

CHAPTER 2

EXPERIMENTAL

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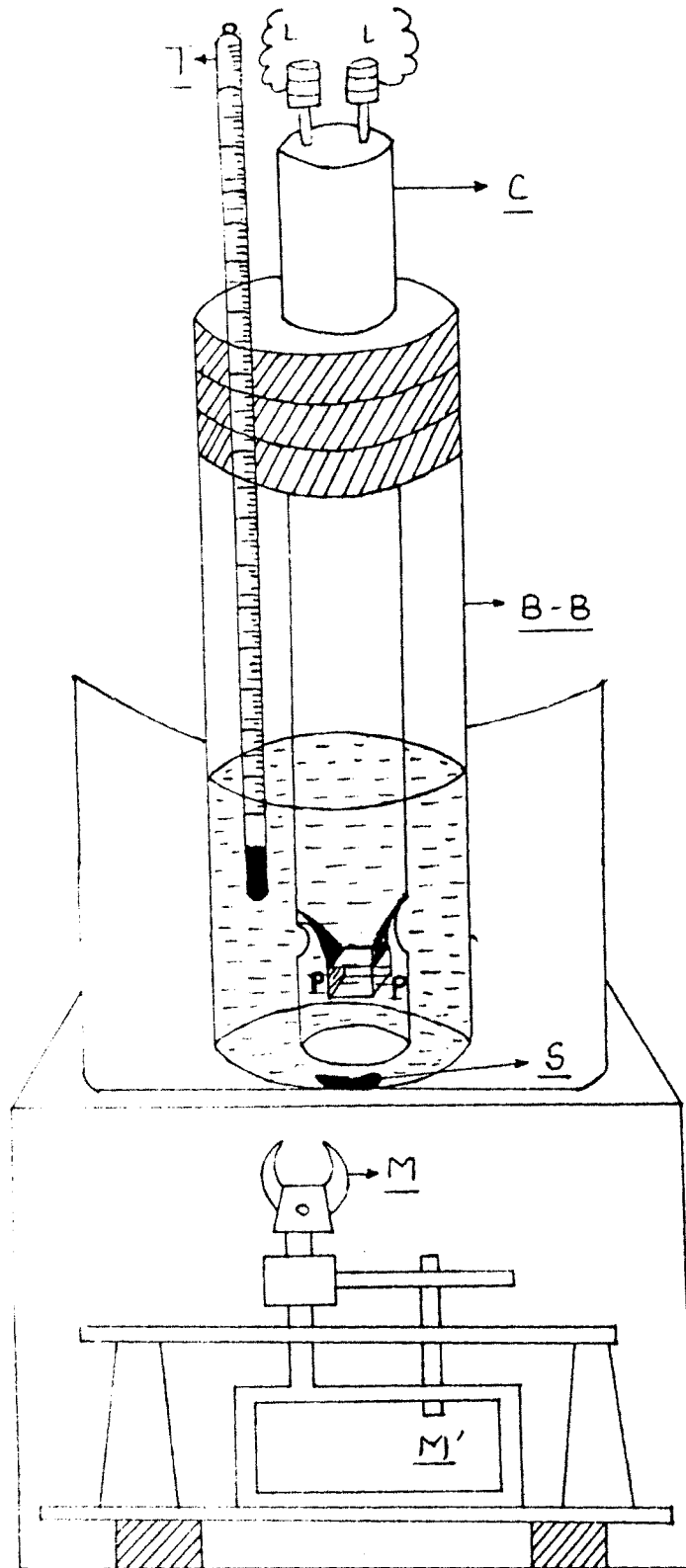
Conductometric measurements :

A precision digital conductivity meter model CM-180 of Elico private limited (Bombay) was used for the measurements of conductance of the solutions. The digital conductivity meter model CM-180 is a direct reading conductivity meter with 3½ digit light emitting diode (LED) digital panel meter.

A Berzelius beaker of corning glass of about 100 ml capacity was used as the cell. It was corked and through the cork Elico dip type conductivity cell (type CC-03) was introduced. About 20 to 25 ml solution was taken in the beaker to dip the electrodes of the conductivity cell properly. During the experiment the cell was placed in a thermostat which maintained the temperature 30°C or 25°C or 20°C or 15°C or 10°C or 5°C. within $\pm 0.1^\circ\text{C}$. The digital conductometer was adjusted to read the specific conductivity of the solution directly on the digital panel meter as per the instruction given by the manufacturer. Hence it may be mentioned here that the reading on digital panel meter read directly the specific conductivity of the solution under test. After fifteen minutes a series of readings were taken till three consecutive readings were constant. It was the specific conductivity of the solution directly shown by the digital panel meter. (DPM)

The molar conductivity of the solution is given by the relation (1).

$$\text{Molar conductivity} = \frac{\text{Specific conductivity} \times 1000}{\text{Molarity}} \quad \dots (1)$$



L-L- Leads, C-conductivity, M-Magnet, M'- Motor, S- stirrer,
T- Thermometer, P-P- Platinised platinum electrodes,
B-B- Berzelius beaker.

FIG- 2.1

Conductivity cell:

A dip type cell with platinised platinum electrodes was used. The cell constant was found to be 1.038 per cm. The conductivity cell was first cleaned with chromic acid and then washed with ample distilled water rinsed with conductivity water and finally rinsed with the solution whose conductivity was to be determined.

Preparation of conductivity water :

Conductivity water was prepared by redistillation of distilled water with alkaline (potassium hydroxide) potassium permanganate in a quickfit pyrex glass apparatus using a potassium hydroxide guard tube.

Purification of solvents :

Purification of ethanol :

Absolute ethanol was distilled first in a quickfit pyrex glass apparatus. It was then refluxed with calcium oxide for three hours, allowed to stand over night and then distilled again. Finally, ethanol thus obtained was redistilled with sodium metal in a quickfit pyrex glass apparatus using fractionating column and calcium chloride guard tube. Finally it was redistilled over tribromobenzoic acid to remove basic impurities.

Purification of acetone :

Acetone was first distilled in a quick fit pyrex glass apparatus using fractionating column. Distilled acetone was then kept with fused calcium chloride for three days and distilled in a quickfit pyrex glass apparatus fitted with fractionating column and calcium chloride guard tube.

Purification of methanol:

Methanol was purified by Lund and Bjerrum method (1967). Methanol was first distilled in a quick fit pyrex glass apparatus using fractionating column and calcium chloride guard tube. To a flask provided with a reflux condenser 0.5g of iodine, 5g of magnesium and 50 to 75 ml of methanol were added. The flask was then warmed until iodine disappeared. 0.5g of iodine was added more in case vigorous evolution of hydrogen did not occur. Mixture was then heated until all magnesium was converted into methylate. Additional 900ml of methanol were then added and the mixture was boiled for half an hour under reflux. The product was distilled using fractionating column and calcium chloride guard tube. Finally it was redistilled over tribromobenzoic acid to remove basic impurities.

Preparation of solutions:

A precision air damping balance, having sensitivity of 0.1mg was used for weighing the salts. Stock solutions, 250ml each of $1 \times 10^{-1} M$, $5 \times 10^{-2} M$, $1 \times 10^{-2} M$, $5 \times 10^{-3} M$, $1 \times 10^{-3} M$ for methanol - water; $1 \times 10^{-1} M$, $5 \times 10^{-2} M$, $1 \times 10^{-2} M$, $1 \times 10^{-3} M$, $5 \times 10^{-4} M$, $1 \times 10^{-4} M$ for ethanol-water and $1 \times 10^{-2} M$, $5 \times 10^{-3} M$, $1 \times 10^{-3} M$, $5 \times 10^{-4} M$, $1 \times 10^{-4} M$ for acetone water mixtures.

To prepare $1 \times 10^{-2} M$ solution of the salt and X % with respect to methanol, acetone or ethanol, 25ml of 0.1M stock solution were taken in a 250ml measuring flask by using a pipette to this solution in the flask 250-(2.5 x +25)ml of conductivity water were added and then 2.5 x ml of methanol, or acetone or ethanol were added and flask was well shaken. The solvents being associated solvents, a contraction in volume took place. The measuring

flask was then placed in a thermostat maintained at 30°C within $\pm 0.1^\circ\text{C}$. After 15 minutes, when the flask attained the temperature of the bath, the volume of solution was made upto the mark by adding conductivity water from a burette. The volume contraction was noted. Similarly the other concentrations were also prepared. In all, ten solutions of different percentages with respect to the non-aqueous solvents were prepared viz. 0, 10, 20, 30, 40, 50, 60, 70, 80 and 90.

After each weighing, the weighing bottle was cleaned with ample distilled water and dried before using for the next system.

AnalaR grade chemicals were used.

Following systems have been studied at 5°C, 10°C, 15°C, 20°C, 25°C and 30°C.

A) Electrolyte - Li₂SO₄ :

- 1) $1 \times 10^{-2}\text{M}$ Li₂SO₄ + X % ethanol
- 2) $5 \times 10^{-3}\text{M}$ Li₂SO₄ + X % ethanol
- 3) $1 \times 10^{-3}\text{M}$ Li₂SO₄ + X % ethanol
- 4) $1 \times 10^{-4}\text{M}$ Li₂SO₄ + X % ethanol
- 5) $5 \times 10^{-5}\text{M}$ Li₂SO₄ + X % ethanol
- 6) $1 \times 10^{-2}\text{M}$ Li₂SO₄ + X % methanol
- 7) $5 \times 10^{-3}\text{M}$ Li₂SO₄ + X % methanol
- 8) $1 \times 10^{-3}\text{M}$ Li₂SO₄ + X % methanol
- 9) $1 \times 10^{-3}\text{M}$ Li₂SO₄ + X % acetone
- 10) $5 \times 10^{-4}\text{M}$ Li₂SO₄ + X % acetone
- 11) $1 \times 10^{-4}\text{M}$ Li₂SO₄ + X % acetone
- 12) $5 \times 10^{-5}\text{M}$ Li₂SO₄ + X % acetone

B) Electrolyte - Na₂SO₄ :

- 13) 1 x 10⁻³ M Na₂SO₄ + X % ethanol
- 14) 1 x 10⁻⁴ M Na₂SO₄ + X % ethanol
- 15) 5 x 10⁻⁵ M Na₂SO₄ + X % ethanol
- 16) 1 x 10⁻⁵ M Na₂SO₄ + X % ethanol
- 17) 5 x 10⁻³ M Na₂SO₄ + X % methanol
- 18) 1 x 10⁻³ M Na₂SO₄ + X % methanol
- 19) 5 x 10⁻⁴ M Na₂SO₄ + X % methanol
- 20) 1 x 10⁻³ M Na₂SO₄ + X % acetone
- 21) 1 x 10⁻⁴ M Na₂SO₄ + X % acetone
- 22) 5 x 10⁻⁴ M Na₂SO₄ + X % acetone
- 23) 1 x 10⁻⁵ M Na₂SO₄ + X % acetone

C) Electrolyte - K₂SO₄ :

- 24) 1 x 10⁻³ M K₂SO₄ + X % ethanol
- 25) 1 x 10⁻⁴ M K₂SO₄ + X % ethanol
- 26) 5 x 10⁻⁵ M K₂SO₄ + X % ethanol
- 27) 1 x 10⁻⁵ M K₂SO₄ + X % ethanol
- 28) 1 x 10⁻³ M K₂SO₄ + X % methanol
- 29) 5 x 10⁻⁴ M K₂SO₄ + X % methanol
- 30) 1 x 10⁻⁴ M K₂SO₄ + X % methanol
- 31) 1 x 10⁻⁴ M K₂SO₄ + X % acetone
- 32) 5 x 10⁻⁵ M K₂SO₄ + X % acetone
- 33) X 1x10⁻⁵ M K₂SO₄ + X % acetone