Irrigation water-sulphate ion analysis by conductometry

Expt No. 1

Date: 14/12/2017

Principle

Electrolyte solutions conduct electricity due to the presence of ions in solution. In case of precipitation titration between BaCl₂ and Na₂SO₄, the conductance decreases slowly due to the replacement of Na⁺ ion by SO₄²⁻ upto the equivalence point. After the equivalence point the conductance increases rapidly due to the excess addition of BaCl₂ which remains in solution as Ba²⁺ and Cl⁻. This makes detection of neutralization point easy from the conductance trend plotted as a graph. This principle is used in the estimation of SO₄²⁻ from an effluent sample.

Apparatus required

TDS-Conductivity bridge, conductivity cell, burette, pipette, volumetric flasks, glass rod and 100 mL beaker.

Chemicals required

BaCl₂, Na₂SO₄, unknown sulphate solution and distilled water.

Procedure

Calibration of TDS-Conductivity meter:

Place a freshly prepared 0.01 N KCl solution (given in bottle) in a 100 mL beaker and 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:

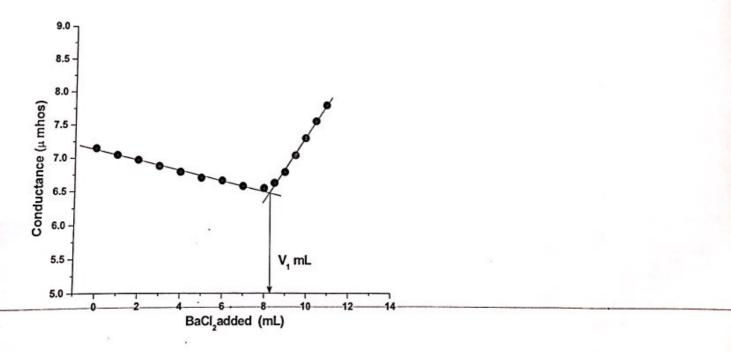
Standard flask A contains Na₂SO₄ which after making up to 50 mL will result in a solution containing 0.96 mg/mL of sulphate ions. Pipette out 20 mL of this solution and add 20 mL of distilled water to it in a 100 mL beaker. The conductivity cell is dipped into the beaker and connected to TDS conductivity meter. Fill the burette with BaCl₂ solution (Solution B). Record the conductivity of the sulphate solution as 0th reading. Add 0.5 mL portion of known concentration of BaCl₂ 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 3mL. 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 and the intersection point from the graph gives the volume of BaCl₂ required for precipitating the sulphate from the known sample.

Table. 1. Standardization of BaCl₂

Volume of BaCl ₂	Conductance	Volume of BaCl ₂	Conductance
added (mL)	(µ mhos)	added (mL)	(μ mhos)
0.0	4.8	\$5 11.0	3.8
9.5 1.0	4.7	6.0 12.0	4.1
# 2.0	4.6	€ 13.0	4.2
≢ 5 3.0	4.4	7.0 14.0	4.4
₹ 4.0	4.3	₹\$ 15.0	4.6
≇ 5.0	4.2	8.0 16.0	4.7
3.0 6.0	4.1	8.5 17.0	4.9
· 35 7·0	4.0	9.0	5.0
4.0 8.0	/3.9	95 19.0	
45 9.0	3.8	10	
5.0 10.0	3.7	10.5	

Fig. Model graph-1 for TDS-Conductometric estimation of known sulphate sample solution



Estimation of unknown sulphate in the given solution:

Make up the unknown sulphate solution given in standard flask B up the 50 mL mark. 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. 2 Estimation of unknown sulphate solution

Volume of BaCl ₂		Conductance	Volum	ne of BaCl ₂	Conductance	
0.0		4.7	5.5	11.0	3.8	
0.5	1.0	4.6	6.0	12.0	3.9	
1.0	2.0	4.5	6:5	13.0	4.1	
1.5	3.0	4.3	7.0	14.0	4.3	
2.0	4.0	4.2	. 7.5	15.0	4.4	
2.5	S-0	4.1	8.0	16.D	4.6	
3.0	6-0	4.0	8.5	17.0	4.7	
3.5	7.0	3-9	9.0	18.0	4.9	
4.0	8-0	3-8	9.5	/		
4.5	9.0	3.7	10			
5.0	10.0	3.7	10-5			

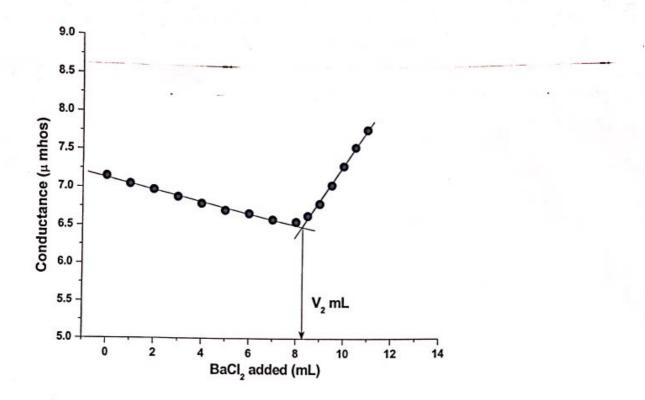


Fig. Model graph-2 for TDS-Conductometric estimation of unknown sulphate sample solution

Calculations:

A) Standardization of BaCl2:

From the first plot,

20 mL of known sulphate solution required V₁ mL of BaCl₂ for complete precipitation of sulphate.

142 g of Na₂SO₄ contains 96 g of SO₄²⁻

Therefore, 0.02N of 1 L Na₂SO₄ contains 0.96 g of SO₄²-ions

20 mL of 0.02N of Na₂SO₄ contains 0.024 g = **24 mg** of SO_4^{2-} ions

Therefore, 1 mL of BaCl₂ will precipitate $24/V_1 = .2.4...$ mg of sulphate (Y)

B) Estimation of unknown sulphate:

From the second plot,

20 mL of unknown sulphate consumed V₂ mL of BaCl₂ for complete precipitation of sulphate.

Sulphate present in 20 mL of the unknown solution = $V_2 \times Y = 23.04$. mg (Z)

Sulphate present in 1 L of unknown solution = $\frac{ZX1000}{20}$ ppm

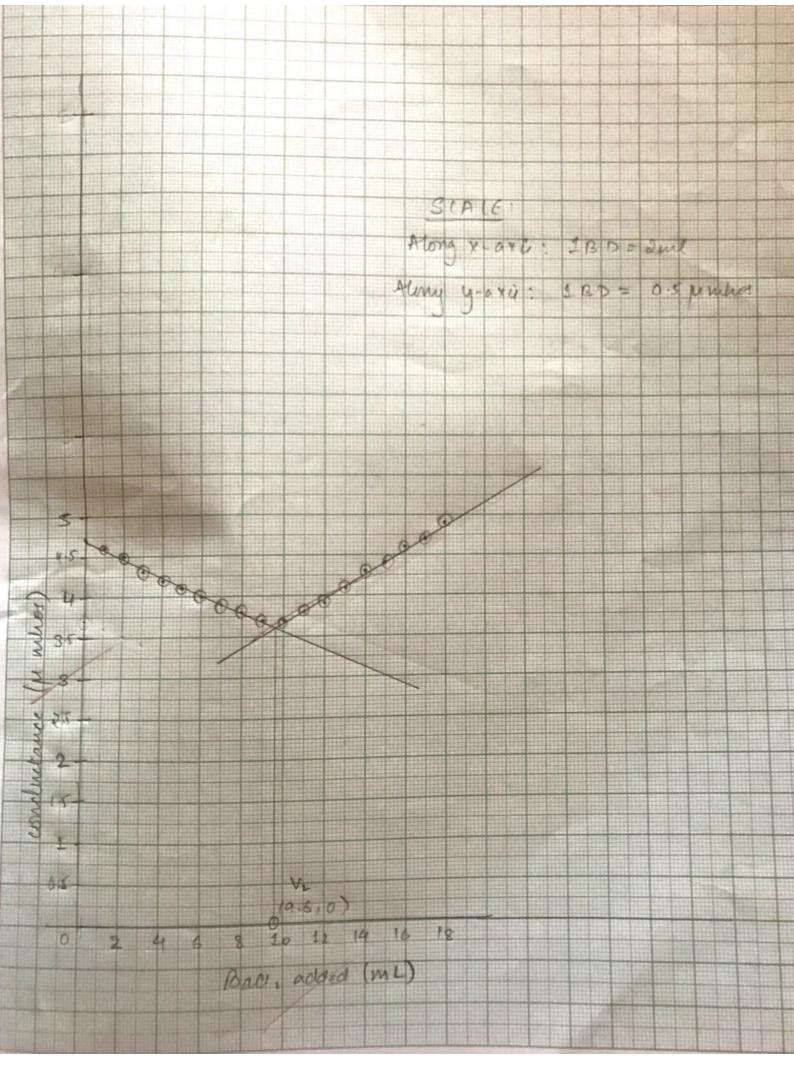
D	74
Res	ult

Amount of sulphate present in the given sample =ppm. (1052)87-/

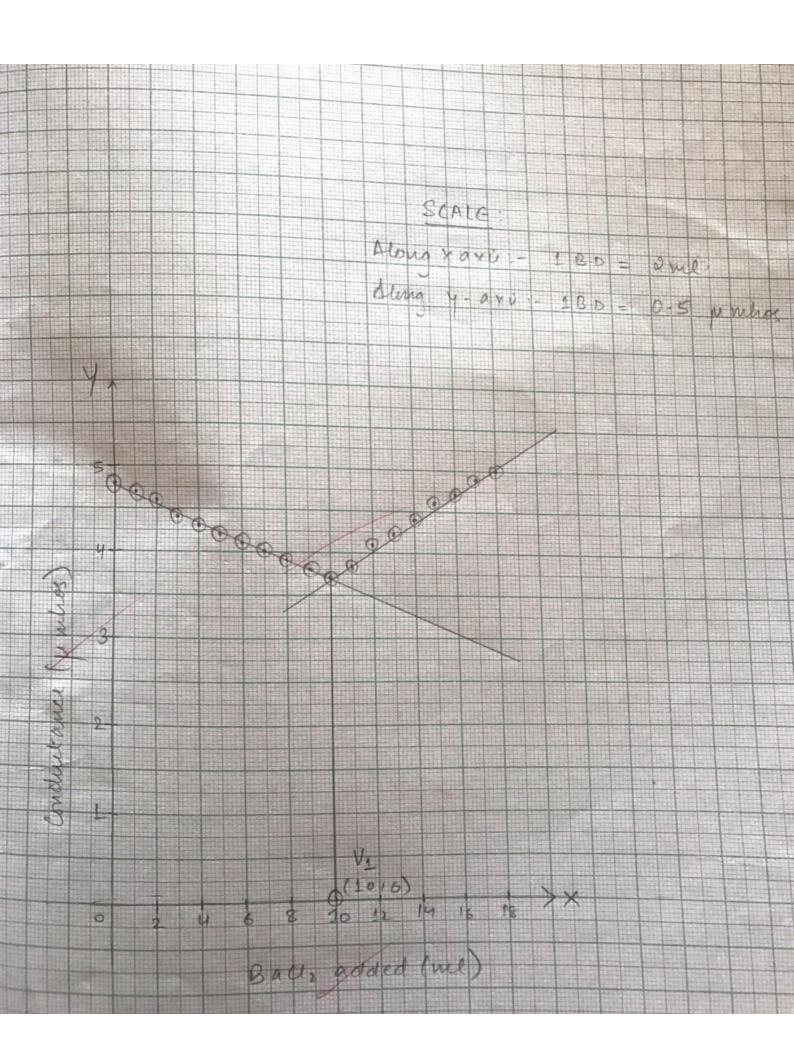
Evaluation of result

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded
	1152	1100	2.1	29/12/12

21/00 = 100 = 9.5



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