphates in water can be precipitated as  $\text{BaSO}_4$  on reaction with barium chloride ( $\text{BaCl}_2$ ) under acidic conditions. Since this compound has very low solubility product, the precipitated  $\text{BaSO}_4$  can be filtered and the residue weighed. The weight of the residue is proportional to the original sulphate concentration in solution.

##### Procedure:

- Dissolve 100 g of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in 1000 mL of distilled water. Label the bottle, "**Barium Chloride Solution for Sulphate Determination**". (*This solution will be available in the laboratory*).
- Dissolve  $\text{Na}_2\text{SO}_4$  in 1000 mL of water to make a sulphate solution containing 1000 mg sulphate/L. Prepare four such bottles. Label, "**Sulphate Standard for Turbidimetric / Gravimetric Method**". (*This solution will be available in the laboratory*).
- Add 50 mL water to 50 mL concentrated HCl. Label, "**1+1 HCl solution**". (*This solution will be available in the laboratory*).
- Take 100 mL aliquot each of sulphate standards of 0, 10, 25, 50 and 100 mg/L sulphate concentrations. Also prepare a 100 mL aliquot of sample.
- Adjust pH of each standard to 4.5-5 with the 1+1 HCL solution.
- While stirring gently, slowly add  $\text{BaCl}_2$  solution until precipitation appears to be complete. Then add about 2 mL of excess  $\text{BaCl}_2$  solution.
- Filter using GF/C filter, and dry at  $103^\circ\text{C}$  overnight. Cool in desiccator and weigh. Draw a calibration curve of the weight of residue versus sulphate concentration in solution.
- Use this calibration curve to determine the sulphate concentration in sample.

#### Turbidimetric Method for Sulphate Determination:

##### Theory:

Nearly all sulphates in water can be precipitated as  $\text{BaSO}_4$  since this compound has very low solubility product. The precipitated  $\text{BaSO}_4$  particles form a turbid suspension, turbidity of which is proportional to the original sulphate concentration in the solution.

**Procedure:**

- Buffer Solution: Dissolve 30 g  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ , 5 g  $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ , 1 g  $\text{KNO}_3$ , and 20 mL of acetic acid (99%) in 500 mL distilled water, and make up to 1000 mL. Label, "**Buffer Solution for Sulphate Determination by Turbidimetric Method**". (*This solution will be available in the laboratory*).
- Dissolve 1.479 g anhydrous  $\text{Na}_2\text{SO}_4$  in 1000 mL of water to make a sulphate solution containing 1000 mg sulphate/L. Label, "**Sulphate Standard for Turbidimetric/Gravimetric Method**". (*This solution will be available in the laboratory*).
- Measure 100 mL of samples containing 0, 50, 100, 150, and 200 mg/L sulphate in 250 mL conical flasks. Add 20 mL of buffer solution and mix on a stirring apparatus. While stirring add a spoonful of  $\text{BaCl}_2$  crystals and stir for a further 60 seconds.
- Measure turbidity of the solutions for obtaining calibration curve.
- Measure sulphate concentration in sample using the above calibration curve.

## Lab Exercise – 8

### 8A Understanding About Sulphate in Water (4 x 10 Marks = 40 Marks)

1. What is the significance of a high sulphate concentration in water supplies and in wastewater disposal?
2. List four precautions that must be observed to ensure an accurate gravimetric determination of sulphate concentration.
3. What are two purposes for the conditioning reagent used in the turbidimetric determination of sulphate concentration?
4. From equilibrium considerations, calculate the relative proportions of sulphide in the  $\text{H}_2\text{S}$ ,  $\text{HS}^-$  and  $\text{S}^{2-}$  forms at a) a pH of 6.0; b) a pH of 7.5; and c) a pH of 10.0.

### 8B Estimation of Sulphate in Water

#### A. Lab Report: (4 x 10 Marks = 40 Marks)

Data: A Mass of unknown sample containing sulphate: 0.6039 g

Mass of empty filter: 24.3849 g

Mass of filter +  $\text{BaSO}_4$ : 35.3639 g

#### Calculations (show work):

1. Calculate the mass of  $\text{BaSO}_4$ .
  2. Calculate the mass of sulphate ion in the original solid.
  3. Calculate the % mass of sulphate in the unknown sample.
  4. The solid unknown was potassium sulphate. Calculate the percent error.
- B.** The sulphate ion concentration in natural water can be determined by measuring turbidity that results when an excess  $\text{BaCl}_2$  is added to a measured quantity of sample.

(1 x 4 Marks + 2 x 8 = 20 Marks)

A turbidimeter, the instrument used for the analysis, was calibrated with a series of standard  $\text{NaSO}_4$  solutions. The following data were obtained in the calibration of sulphate concentration,  $C_x$ :

$C_x$ , mg $\text{SO}_4^{2-}$ /L	Turbidimeter reading, R
0.00	0.06
5.00	1.48
10.00	2.28
15.00	3.98
20.00	4.61

Assume that a linear relationship exists between the instrument reading and the concentration.

1. Plot the data on a graph paper
2. Calculate using the least-squares method, slope and intercept, and write the equation of the line.
3. Calculate the concentration of sulphate in a sample yielding a turbidimeter reading of 2.84. Find the standard deviation and the coefficient of variation.