

## CHAPTER III

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

#### **3.1.1. Solvents**

1,4-dioxane ( $C_4H_8O_2$ , M.W. 88.11), 1,3-dioxolane ( $C_3H_6O_2$ , M.W. 74.08), ethylenediamine ( $C_2H_8N_2$ , M.W. 60.09), 1,2-dichloroethane ( $C_2H_4Cl_2$ , M. W. 98.97), monoethanolamine ( $C_2H_7NO$ , M.W. 61.08), 2-ethyl-1-hexanol ( $C_8H_{18}O$ , M.W. 130.23), cyclohexane ( $C_6H_{12}$ , M.W. 84.16), cyclohexanone ( $C_6H_{10}O$ , M.W. 98.15), methyl acetate ( $C_3H_6O_2$ , M.W. 74.08), ethyl acetate ( $C_4H_8O_2$ , M.W. 88.11), methyl salicylate ( $C_8H_8O_2$ , M.W. 152.15), tetrahydrofuran ( $C_4H_8O$ , M.W. 72.11) and dimethylformamide ( $C_3H_7NO$ , M.W. 73.10) were used as solvents for preparing different binary/ternary/quaternary solvent mixtures in the research works embodied in this thesis.

Dimethylformamide was purified by distilling the azeotropes obtained by mixing it with a 10% of benzene by volume at a temperature of 80 °C under atmospheric pressure.<sup>1</sup> The product thus obtained was dried over silica gel followed by distillation under reduced pressure. The collected middle fractions were stored in a desiccator over  $P_2O_5$  before use.<sup>1</sup> Tetrahydrofuran was purified by distilling it over  $LiAlH_4$  after keeping it for several days over  $KOH$ .<sup>2</sup> 1,3-dioxolane was purified by refluxing it for two hours with  $PbO_2$ . It was cooled and filtered, the filtrate was then fractionally distilled after mixing with xylene and  $PbO_2$ .<sup>3</sup> Similarly 1,4-dioxane was purified by refluxing it with concentrated  $HCl$  and water for about 12 hours by passing nitrogen slowly. The product obtained was cooled, treated with  $KOH$  solution slowly with constant shaking until a second layer was separated. 1,4-dioxane was decanted and again treated with fresh  $KOH$  pellets to remove the aqueous phase if any. It was then transferred to a new clean flask and refluxed with sodium for 6-12 hours and then distilled from it.<sup>4</sup> All other solvents were of spectroscopic grade and were used as received from the vendors. The purity of these solvents was ascertained by comparing their densities and viscosities to literature values<sup>5-39</sup> as presented in the respective chapters wherein they were used. Provenance and purity of these solvents are given in Table 3.1.

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**Table 3.1.** Provenance and purity of the solvents used.

Chemical	Source	Mass Fraction purity	CAS No.
1,4-dioxane	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	123-91-1
	Merck, India	>0.99	
1,3-dioxolane	S.D. Fine Chemicals, India	>0.99	646-06-0
Ethylenediamine	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	107-15-3
1,2-dichloroethane	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	107-06-2
Monoethanolamine	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	141-20-9
2-ethyl-1-hexanol	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	104-76-7
Cyclohexane	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	110-82-7
Cyclohexanone	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	108-94-1
Methyl acetate	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	79-20-9
Ethyl acetate	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	141-78-6
Methyl salicylate	Sigma Aldrich, Germany	>0.998 <sup>a</sup>	119-36-8
N, N-dimethylformamide	S.D. Fine Chemicals, India	>0.99	68-12-2
Tetrahydrofuran	Merck, India	>0.99	109-99-9

<sup>a</sup>Moisture content as prescribed by the respective vendors were 0.05%

#### 3.1.2. Mixed Solvents

In this research work the binary/ternary/quaternary mixtures required for the experiments were prepared by mass in specially designed air-tight bottles with stopper inside a dry box at 298.15 K. The binary mixtures containing 10, 20, 30, 40, 50, 60, 70, 80, 90 mass% of component 1 were prepared at 298.15 K so as to cover the whole composition range. The binary mixtures selected for the investigations are as follows: (i) 1,4-dioxane with ethylenediamine, 1,2-dichloroethane and monoethanolamine, (ii) 2-ethyl-1-hexanol with ethylenediamine, 1,2-dichloroethane and monoethanolamine, (iii) cyclohexane with methyl acetate, ethyl acetate and methyl salicylate, (iv) cyclohexanone with methyl acetate, ethyl acetate and methyl salicylate. The ternary mixtures containing varying mass% of the components were also prepared at 298.15 K in order to cover the whole range of mass fractions for each component as much as possible. The ternary mixtures studied are as follows: (i) cyclohexane + methyl acetate + ethyl acetate, (ii) cyclohexane + methyl acetate + methyl salicylate, (iii) cyclohexane + ethyl acetate + methyl salicylate, (iv) methyl acetate + ethyl acetate +

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methyl salicylate, (v) cyclohexanone + methyl acetate + ethyl acetate, (vi) cyclohexanone + methyl acetate + methyl salicylate and (vii) cyclohexanone + ethyl acetate + methyl salicylate. Similarly the quaternary mixtures were prepared with varying mass% of the components containing: (i) cyclohexane + methyl acetate + ethyl acetate + methyl salicylate and (ii) cyclohexanone + methyl acetate + ethyl acetate + methyl salicylate at 298.15 K.

### 3.2. Experimental Methods

#### 3.2.1. Density Measurement

The densities of the experimental liquids or liquid mixtures were measured at the experimental temperatures using a digital density meter (Anton Paar, DMA 4500M) as shown in Figures 3.1-3.4.



Fig. 3.1. Anton Paar densitometer (DMA 4500M).

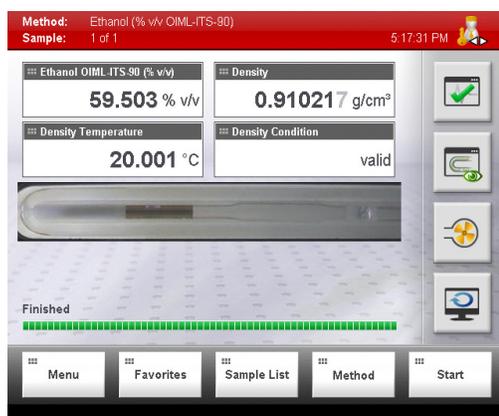


Fig. 3.2. Main screen view of Anton Paar densitometer.

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**Fig. 3.3.** Filling of sample with a syringe.



**Fig. 3.4.** Drying the measuring cell.

In the digital density meter, the mechanic oscillation of the U-tube is electromagnetically transformed into an alternating voltage of the same frequency. The period of oscillation ( $\tau_0$ ) can be measured with high resolution and it follows a simple relation to the density ( $\rho$ ) of the sample in the oscillator:<sup>40</sup>

$$\rho = A\tau_0^2 - B \quad (1)$$

where A and B are the respective instrument constants of each oscillator. Their values are determined by calibrating with two liquids of precisely known densities  $\rho_1$  and  $\rho_2$ . For the special adjustment the densities of these two liquids must differ by at least  $\pm 0.01 \text{ g} \cdot \text{cm}^{-3}$  and  $\tau_0$  values of the adjustment media must differ by at least 0.0001. Modern instruments calculate and store the constants A and B after the two calibration measurements, mostly performed with air and water. They employ suitable measures to compensate various parasitic influences on the measuring result, *e.g.*, the influence of the sample's viscosity and the non-linearity caused by the measuring instrument's finite mass. The density meter was calibrated at the experimental

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temperatures with doubly distilled, degassed water and dry air at atmospheric pressure. The temperature was kept constant with an accuracy of  $\pm 1 \times 10^{-2}$  K by using an automatic built-in Peltier technique. The stated repeatability and accuracy of the densities were  $\pm 1 \times 10^{-5}$  g · cm<sup>-3</sup> and  $\pm 5 \times 10^{-5}$  g · cm<sup>-3</sup>, respectively. However, when the accuracy of the measured densities was tested with the density of an aqueous NaCl solution of known molality using the data given by Pitzer,<sup>41</sup> the estimated uncertainty of the density measurements for most of the solutions was found to be better than  $\pm 2 \times 10^{-5}$  g · cm<sup>-3</sup>.

### 3.2.2. Measurement of Viscosity

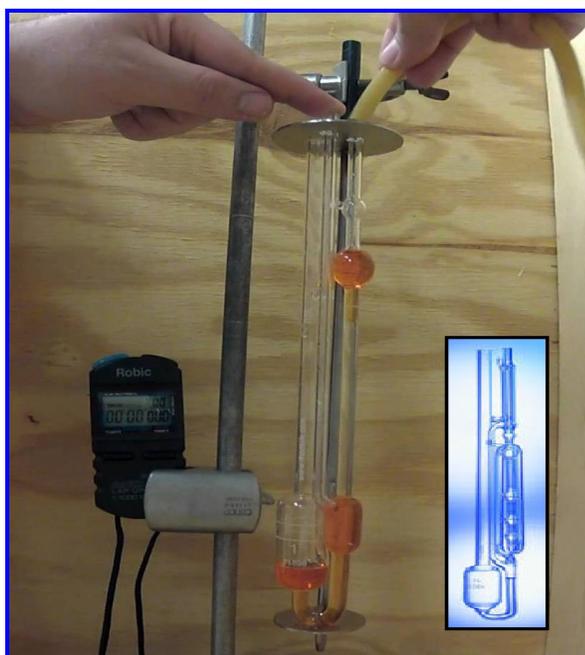
The kinematic viscosities were measured by means of a suspended Cannon type Ubbelohde viscometer (capillary viscometer). The time of efflux of a constant volume of the experimental liquid mixtures through the capillary was measured with the aid of a digital stopwatch capable of measuring time accurate to  $\pm 0.01$ s. The viscometer was always kept immersed in a vertical position in the thermostatic bath with an accuracy of  $\pm 0.01$  K of the desired temperature. After attainment of thermal equilibrium, the flow times of pure liquids or liquid mixtures were measured thrice and the average of the readings were taken into account. During the measurements adequate precautions were taken to minimize evaporation losses. The efflux time for water at 298.15 K was measured to be 428.9 s. The kinematic viscosity ( $\nu$ ) and the absolute viscosity ( $\eta$ ) are obtained using the following equations:

$$\nu = kt - \frac{L}{t} \quad (2)$$

$$\eta = \nu\rho \quad (3)$$

where  $k$  and  $L$  are the viscometer constants and  $t$  and  $\rho$  are the efflux time of flow in seconds and the density of the experimental liquid, respectively. The values of the constants  $k$  and  $L$ , determined by using water and methanol as the calibrating liquids, were found to be  $1.9602 \times 10^{-3}$  and 4.2019, respectively. The kinetic energy corrections were done from these values and they were found to be negligible. The uncertainty in viscosity measurements, based on our work on several pure liquids, was within  $\pm 4 \times 10^{-4}$  mPa · s. Figure 3.5 depicts the capillary viscometers used for measuring viscosities of the solutions studied.

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**Fig. 3.5.** A suspended-level Cannon type Ubbelohde viscometer (capillary viscometer).

### 3.2.3. Measurement of Mass

Mass measurements were made on digital electronic analytical balance (Mettler Toledo, AG 285, Switzerland) as shown in Figure 3.6.



**Fig. 3.6.** Digital electronic analytical balance (Mettler Toledo, AG 285).

It can measure mass with a very high precision and accuracy. The weighing pan is inside a transparent enclosure with doors so that dust particles do not collect and any air currents in the room do not affect the operation of the balance. The mass measurements were accurate to  $\pm 0.01$  mg.

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### 3.2.4. Refractive Index Measurement

Refractive indices of the experimental liquid mixtures (binary/ternary/quaternary) were measured with an Abbe-refractometer (Cyberlab, MA01527, USA) using yellow sodium D-line light ( $\lambda=589.3$  nm, which is the average of the two emission lines at 589.0 nm and 589.6 nm) at 298.15 K. The Abbe-refractometer is one of the most convenient and widely used refractometer and owes its popularity to its wide range ( $n_D = 1.3$  to 1.7) and to the minimal sample needed. Figure 3.7 shows a schematic diagram of its optical system. The sample liquid is directly injected into the prism assembly of the instrument using an airtight hypodermic syringe and is sandwiched as a thin layer ( $\sim 0.1$ mm) between two prisms. The upper prism is firmly mounted on a bearing that allows its rotation by means of the side arm shown in dotted lines. The lower prism is hinged to the upper to allow separation for cleaning and for introduction of the sample. When light reflects into the prism (the lower surface being rough) effectively becomes the source for an infinite number of rays that passes through the sample at all angels.

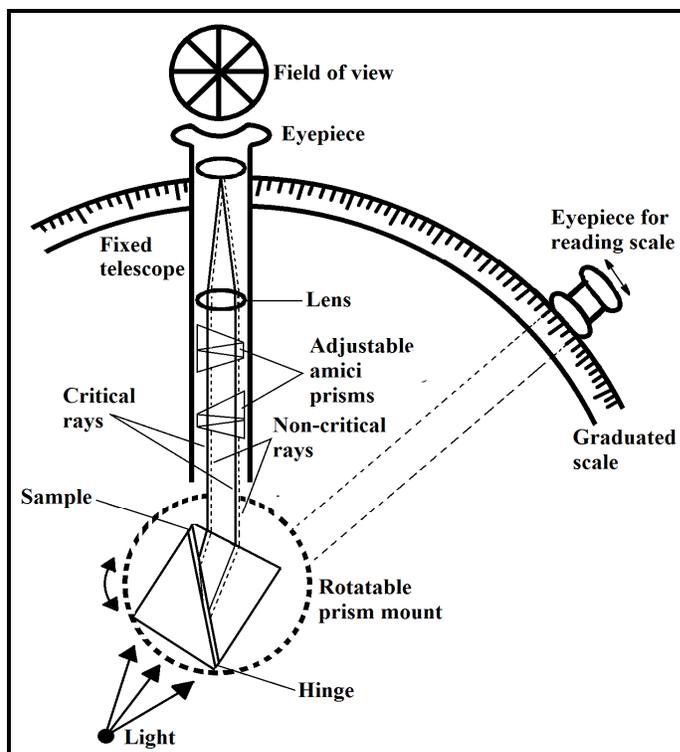
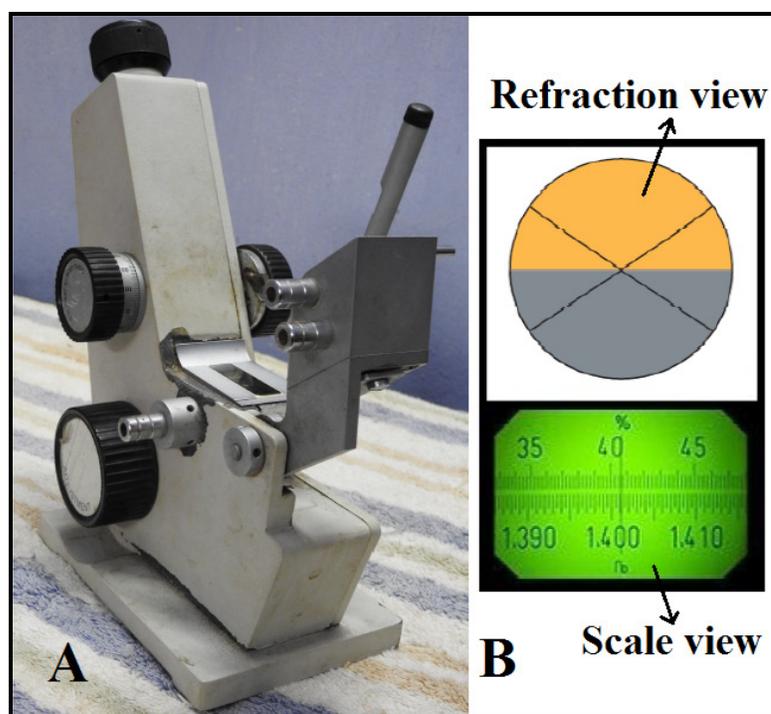


Fig 3.7. Schematic diagram of Abbe-refractometer optical system.

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The light rays get refracted at the interface of the sample and the smooth-ground face of the upper prism. After this it passes through the fixed telescope. Two triangular prisms in contact (Amici prisms) serve to collect the divergent rays of different colors into a single white beam that corresponds in path to that of the sodium D ray. The eyepiece of the telescope is provided with crosshairs as shown in Figure 3.8. During the measurement of the refractive index the prism angle is changed until the light-dark interface just coincides with the crosshairs. The position of the prism is then established from the fixed scale. The Abbe-refractometer is depicted in Figure 3.8. Water was circulated through the refractometer from a thermostatic bath maintained at  $298.15 \pm 0.01$  K. It was calibrated by measuring the refractive indices of doubly distilled, degassed water at 298.15 K.<sup>42,43</sup> The uncertainty in refractive indices was within  $\pm 0.0002$ . An average of three measurements was taken for each mixture.



**Fig. 3.8.** A: Abbe-refractometer (Cyberlab, MA01527, USA); B: View of the refractometer through the eyepiece.

#### 3.2.5. Measurement of Ultrasonic Speed

There are mainly three experimental techniques to measure the velocity of ultrasonic sounds in pure liquids and liquid mixtures. They are: (i) Continuous wave

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technique, (ii) Pulse technique and (iii) Interferometer method. From a comparison of the relative merits of the various methods, interferometer method is found to be the most accurate method available for speed measurements. Hunter and Dardy<sup>44</sup>, Dobbs and Fine gold<sup>45</sup>, Fort and Moore<sup>46</sup> measured the speed of sound for liquids and liquid mixtures by using interferometric technique with  $\pm 0.15\%$  uncertainty. In the present research work ultrasonic speeds of the experimental liquid mixtures were measured with an accuracy of 0.3% using a multi-frequency ultrasonic interferometer (F-05, Mittal Enterprises, New Delhi, India) operating at 2 MHz. It was calibrated with doubly distilled degassed water and purified methanol or benzene maintained at 298.15 K by circulating thermostatic water around a jacketed cell (2 MHz) containing the experimental liquids with a circulating pump. The uncertainty in ultrasonic speeds<sup>47</sup> was around  $\pm 0.2 \text{ m s}^{-1}$ .

The measurement of the ultrasonic speed ( $u$ ) by ultrasonic interferometer is based on the accurate determination of the wavelength ( $\lambda$ ) in the medium. In this process ultrasonic waves of known frequency ( $f$ ) are generated by a quartz crystal fixed at the bottom of the cell. These waves are then reflected by a movable metallic plate, which is kept parallel to the quartz crystal. If the distance between these two plates is exactly a whole multiple of the sound wavelength, standing waves are formed in the medium. Under these circumstances, acoustic resonance occurs and this acoustic resonance gives rise to an electrical reaction on the generator driving the quartz crystal and the anode current of the generator becomes a maximum. But If the distance is increased or decreased by exactly one half of the wavelength ( $\lambda/2$ ) or an integer multiple of it, the anode current again becomes maximum. If  $d$  is the separation between successive adjacent maxima of anode current and the total number of oscillation (usually  $n = 20$ ) counted. Then the total distance moved by the micrometer in  $n$  oscillations is given by:

$$d = n \times \frac{\lambda}{2} \quad (4)$$

Now the speed ( $u$ ) of the wave and frequency ( $f$ ) of the cell is related to its wavelength ( $\lambda$ ) by the relation,

$$u = \lambda \times f \quad (5)$$

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$$\text{or } u = \lambda \times f = \frac{2d}{n} \times f \quad (6)$$

Thus with a known frequency of the cell the ultrasonic speed ( $u$ ) can be obtained. The ultrasonic interferometer consists of the following three parts: (i) The high frequency generator (Single and Multi-frequency), specially designed to excite the quartz crystal fixed at the bottom of the measuring cell at its resonant frequency to generate ultrasonic wave in the experimental liquid filled in the measuring cell, (ii) The measuring cell (1, 2, 3 and 4 MHz) is specially designed double walled cell for maintaining the temperature of the liquid sample constant during the experiment. A fine micrometer has been provided at the top, which can lower or raise the reflector plate in the liquid in the cell through a known distance and (iii) shielded cable.

The total assembly is shown Figure 3.9 in which 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 shows a maximum deflection. A number of maxima of anode current are observed and the total number of oscillation ( $n$ ) is counted. The total distance ( $d$ ) thus moved by the micrometer gives the value of the wavelength ( $\lambda$ ) using the Eq. (4).



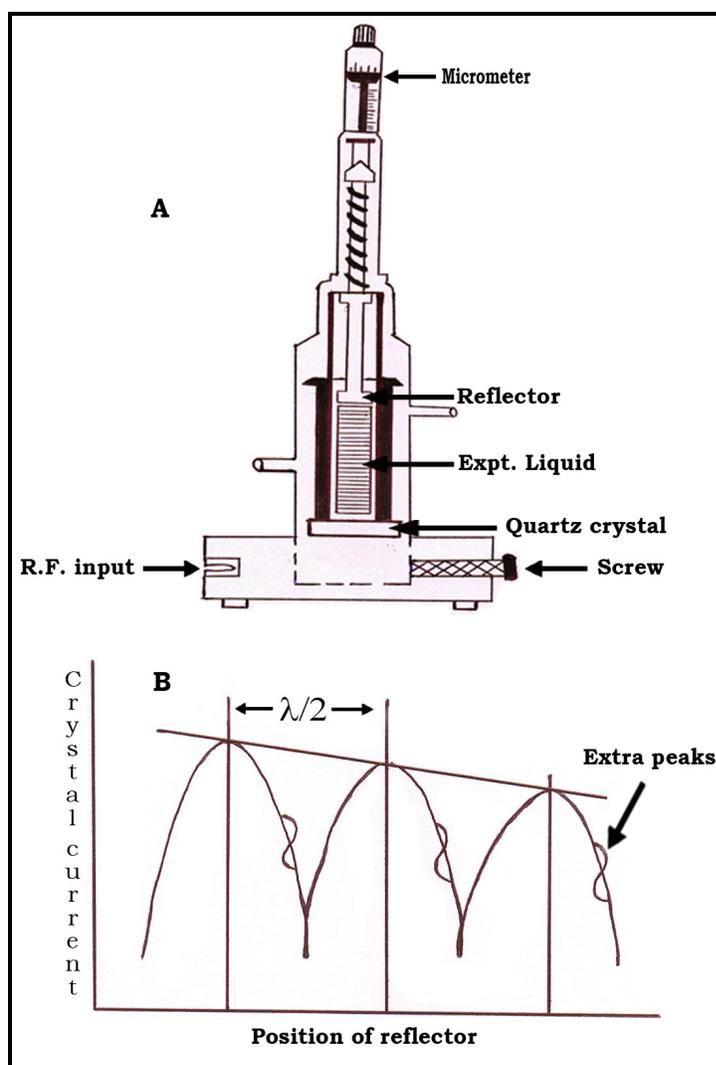
**Fig 3.9.** ultrasonic interferometer (F-05, Mittal Enterprises, New Delhi, India).

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

$$K_s = 1/(u^2 \rho) \quad (7)$$

where  $\rho$  is the density of the experimental liquid. In Figure 3.10(A) cross-section of the measuring cell of a multi-frequency ultrasonic interferometer and (B) position of reflector *versus* crystal current are depicted. However, the extra peaks [Figure 3.10(B)] in between minima and maxima occur due to a number of reasons but these do not effect the  $\lambda/2$  values.



**Figure 3.10.** (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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### 3.2.5. Measurement of FTIR Spectra

Fourier transform infrared spectroscopy (FTIR)<sup>48</sup> is a technique which is used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. The term Fourier transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum. In the present study FT-IR spectra of the experimental liquid mixtures (binary/ternary/quaternary) were recorded at 298.15 K using Elmer FT-IR spectrophotometer (Spectrum RX-1) in the range 400-4000  $\text{cm}^{-1}$  and is depicted in Figure 3.11. In our study the experimental liquid mixtures were sandwiched between two potassium bromide aperture plates<sup>49</sup> (Sigma-Aldrich, Germany) taking an adequate precaution such that no gas bubbles are trapped. The thickness of the liquid membrane between the KBr aperture plate is adjusted using spacer of fixed path length and by appropriately tightening the screws (without breaking the aperture plates). These plates are transparent to the infrared light and do not introduce any lines onto the spectra. This type of cell is often called a "liquid cell." Figure 3.12 shows the appearance of a liquid cell.



**Fig. 3.11.** Perkin-Elmer FTIR spectrophotometer (Spectrum RX-1).

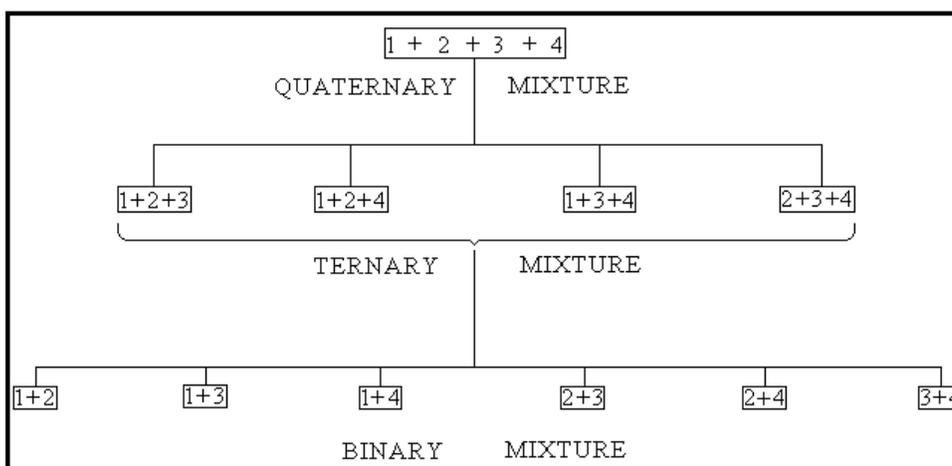
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**Fig. 3.12.** Appearance of a Liquid Cell.

### 3.2.6. Methodology

To reveal the nature and extent of molecular interactions in liquid mixtures various properties like density, viscosity, ultrasonic speed and refractive index were measured and various excess properties were determined by using standard relations as available in literatures, infra red (IR) spectroscopic technique was used to study molecular interactions and structural changes in the liquid mixtures. To study molecular interactions in a quaternary system, information on various properties of the binary and ternary mixtures of the components comprising the quaternary system is required. So a bottom to top method of study as shown in Figure 3.13 was adopted.



**Fig. 3.13.** Hierarchy of combination of different solvent mixtures.

Once experimental data for such systems are measured various excess or deviation and thermodynamic properties were derived and discussed in terms of various specific and nonspecific forces, various empirical and semi empirical models for excess molar volume, speed of sound, refractive index, *etc.*, were evaluated for their applicability.

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The equation of states like PREOS along with PFP model were used for binary mixtures. However, response surface model<sup>50</sup> may be utilized to determine the partial molar volume and partial molar refraction for the ternary or quaternary liquid mixtures.

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