

## **CHAPTER III**

### **3.1. Source and Purification of the Chemicals used**

#### **3.1.1. Solvents**

Formamide ( $\text{H.CO.NH}_2$ , M.W. 45.04), S. D. Fine Chemical Limited, Mumbai, India, was purified by passing ammonia gas into the solvent until the solution is slight alkaline, dry acetone was added, filtered, dried over magnesium sulphate and then distilled. Purified formamide has boiling point 378.15 K /760 mmHg.<sup>1</sup>

2-methoxyethanol ( $\text{C}_3\text{H}_8\text{O}_2$ , M.W. 76.10), S. D. Fine Chemical Limited, Mumbai, India, was dried with potassium carbonate and distilled twice in an all-glass distillation set immediately before use. The middle fraction was collected for use.<sup>2</sup>

Acetophenone ( $\text{C}_8\text{H}_8\text{O}$ , M.W. 120.15), S. D. Fine Chemical Limited, Mumbai, India, was dried over anhydrous potassium carbonate for three days, filtered and then distilled. The middle fraction of the distillates was collected for use.<sup>3</sup>

Acetonitrile ( $\text{CH}_3\text{CN}$ , M.W. 41.05), S. D. Fine Chemical Limited, Mumbai, India, was distilled with  $\text{P}_2\text{O}_5$  and then redistilled over  $\text{CaH}_2$  in an all-glass distillation apparatus.<sup>4</sup> The middle fraction was collected for use.

1,2-dimethoxyethane ( $\text{C}_4\text{H}_{10}\text{O}_2$ , M.W. 90.12), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was purified by double-fractional distillation over  $\text{LiAlH}_4$  to eliminate traces of acids, peroxides and water; and the middle portion was collected for the preparation of mixtures.<sup>5</sup>

Dimethylsulfoxide ( $\text{C}_2\text{H}_6\text{SO}$ , M.W. 78.13), S. D. Fine Chemical Limited, Mumbai, India, was dried by standing overnight over freshly activated alumina, calcium hydride and calcium sulphate. The filtered solvent was then distilled over calcium hydride and stored over a 4Å molecular sieve.<sup>6</sup>

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Chlorobenzene ( $C_6H_5Cl$ , M.W. 112.56), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99.5%), was directly used without further purification.

Benzene ( $C_6H_6$ , M.W. 78.11), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was purified by means of a simple distillation technique with the first and last 20% of the distillate being discarded. Toluene ( $C_6H_5CH_3$ , M.W. 92.14), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was double distilled and the middle fraction was collected.<sup>7</sup>

Benzylchloride, ( $C_6H_5CH_2Cl$ , M.W. 126.59) and Aniline ( $C_6H_5NH_2$ , M.W. 93.13), S. D. Fine Chemical Limited, Mumbai, India, were purified according to the standard procedures.<sup>8</sup>

Benzaldehyde ( $C_6H_5CHO$ , M.W. 106.12), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 98%), was washed with 10 %  $Na_2CO_3$  (until no more  $CO_2$  evolved), then with saturated  $Na_2CO_3$  and water, followed by drying with anhydrous  $CaCl_2$ .

Nitrobenzene ( $C_6H_5NO_2$ , M.W. 123.11), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was purified by distillation and stored over activated 4 Å molecular sieves.<sup>7</sup>

Cyclohexane ( $C_6H_{12}$ , M.W. 84.16) and cyclohexanone ( $C_6H_{10}O$ , M.W. 98.15); S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), were purified by distillation and stored over activated 4 Å molecular sieves to reduce water content.

Methyl acetate ( $CH_3COOCH_3$ , M.W. 74.08), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was washed with a saturated solution of NaCl, dried with anhydrous  $MgCl_2$  and then distilled. The purified compound has boiling point 330.15 K /760 mmHg.<sup>1</sup>

Ethyl acetate ( $CH_3COOC_2H_5$ , M.W. 88.11), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was dried over  $K_2CO_3$ , filtered and distilled. The first and last portions of the distillate were discarded. The entire middle fraction was then distilled over  $P_2O_5$ . The purified liquid has a purity of about 99.7% and boils at 350.15 K /760 mmHg.<sup>1</sup>

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Propyl acetate ( $\text{CH}_3\text{COOC}_3\text{H}_7$ , M.W. 102.13), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99%), was purified by drying over  $\text{CaCO}_3$  overnight and was then filtered and freshly distilled.

Glycerol ( $\text{C}_3\text{H}_8\text{O}_3$ , M.W. 92.10), Merck, India (purity > 99.5%), was purified as described in the literature.<sup>9</sup>

Methanol ( $\text{CH}_3\text{OH}$ , M.W. 32.04, A. R. grade, purity > 99%), Merck, India, was dried over 4Å molecular sieves and then distilled fractionally. Middle fraction was collected and redistilled.<sup>10</sup>

n-amyl alcohol ( $\text{C}_5\text{H}_{11}\text{OH}$ , M.W. 88.15, A. R. grade, purity > 99%) and iso-amyl alcohol ( $\text{C}_5\text{H}_{11}\text{OH}$ , M.W. 88.15, A. R. grade, purity > 99%), Merck, India, were dried with anhydrous  $\text{K}_2\text{CO}_3$  and distilled. The middle fractions for both the liquids were collected and kept free from humidity with 3 Å molecular sieves.<sup>11</sup>

Water ( $\text{H}_2\text{O}$ , M.W. 18.016), was first de-ionized and then distilled in an all glass distilling set along with alkaline  $\text{KMnO}_4$  solution to remove any organic matter<sup>12</sup> therein. The doubly distilled water was finally distilled using an all glass distilling set. Precautions were taken to prevent contamination from  $\text{CO}_2$  and other impurities. The triply distilled water had specific conductance less than  $1 \times 10^{-6} \text{ S.cm}^{-1}$ .

Densities and viscosities of the purified solvents were in good agreement with the literature values<sup>12-18</sup> and are listed in the respective chapters. The purity of most of the solvents finally obtained was better than 99.5 %. All solvents, pure or mixed, were stored over 3 Å molecular sieves for three days before use.

### 3.1.2. *Solutes*

Lithium acetate ( $\text{CH}_3\text{COOLi}$ ,  $2\text{H}_2\text{O}$  M.W. 102.02), sodium acetate ( $\text{CH}_3\text{COONa}$ ,  $3\text{H}_2\text{O}$  M.W. 136.08) and potassium acetate ( $\text{CH}_3\text{COOK}$ , M.W. 98.14) were procured from Merck, India (A. R. grade); and purified as described in the literature.<sup>7</sup>

Sodium bromide ( $\text{NaBr}$ , M.W. 102.90), procured from Fluka puram, was used without further purification.

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Sodium tetrphenylborate ( $\text{NaB}(\text{C}_6\text{H}_5)_4$ ), M.W. 342.23), procured from Fluka puram, was recrystallized three times from acetone and dried under vacuum at 353.15 K for 72 hours.

Tetrabutylammonium bromide ( $(\text{C}_4\text{H}_9)_4\text{NBr}$ , M.W. 322.37), procured from Fluka puram, was purified by recrystallization from acetone and dried in vacuo at 333.15 K for 48 hours.<sup>19</sup>

Sodium molybdate ( $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ , M. W. 241.95) and sodium tungstate ( $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ , M. W. 329.86), E. Merck, India, were purified by re-crystallizing twice from the water prepared this way and then dried in a vacuum desiccator over  $\text{P}_2\text{O}_5$  for 24 hours before use.

Ammonium sulphate ( $(\text{NH}_4)_2\text{SO}_4$ , M.W. 132.14), Sodium sulphate ( $\text{Na}_2\text{SO}_4$ , M.W. 142.04), Potassium sulphate ( $\text{K}_2\text{SO}_4$ , M.W. 174.27), Magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , M.W. 246.48), Zinc sulphate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ , M.W. 287.54) and Cadmium sulphate ( $(\text{CdSO}_4) \cdot 8\text{H}_2\text{O}$ , M.W. 769.52) were procured from E. Merck, India (A. R. grade). They were used after drying over  $\text{P}_2\text{O}_5$  in a desiccator for few hours. The reagents were always placed in the desiccator over  $\text{P}_2\text{O}_5$  to keep them in dry atmosphere.

Nicotinamide ( $\text{C}_6\text{H}_6\text{N}_2\text{O}$ , M.W. 122.13), was purchased from Sigma Chemical Company, USA and used as such. Its mass purity as supplied by the vendor is 99%.

Nicotinic acid ( $\text{C}_6\text{H}_5\text{NO}_2$ , M.W. 123.11) and benzoic acid ( $\text{C}_6\text{H}_5\text{COOH}$ , M.W. 122.12) were purchased from Sigma Chemical Company, USA and used as received. Their purity as supplied by the vendor was 99%.

The purity of the solutes finally obtained was better than 99.0 % as checked by melting point determination.

Stock solutions of different salts/solutes in different mixed solvents and in pure solvents were prepared by mass and the working solutions were prepared by mass dilution. The conversion of molality into molarity was accomplished using the experimental density values. Great care was taken in minimizing evaporation losses and preventing moisture pick-up. All solutions were prepared afresh before use.

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### 3.1.3. *Mixed solvents*

The research work has been carried out with binary or ternary solvent systems with formamide, 1,2-dimethoxyethane, glycerol, acetonitrile, methanol, cyclohexane or cyclohexanone as primary solvents with some polar, weakly polar and non-polar solvents as well as with some solutes.

For the preparation of mixed binary and ternary mixtures, pure components were taken separately in glass stoppered bottles and thermostated at the desired temperature for sufficient time. When the thermal equilibrium was ensured, the required volumes of each component were transferred in a different bottle which was already cleaned and dried thoroughly. Conversion of required mass of the respective solvents to volume was accomplished by using experimental densities of the solvents at experimental temperature. It was then stoppered and the mixed contents were shaken well before use. While preparing different binaries and ternaries care was taken to ensure that the same procedure was adopted throughout the entire work. The physical properties of different pure and mixed solvents have been presented in the respective chapters.

The following different binary and ternary solvent mixtures have been prepared and used for my research studies.

#### **Binary mixtures studied:**

Formamide (1) + Acetophenone (2)

Formamide (1) + Acetonitrile (2)

Formamide (1) + 1,2-dimethoxyethane (2)

Formamide (1) + Dimethylsulphoxide (2)

Formamide (1) + 2-methoxyethanol (2)

1,2-dimethoxyethane (1) + Benzene (2)

1,2-dimethoxyethane (1) + Toluene (2)

1,2-dimethoxyethane (1) + Chlorobenzene (2)

1,2-dimethoxyethane (1) + Nitrobenzene (2)

1,2-dimethoxyethane (1) + Benzylchloride (2)

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1,2-dimethoxyethane (1) + Benzaldehyde (2)

1,2-dimethoxyethane (1) + Aniline (2)

Cyclohexane (1) + Methyl acetate (2)

Cyclohexane (1) + Ethyl acetate (2)

Cyclohexane (1) + Propyl acetate (2)

Cyclohexanone (1) + Methyl acetate (2)

Cyclohexanone (1) + Ethyl acetate (2)

Cyclohexanone (1) + Propyl acetate (2)

Cyclohexane (1) + Cyclohexanone (2)

### **Ternary mixtures studied:**

Cyclohexane (1) + Cyclohexanone (2) + Methyl acetate (3)

Cyclohexane (1) + Cyclohexanone (2) + Ethyl acetate (3)

Cyclohexane (1) + Cyclohexanone (2) + Propyl acetate (3)

### **Solute-solute / ion-ion and solute-solvent / ion-solvent interactions studied in the following mixed solutions:**

Glycerol (1) + Water (2) + Lithium acetate (3)

Glycerol (1) + Water (2) + Sodium acetate (3)

Glycerol (1) + Water (2) + Potassium acetate (3)

Glycerol (1) + Water (2) + Sodium bromide (3)

Glycerol (1) + Water (2) + Tetrabutylammonium bromide (3)

Glycerol (1) + Water (2) + Sodium tetraphenyl borate (3)

Acetonitrile (1) + Water (2) + Sodium molybdate (3)

Acetonitrile (1) + Water (2) + Sodium tungstate (3)

Formamide (1) + Water (2) + Ammonium sulphate (3)

Formamide (1) + Water (2) + Sodium sulphate (3)

Formamide (1) + Water (2) + Potassium sulphate (3)

Formamide (1) + Water (2) + Magnesium sulphate (3)

Formamide (1) + Water (2) + Zinc sulphate (3)

Formamide (1) + Water (2) + Cadmium sulphate (3)

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Tetrabutylammonium bromide(1) + Water(2)+ Nicotinamide(3)

Methanol (1) + n-amyl alcohol (2) + Nicotinic acid (3)

Methanol (1) + i-amyl alcohol (2) + Nicotinic acid (3)

Methanol (1) + n-amyl alcohol (2) + Benzoic acid (3)

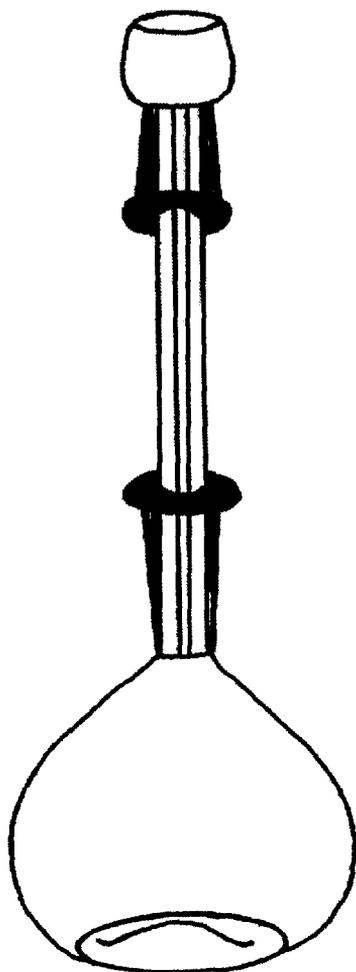
Methanol (1) + i-amyl alcohol (2) + Benzoic acid (3)

### 3.2. Experimental Methods

#### 3.2.1. Measurement of Density

Densities ( $\rho$ ) were measured with an Ostwald-Sprengel-type pycnometer having a bulb volume of 25 cm<sup>3</sup> and an internal diameter of the capillary of about 0.1 cm. The pycnometer was calibrated at 298.15, 308.15 and 318.15 K with doubly distilled water and benzene using density and viscosity values from the literature.<sup>8,20</sup> The pycnometer filled with air bubble free experimental liquid was kept vertically in a thermostatic water bath maintained at  $\pm 0.01$  K of the desired temperatures for few minutes to attain thermal equilibrium. The pycnometer was then removed from the thermostatic bath, properly dried, and weighed. Adequate precautions were taken to avoid evaporation losses during the time of actual measurements. An average of triplicate measurements was taken into account. Mass measurements accurate to  $\pm 0.01$  mg were made on a digital electronic analytical balance (Mettler, AG 285, Switzerland). The precision of the density measurement was  $\pm 3 \times 10^{-4}$  g.cm<sup>-3</sup>. Figure 1 shows an Ostwald-Sprengel-type pycnometer (Single arm) used in our works.

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**Figure 1:** An Ostwald- Sprengel-type pycnometer.

The temperature of the thermostatic water bath was preset at the desired temperature using a contact thermometer and relay system. The absolute temperature was determined by a calibrated platinum resistance thermometer and Muller bridge.<sup>21</sup> The solutions were prepared by mixing known volume of pure liquids in airtight-stopper bottles and each solution thus prepared was distributed into three recipients to perform all the measurements in triplicate, with the aim of the determining possible dispersion of the results obtained. Details of methods and techniques of density measurement is given in the literature.<sup>22-26</sup>

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### 3.2.2. Measurement of Viscosity

The kinematic viscosities were measured by means of a suspended-level Ubbelohde<sup>27</sup> viscometer. The time of efflux of a constant volume of the experimental liquid through the capillary was measured with the aid of a Racer stop watch capable of measuring times accurate to  $\pm 0.1$ s. The viscometer was always kept in a vertical position in the thermostatic bath with an accuracy of  $\pm 0.01$  K of the desired temperature. The efflux time for water at 298.15 K was measured to be 428.9 s. The flow times of pure liquids and liquid mixtures were measured a number of times and the average of the readings was taken into account. The kinematic viscosity ( $\nu$ ) and the absolute viscosity ( $\eta$ ) are given by the following equations:

$$\nu = Kt - L/t \quad (1)$$

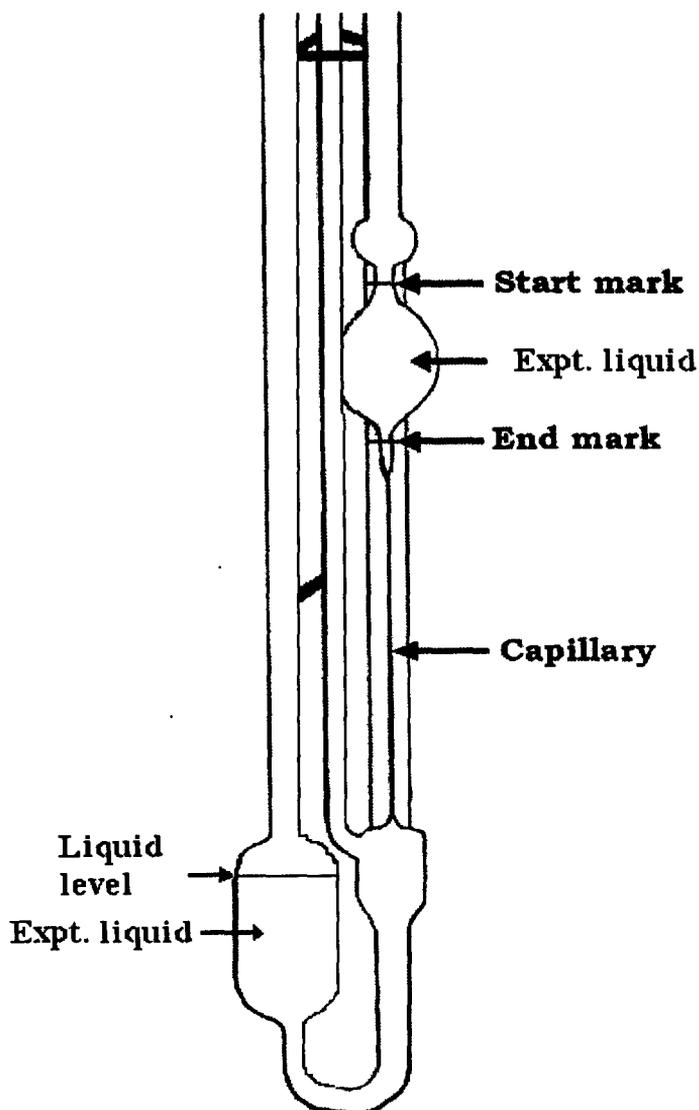
$$\eta = \nu\rho \quad (2)$$

where  $t$  is the average time of flow,  $\rho$  is the density and  $K$  and  $L$  are the characteristic constants of the particular viscometer. The values of the constants  $K$  and  $L$ , determined by using water and methanol as the calibrating liquids, were found to be  $2.1298 \times 10^{-3}$  and 8.7894, respectively. The kinetic energy corrections were done from these values and they were found to be negligible.

Relative viscosities ( $\eta_r$ ) were obtained using the equation:

$$\eta_r = \eta/\eta_0 = \rho t/\rho_0 t_0 \quad (3)$$

where  $\eta$ ,  $\eta_0$ ;  $\rho$ ,  $\rho_0$ ; and  $t$ ,  $t_0$  are the absolute viscosities, densities and flow times for the solution and solvent, respectively. The uncertainty in the viscosity measurements, based on our work on several pure liquids, was  $\pm 2 \times 10^{-4}$  mPa.s. Figure 2 shows a suspended-level Ubbelohde viscometer used in our works.



**Figure 2:** A suspended-level Ubbelohde viscometer.

### 3.2.3. Measurement of Ultrasonic Speed

Ultrasonic speeds were measured, with an accuracy of 0.3 %, using a single-crystal variable-path ultrasonic interferometer<sup>28</sup> (Model M-81, Mittal Enterprise, New Delhi) operating at 2 MHz, which was calibrated with water, methanol and benzene at the experimental temperature. The temperature stability was maintained within  $\pm 0.01$  K of the desired temperature by circulating thermostatic water around the cell with the aid of a circulating pump.

The principle used in the measurement of the ultrasonic speed ( $u$ ) is based on the accurate determination of the wavelength ( $\lambda$ ) in the

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medium. Ultrasonic waves of known frequency ( $f$ ) are produced by a quartz crystal fixed at the bottom of the cell. These waves are reflected by a movable metallic plate kept parallel to the quartz crystal. If the separation between these two plates is exactly a whole multiple of the sound wavelength, standing waves are formed in the medium. This acoustic resonance originates an electrical reaction on the generator driving the quartz crystal and the anode current of the generator becomes a maximum.

If the distance is increased or decreased maintaining the variation of exactly one half of wave length ( $\lambda/2$ ) or integral multiple of it, the anode current becomes maximum. From the knowledge of the wave length ( $\lambda$ ), the speed ( $u$ ) can be obtained by the relation:

$$u = \lambda \times f \quad (4)$$

The ultrasonic interferometer consists of the following two parts:

(i) the high frequency generator and (ii) the measuring cell. The measuring cell is connected to the output terminal of the high frequency generator through a shielded cable. The cell is filled with the experimental liquid before switching on the generator. The ultrasonic waves move normal from the quartz crystal till they are reflected back from the movable plate and the standing waves are formed in the liquid in between the reflector plate and the quartz crystal. The micrometer is slowly moved till the anode current on the meter on the high frequency generator deflects a maximum. A number of maxima of anode current are observed and their number ( $n$ ) is counted. The total distance ( $d$ ) thus moved by the micrometer gives the value of the wavelength ( $\lambda$ ) with the following relation.

$$d = n \times \lambda/2 \quad (5)$$

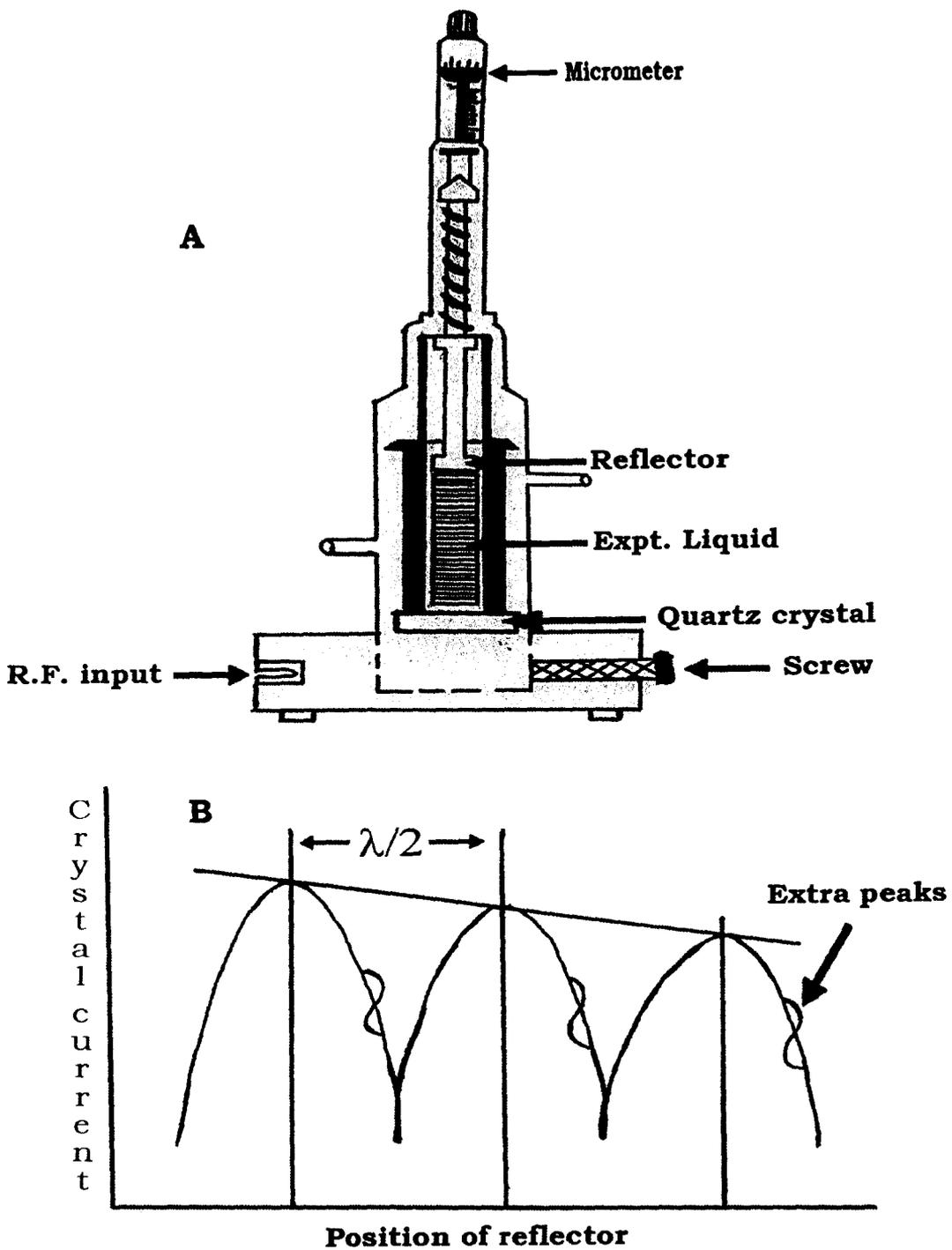
Further, the speed ( $u$ ) determined thus is used for the calculation of the isentropic compressibility ( $\kappa_s$ ) using the following formula:

$$\kappa_s = (u^2 \rho)^{-1} \quad (6)$$

where  $\rho$  is the density of the experimental liquid.

In figure 3- (A) Cross-section of the measuring cell of a Multi-frequency Ultrasonic Interferometer and (B) Position of reflector vs. crystal current are depicted.

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**Figure 3:** (A) Cross-section of the measuring cell of a Multi-frequency Ultrasonic Interferometer and (B) Position of reflector vs. crystal current.

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However, the extra peaks (Figure 3B) in between minima and maxima occurs due to a number of reasons, but these do not effect the value of  $\lambda/2$ .

### 3.2.4. Measurement of Conductance

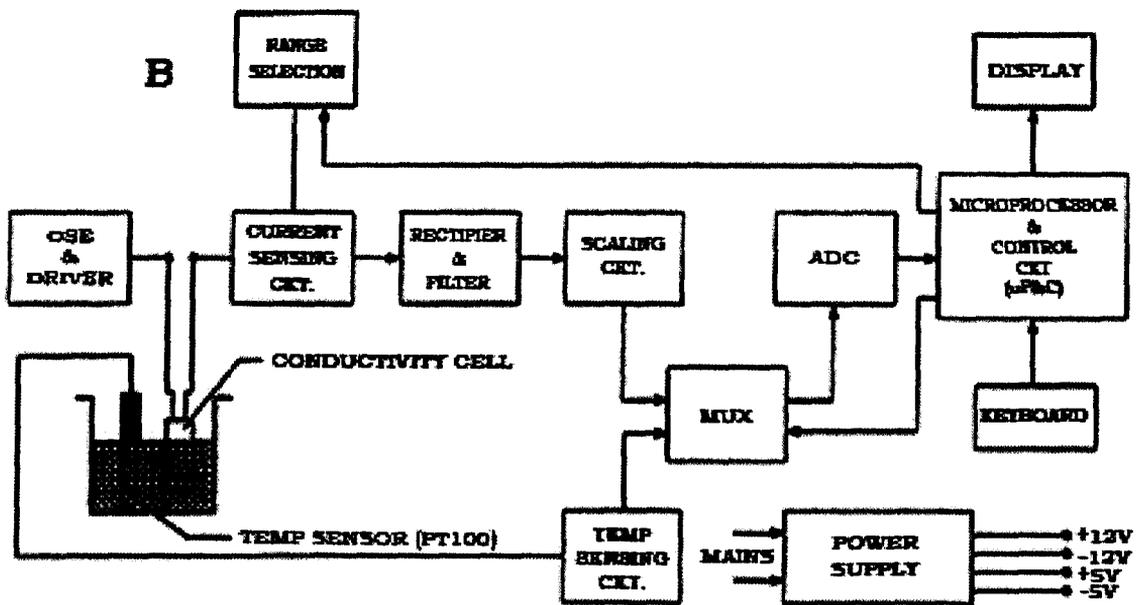
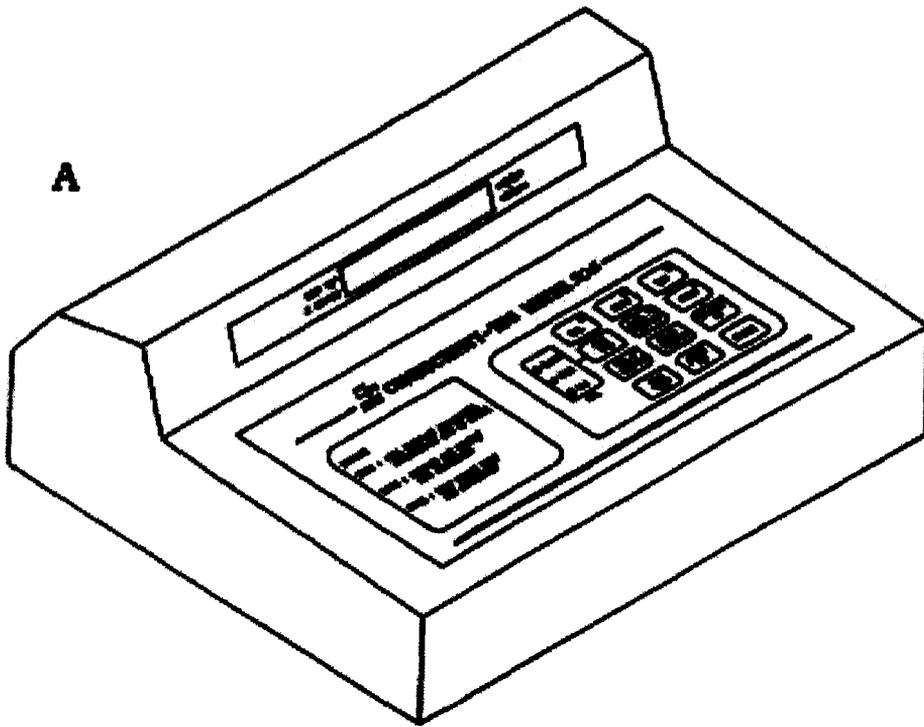
Systronic Conductivity meter- 308 is used for measuring specific conductivity of electrolytic solutions. It is a microprocessor based instrument and can provide both automatic and manual temperature compensation. The instrument shows the conductivity of the solution under test at the existing temperature or with temperature compensation. Provision for storing the cell constant and the calibrating solution type, is provided with the help of battery back-up. This data can be further used for measuring the conductivity of an unknown solution without recalibrating the instrument even after switching it off.

The conductance measurements were carried out on this conductivity bridge using a dip-type immersion conductivity cell, CD-10 with a cell constant of  $1.0 \pm 10\% \text{ cm}^{-1}$ . The instrument was standardized using 0.1 (M) KCl solution. The cell was calibrated by the method of Lind and co-workers.<sup>29</sup> The measurements were made in a thermostatic water bath maintained at the required temperature with an accuracy of  $\pm 0.01 \text{ K}$ .

Several solutions were prepared by mass accurate to  $\pm 0.01 \text{ mg}$  with the aid of a Mettler electronic analytical balance (AG 285, Switzerland) and runs were performed to ensure the reproducibility of the results. Due correction was made for the specific conductance of the solvents at desired temperatures.

Figure 4 shows the Systronic Conductivity meter- 308, i.e., (A) Isometric view and (B) Block diagram of the instrument.

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**Figure 4:** The Systronic Conductivity meter 308- (A) Isometric view and (B) Block diagram of the instrument.

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