

LAB EXPERIMENT – 1

Course: Engineering Chemistry

Course Code: CHY1701

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Estimation of Sulphate for Assessing Water Contamination by Conductivity Method

Sulphate (SO_4^{2-}) is found in almost all natural water. Origin of most sulphate compounds is the oxidation of sulphite ores, presence of shales or the industrial wastes. Ground water moving through soil and rocks containing sulphate minerals result in higher dissolved sulphate ions than permissible limit.

Problems due to excess sulphate ion concentration in water:

- Sulphates cause scale formation in boilers, pipes, etc.
- High sulphate concentration will leads to corrosion on copper piping.
- Sulphate has a laxative effect and creates diarrhoea leading to dehydration in humans and animals.
- High sulphate concentration leads to eutrophication of water bodies leads to reduction of dissolved oxygen Sulphate will give bitter taste to water if the concentration exceeds beyond 250 ppm.

Methods to estimate sulphate ion concentration in water:

1. **Turbidimetry method:** It involves the measurement of turbidity formed when an aliquot of BaCl_2 -gelatin reagent is added to acidified sulphate solution.
2. **Titrimetric method:** By dissolving precipitated BaSO_4 in excess of EDTA solution and the excess EDTA is back titrated with standard Zinc solution.
3. **Colorimetric Measurement:** Based on the reaction of barium chloranilate with sulphate ion at pH 4 in ethanol yield highly coloured acid-chloranilate ion and is measured at 530 nm.
4. **Conductometric method:** This method measures the conductivity of the solution as the titration proceeds. Conductance tends to vary with the characteristics of the solvent, number, size and charge of ions involved. When one ion is replaced by another ion significantly during the titration, conductance will change in a linear manner until the replacement is complete. After that, the line will change to different slope due to the additional inclusion of another ion of difference conductance.

Expt. No.: 1

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Experiment	Estimation of Sulphate for Assessing Water Contamination by Conductivity Method
Problem definition	People using water with high levels of sulfate are vulnerable to dehydration and diarrhea. Kids are more sensitive to sulfate than adults.
Methodology	Conductivity of the soluble sulphate solution will change when it is precipitated by BaCl_2 . Conductivity will reach minima when all sulphate ions are precipitated, and from which, the total amount of sulphate ion present in the water can be determined.
Solution	Amount of BaCl_2 required to remove the dissolved sulphate can be estimated.
Student learning outcomes	Students will learn to a) perform conductometric method b) remove sulphate ion from irrigate water

Principle:

Electrolyte solutions conduct electricity due to the presence of ions in solution. In case of precipitation titration between BaCl_2 and Na_2SO_4 , the conductance decreases slowly due to the replacement of Na^+ ion by SO_4^{2-} upto the equivalence point. After the equivalence point, the conductance increases rapidly due to the excess addition of BaCl_2 which remains in solution as Ba^{2+} and Cl^- . This makes detection of neutralization point easy from the conductance trend plotted as a graph. This is the principle used in the estimation of SO_4^{2-} from contaminated water sample.

Requirements:

Reagents and solutions: BaCl_2 (0.05 N), Na_2SO_4 (0.25 N), unknown sulphate solution and distilled water.

Apparatus: TDS-Conductivity Bridge, Conductivity cell, Burette, Pipette, Volumetric flasks, Glass rod, Beaker (100 mL).

Procedure:

Calibration of TDS-Conductivity meter: Place a freshly prepared 0.01 N KCl solution (given in bottle) in a 100 mL beaker. Dip the conductivity cell in this solution and connect to the TDS-Conductivity meter. Press “CAL” button and complete the internal calibration of the instrument.

Standardization of BaCl₂ (Titration – 1):

In a 50 mL standard flask, fill with Na₂SO₄ (Bottle A) solution upto the mark resulting in a solution containing 0.96 mg/mL of sulphate ions (Mol. wt. of SO₄²⁻ = 96.06). Pipette out 20 mL of this solution in a 100 mL beaker and add 20 mL of distilled water to it. The conductivity cell is dipped into the beaker and connected to TDS conductivity meter. Fill the burette with BaCl₂ solution (Bottle B). Record the conductivity of the sulphate solution as 0th reading. Add 0.5 mL BaCl₂ of known concentration into the beaker, stir with glass rod and note down the conductance. Continue the addition of BaCl₂ (0.5 mL each time) and note the conductance after each addition. Continue the titration beyond the equivalence point for about 3 mL. The conductance will decrease till complete precipitation of BaSO₄ and then starts increasing on continuing the addition of BaCl₂. A graph is now drawn by plotting conductance vs volume of BaCl₂ added. Intersection point from the plot gives the volume of BaCl₂ required for precipitating the sulphate present in the known sample.

Estimation of unknown sulphate in the given solution (Titration – 2):

Make up the unknown sulphate solution given in a 50 mL standard flask. Pipette out 20 mL of this solution into a 100 mL beaker and add 20 mL distilled water to it. Dip the conductivity cell and repeat the above procedure with the unknown sulphate solution and determine the amount of BaCl₂ required for precipitating the unknown sulphate in the sample.

From the two titrations carried out, calculate the amount of sulphate present in the effluent sample.

Table 1: Conductometric Titrations

Titration-1: Standardization of BaCl₂		Titration-2: Estimation of sulphate content	
Volume of BaCl ₂ added (mL)	Conductance (μ mhos)	Volume of BaCl ₂ added (mL)	Conductance (μ mhos)
0.0	2.9	0.0	3.1
1.0	2.9	1.0	3.1
2.0	2.9	2.0	3.0
3.0	2.9	3.0	3.0
4.0	2.8	4.0	2.9
5.0	2.8	5.0	2.9
6.0	2.8	6.0	2.8
7.0	2.7	7.0	2.8
8.0	2.7	8.0	2.8
9.0	2.7	9.0	2.8
10.0	2.9	10.0	2.7
11.0	3.1	11.0	2.9
12.0	3.3	12.0	3.1
13.0	3.7	13.0	3.3

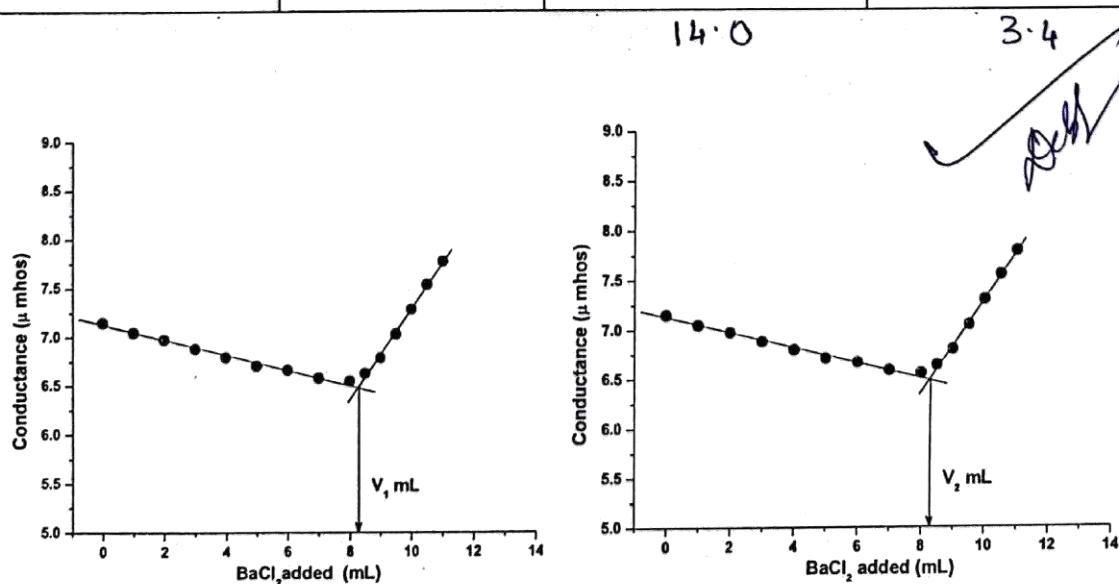
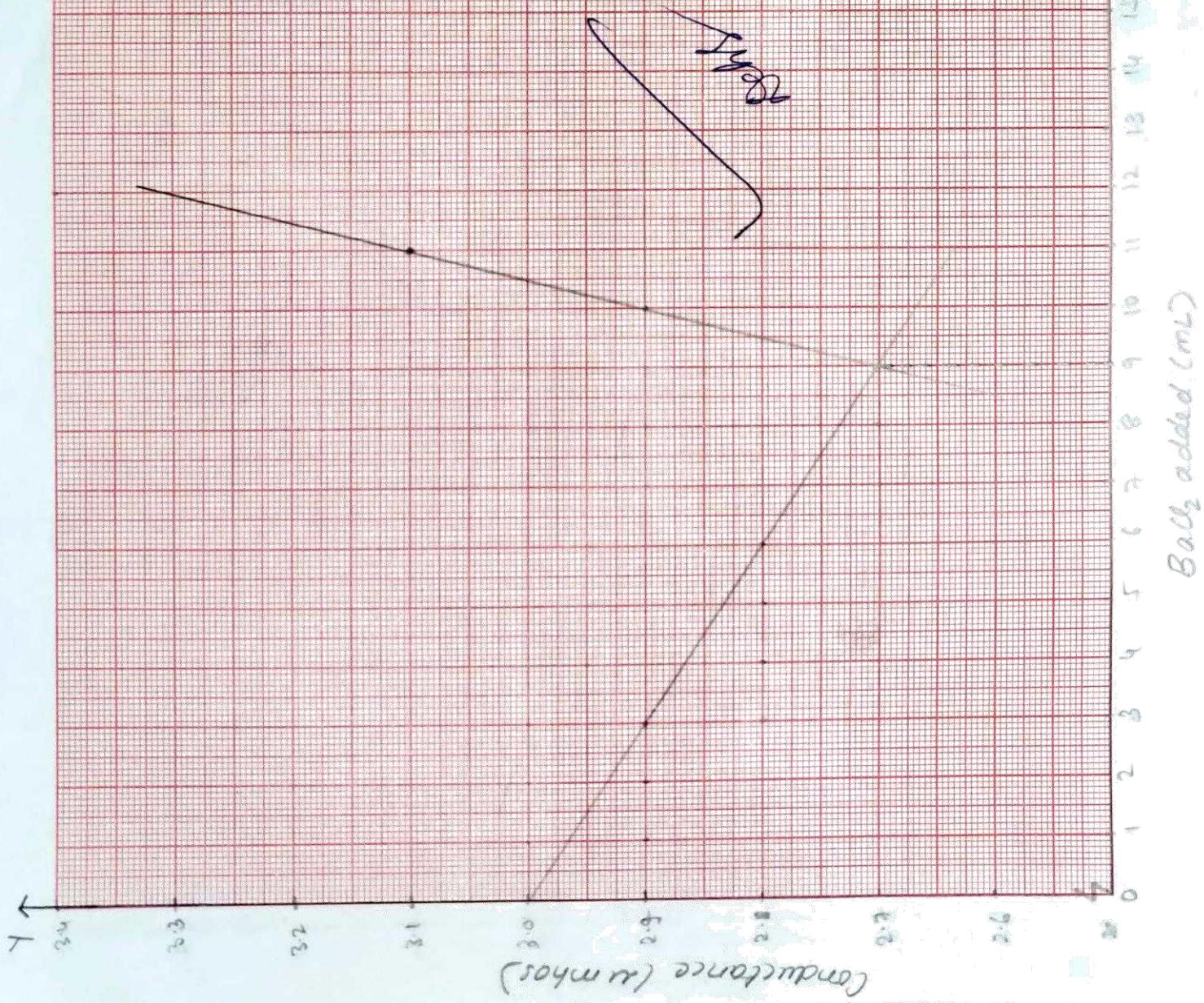


Fig 1: Model graphs - 1 and 2 for TDS-Conductometric estimation of known and unknown sulphate sample solutions, respectively.

Scale:
X-axis \rightarrow 1 cm = 1 unit
Y-axis \rightarrow 1 cm = 0.05 unit

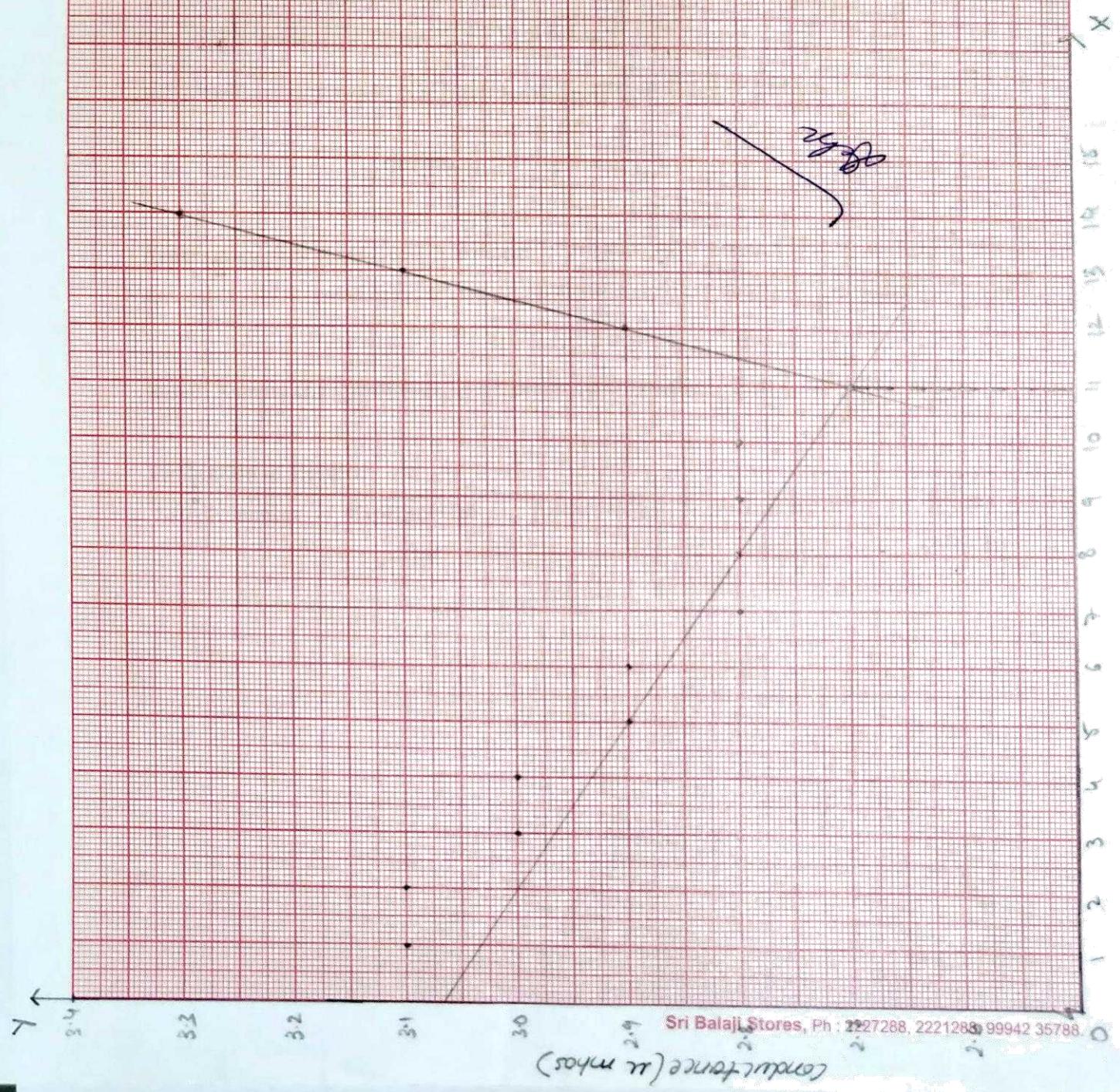
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BaCl_2 added (mL)

Na_2SO_4

Scale:
 χ -axis $\rightarrow 1\text{cm} = 1\text{unit}$
 y -axis $\rightarrow 1\text{cm} = 0.05\text{ unit}$



Unknown
subphase

$\beta \text{BaCl}_2 \text{ added (mL)}$

Calculations:

A). Standardization of 0.05 N BaCl₂:

(N x V) of BaCl₂ solution = (N x V) of sodium sulphate

$$\text{N of BaCl}_2 \text{ solution} = \frac{0.025}{0.25} \text{ N} \times 20 \text{ mL}$$

q
Volume measured from Plot-1 (V₁)
= 0.05 N

B). Estimation of unknown sulphate:

(N x V) of irrigation water sample = (N x V) of BaCl₂ solution

$$\text{N of irrigation water sample} = \frac{0.05}{0.025} \text{ N} \times \frac{20 \text{ mL}}{\text{Volume measured from Plot-2 (V}_2)}$$

= 0.0275 N

Amount of sulphate present in 1L = Normality of irrigation water sample x Mol. wt. of SO₄²⁻ (96.06)

$$\text{Amount of sulphate present in given sample solution} = \frac{\text{Strength of irrigation water sample} \times 96.06 \times 100}{1000 \times 2}$$

= 0.132 grams in 100 mL

Result: Amount of sulphate in given irrigation water sample = 0.132 grams.

Evaluation of Result:

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded
14				