CHAPTER - 2

EXPERIMENTAL

EXPERIMENTAL

CONDUCTOMETRIC MEASUREMENTS

A precision digital conductivity meter model CM-180 designed by Elico private limited, was used for the measurements of conductivity of the solutions. The digital conductivity meter model CM-180 is a direct reading conductivity meter provided with $3\frac{1}{2}$ digit light emitting diode (LED) panel meter.

A Berzelius beaker of corning glass of about 100ml capacity was used as the cell. It was corked and through the cork elico's dip-type conductivity cell was introduced. About 20-25 ml solution was taken in the beaker to dip the electrodes of conductivity cell properly. During the experiment the cell was placed in a thermostat which maintained the temperature at $30^{\circ}c$ or $25^{\circ}c$ or $20^{\circ}c$ or $15^{\circ}c$ or $10^{\circ}c$ or $5^{\circ}c$ within $\pm$ $0.1^{\circ}c$.

The digital conductometer was adjusted to read the specific conductance of the solution directly on the digital panel meter, as per the instructions given by the manufacturer. Hence it may be mentioned here that the reading on the digital panel meter directly read the specific conductance of the solution under test.

After 15 minutes a series of readings were taken till three consecutive readings were constant. It was the
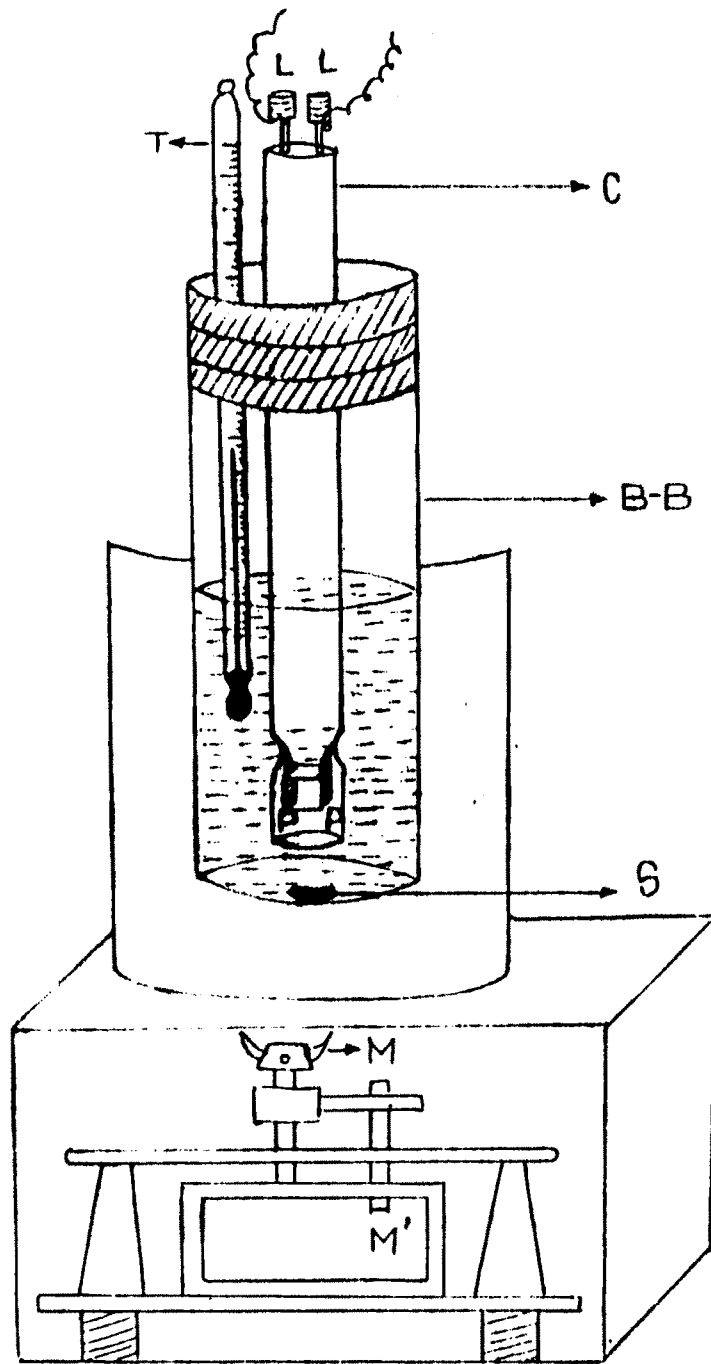
FIG - 2.1

L-L Leeds,   C - Conductivity Cell,

M' - Motor ,   S - Stirrer ,

T - Thermometer, B-B - Berzelius beaker,

P-P Platinised Platinum electrodes.

specific conductance of the solution directly shown by the digital panel meter.

The molar conductance is given by the relation

$$\text{Molar conductance} = \frac{\text{Specific conductance} \times 10^{3}}{\text{Molarity}} \quad \dots (1)$$

CONDUCTIVITY CELL

A dip-type conductivity cell with platinised platinum electrodes was used. The cell constant was found to be 0.982 per cm. The conductivity cell was first cleaned with cromic acid 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 quick-fit pyrex glass apparatus using a potassium hydroxide guard tube.

PURIFICATION OF SOLVENTS

Purification of ethanol

Absolute ethanol was distilled first in a quick-fit pyrex glass apparatus. It was then refluxed with calcium oxide for three hours, allowed to stand overnight and then distilled again. Ethanol thus obtained was redistilled with sodium metal in quick-fit pyrex glass apparatus using

fractionating column and calcium chloride guard tube. Finally it was redistilled over tribromobenzoic acid to remove basic impurities.

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 condensor 0.5 g of iodine, 5 g magnessium and 60 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 900 ml of methanol were then added and the mixture was boiled for half an hour under reflux. The product was distilled using fractionating column and clacium chloride guard tube. Finally it was redistilled over tribromo benzoic 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 quick-fit pyrex glass apparatus

fitted with fractionating column and calcium chloride guard tube.

PREPARATION OF SOLUTIONS

A precision air damping balance, having sensitivity of 0.2mg was used for weighing the salts. Stock solutions, 250ml each of $1x10^{-1}$ M, $5x10^{-2}$ M, $1x10^{-2}$ M for methanol-water, $1x10^{-1}$ M, $1x10^{-2}$ M, $5x10^{-3}$ M for acetone-water and $1x10^{-1}$ M, $1x10^{-2}$ M, $5x10^{-3}$ M, $1x10^{-3}$ M $5x10^{-4}$ M $1x10^{-4}$ M for ethanol-water mixtures were prepared in conductivity water by taking appropriate weight of the salt.

To prepare $1x10^{-2}$ M solution of the salt and X % with respect to methanol, acetone or ethanol, 25 ml of $1x10^{-1}$ M stock solution were taken in a 250ml measuring flask by using a pipette, to this solution in the flask $250-(2.5X'+25)$ 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 $30°c$ with in $\pm 0.1°c$. After 15 minutes, when the flask attained the temperature of the bath, the volume of solution was made up to 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 it 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.

(1) Electrolyte - NH_4Cl

1) 1×10^{-2} M NH_4Cl + ´ X ´ % ethanol.

2) 1×10^{-3} M NH_4Cl + ´ X ´ % ethanol.

3) 5×10^{-4} M NH_4Cl + ´ X ´ % ethanol.

4) 1×10^{-4} M NH_4Cl + ´ X ´ % ethanol.

5) 5×10^{-5} M NH_4Cl + ´ X ´ % ethanol.

6) 1×10^{-2} M NH_4Cl + ´ X ´ % methanol.

7) 5×10^{-3} M NH_4Cl + ´ X " % methanol.

8) 1×10^{-3} M NH_4Cl + ´ X ´ % methanol.

9) 1×10^{-2} M NH_4Cl + ´ X ´ % acetone.

10) 1×10^{-3} M NH_4Cl + ´ X ´ % acetone.

11) 5×10^{-4} M NH_4Cl + ´ X ´ % acetone.

(2) Electrolyte - $(CH_3)_2 NH_2 Cl$

12) 1×10^{-4} M $(CH_3)_2 NH_2Cl$ + X % ethanol.

13) 5×10^{-5} M $(CH_3)_2 NH_2Cl$ + X % ethanol.

14) 1×10^{-5} M $(CH_3)_2 NH_2Cl$ + X % ethanol.

15) 1×10^{-2} M $(CH_3)_2$ NH_2Cl + X % methanol.

16) 5×10^{-3} M $(CH_3)_2$ NH_2Cl + X % methanol.

17) 1×10^{-3} M $(CH_3)_2$ NH_2Cl + X % methanol.

18) 1×10^{-2} M $(CH_3)_2$ NH_2Cl + X % acetone.

19) 1×10^{-3} M $(CH_3)_2$ NH_2Cl + X % acetone.

20) 5×10^{-4} M $(CH_3)_2$ NH_2Cl + X % acetone.

(3) Electrolyte - (CH_3)$_4$ N.Cl.

21) 1×10^{-4} M (CH_3)$_4$ N.Cl + X % ethanol.

22) 5×10^{-5} M (CH_3)$_4$ N.Cl + X % ethanol.

23) 1×10^{-5} M (CH_3)$_4$ N.Cl + X % ethanol.

24) 1×10^{-2} M (CH_3)$_4$ N.Cl + X % methanol.

25) 5×10^{-3} M (CH_3)$_4$ N.Cl + X % methanol.

26) 1×10^{-3} M (CH_3)$_4$ N.Cl + X % methanol.

27) 1×10^{-2} M (CH_3)$_4$ N.Cl + X % acetone.

28) 1×10^{-3} M (CH_3)$_4$ N.Cl + X % acetone.

29) 5×10^{-4} M (CH_3)$_4$ N.Cl + X % acetone.