

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), Merck, India, was kept several days over potassium hydroxide (KOH), followed by refluxing over excess of sodium for 12 hours. Finally, it was distilled from sodium [1].

Tetrahydrofuran (C_4H_8O , M.W: 72.11), Merck, India, for synthesis, was kept several days over potassium hydroxide (KOH), refluxed for 24 hours and distilled over $LiAlH_4$ [2].

Methanol (CH_3OH , M.W: 32.04, A.R. grade) Merck, India, was dried over $4A^{\circ}$ molecular sieves and then distilled fractionally. Middle fraction was collected and redistilled [3].

n-amyl alcohol ($C_5H_{11}OH$, M.W: 88.15, A.R. grade) and iso-amyl alcohol ($C_5H_{11}OH$, M.W:88.15, A.R. grade) Merck, India, were dried with anhydrous K_2CO_3 . The middle fractions for both the liquids were collected and kept free from humidity with $3A^{\circ}$ molecular sieves [4].

Benzene (C_6H_6 , M.W: 78.11) Merck, India, 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) Merck, India, was double distilled and the middle fraction was collected [5].

Nitrobenzene ($C_6H_5NO_2$, M.W: 123.11) Merck, India, was purified by distillation and stored over $4A^{\circ}$ molecular sieves [5].

Cyclohexane (C_6H_{12} , M.W: 84.16) and cyclohexanone ($C_6H_{10}O$, M.W: 98.15) Merck, India, were purified by distillation and stored over activated $4A^{\circ}$ molecular sieves to reduce water content.

Acetophenone (C_8H_8O , M.W: 120.15) Merck, India, was dried over

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anhydrous potassium carbonate for three days, filtered and then distilled. The middle fraction of the distillates was collected for use [6].

Ethane-1, 2-diol ($C_2H_6O_2$, M.W: 62.07 A.R.) S.D. Fine was first dried over fused CaO overnight and then distilled twice under vacuum. The middle fraction was further dried using $4A^{\circ}$ molecular sieves (Linde) and stored and protected against moisture and CO_2 [7].

2-methoxyethanol ($C_3H_8O_2$, 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 [8]. 2-ethoxyethanol ($C_4H_{10}O_2$, M.W: 90.12) and 2-butoxyethanol ($C_6H_{14}O_2$, M.W: 118.17) were purified as described in the literature [5].

Chlorobenzene (C_6H_5Cl , M.W: 112.56), S. D. Fine Chemical Limited, Mumbai, India (A. R. grade, purity > 99.5%), was purified as described in the literature [5].

Water was first deionized and then distilled from an all glass distilling set using alkaline $KMnO_4$ solution [9]. The double distilled water was then finally distilled using an all glass distilling set. Precautions were taken to prevent contamination from CO_2 and other impurities. The triply distilled (TD) water had specific conductance less than $1 \times 10^{-6} \Omega^{-1}cm^{-1}$.

Densities and viscosities of the purified solvents were in good agreement with the literature values [10-29] 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 $3A^{\circ}$ molecular sieves for three days before use.

3.1.2. Solutes

Sodium molybdate ($Na_2MoO_4 \cdot 2H_2O$, M.W: 241.95) and sodium tungstate ($Na_2WO_4 \cdot 2H_2O$, M.W: 329.86), were of A. R. grade (NICE, India) and were purified by re-crystallizing twice from conductivity water and then dried in a

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vacuum desiccator over P_2O_5 for 24 hours before use.

Commercial sample of catechol (C_6H_6OH , M.W: 110.1), was purified by repeated crystallization from mixture of chloroform-methanol. The sample was dissolved in chloroform in hot condition, filtered and to the filtrate dried and distilled methanol was added drop wise. Fine plate like crystal separated and was recovered by rapid filtration and ready for use.

Phosphomolybdic acid ($H_3[P(W_3O_{10})_4].24H_2O$, M.W: 2257.62) of analytical grade was purchased from Thomas Baker and was used without further purification. However, the salt was dried in a vacuum desiccator over P_2O_5 for 24 hours before use.

Sodium metal salts viz., Sodium iodide (NaI, M.W: 58.44), Sodium thiocyanate (NaSCN, M.W: 81.07), Sodium acetate (CH_3COONa , M.W: 82.03), A.R. grade were procured from Merck, India and purified as described in the literature [10, 11].

Tetraalkylammonium iodides viz., tetrabutylammonium iodide (Bu_4NI , M.W: 369.38), tetrapentylammonium iodide (Pen_4NI , M.W: 425.47), tetrahexylammonium (Hex_4NI , M.W. : 481.58) and tetraheptylammonium iodide (Hep_4NI , M.W. : 537.69) of Pruis grade were purchased from Aldrich, Germany and purified by dissolving in mixed alcohol medium and recrystallized from solvent ether medium [5]. After filtration, the salts were dried in oven for few hours.

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. Utmost care was taken in minimizing evaporation losses and preventing moisture pick-up. All solutions were prepared afresh before use.

3.1.3. Mixed solvents

The research work has been carried out with binary or ternary solvent systems with methanol isoamyl alcohol, acetophenone, amyl alcohol, dichloromethane, carbontetrachloride, tetrahydrofuran, 1, 4 dioxane 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:

Isoamyl alcohol (1) + 2-Methoxyethanol (2)

Isoamyl alcohol (1) + 2-Ethoxyethanol (2)

Isoamyl alcohol (1) + 2-Butoxyethanol (2)

Acetophenone (1) + Dichloromethane (2)

Acetophenone (1) + Amyl alcohol (2)

Amyl alcohol (1) + Dichloromethane (2)

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Ternary mixtures studied:

Tetrahydrofuran (1) + Methanol (2) + Benzene (3)

Tetrahydrofuran (1) + Methanol (2) + Toluene (3)

Tetrahydrofuran (1) + Methanol (2) + Chlorobenzene (3)

Tetrahydrofuran (1) + Methanol (2) + Nitrobenzene (3)

Tetrahydrofuran (1) + Methanol (2) + Anisole (3)

Tetrahydrofuran (1) + Methanol (2) + Cyclohexane (3)

Tetrahydrofuran (1) + Methanol (2) + Cyclohexanone (3)

Acetophenone (1) + Amyl alcohol (2) + Dichloromethane (3)

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

1,4-Dioxane (1) + Tetrahydrofuran (2) + Tetrapentylammonium iodide (3)

1,4-Dioxane (1) + Tetrahydrofuran (2) + Tetrahexylammonium iodide (3)

1,4-Dioxane (1) + Tetrahydrofuran (2) + Tetraheptylammonium iodide (3)

Water (1) + Ethane 1,2-diol (2) + Sodium molybdate (3)

Water (1) + Ethane 1,2-diol (2) + Sodium tungstate (3)

Water (1) + Catechol (2) + phosphomolybdic acid (3)

Methanol (1) + Ethane 1,2-diol (2) + Sodium acetate (3)

Methanol (1) + Ethane 1,2-diol (2) + Sodium iodide (3)

Methanol (1) + Ethane 1,2-diol (2) + Sodium thiocyanate (3)

Carbontetrachloride (1) + Nitrobenzene (2) + Tetraethylammonium iodide (3)

Carbontetrachloride (1) + Nitrobenzene (2) + Tetrapentylammonium iodide (3)

Carbontetrachloride (1) + Nitrobenzene (2) + Tetrahexylammonium iodide (3)

Carbontetrachloride (1) + Nitrobenzene (2) + Tetraheptylammonium iodide (3)

3.2. Experimental Methods

3.2.1 Density Measurements

Densities (ρ) were measured with an Ostwald-Sprengel-type pycnometer having a bulb volume of 25cm^3 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 [30, 31]. The pycnometer filled with air bubble free experimental liquid was kept vertically in a thermostatic water bath maintained at ± 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 ± 0.01 mg were made on a digital electronic analytical balance (Mettler, AG 285, Switzerland). The precision of the density measurement was $\pm 3 \times 10^{-4} \text{gm cm}^{-3}$. Figure 1 shows an Ostwald-Sprengel-type pycnometer (Single arm) used in our works.

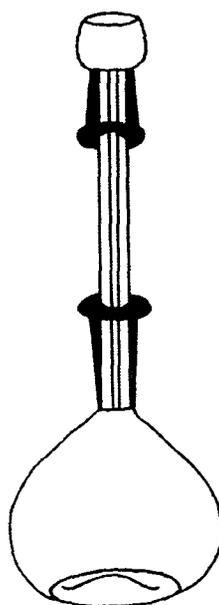


Figure 1: An Ostwald- Sprengel-type pycnometer.

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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 [32]. 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 [33-36].

3.2.2 Viscosity Measurements

The kinematic viscosities were measured by means of a suspended level Ubbelohde [37] 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 ± 0.1 s. The viscometer was always kept in a vertical position in the thermostatic bath with an accuracy of ± 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 (ν) and the absolute viscosity (η) are given by the following equations:

$$\nu = \left(Kt - \frac{L}{t} \right) \quad (1)$$

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

where t is the average time of flow, ρ 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 2.4065×10^{-3} and -5.9535152 , respectively. The

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kinetic energy corrections were done from these values and they were found to be negligible.

Relative viscosities (η_r) were obtained using the equation:

$$\eta_r = \frac{\eta}{\eta_0} = \frac{\rho t}{\rho_0 t_0} \quad (3)$$

where, η , η_0 , ρ , ρ_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.

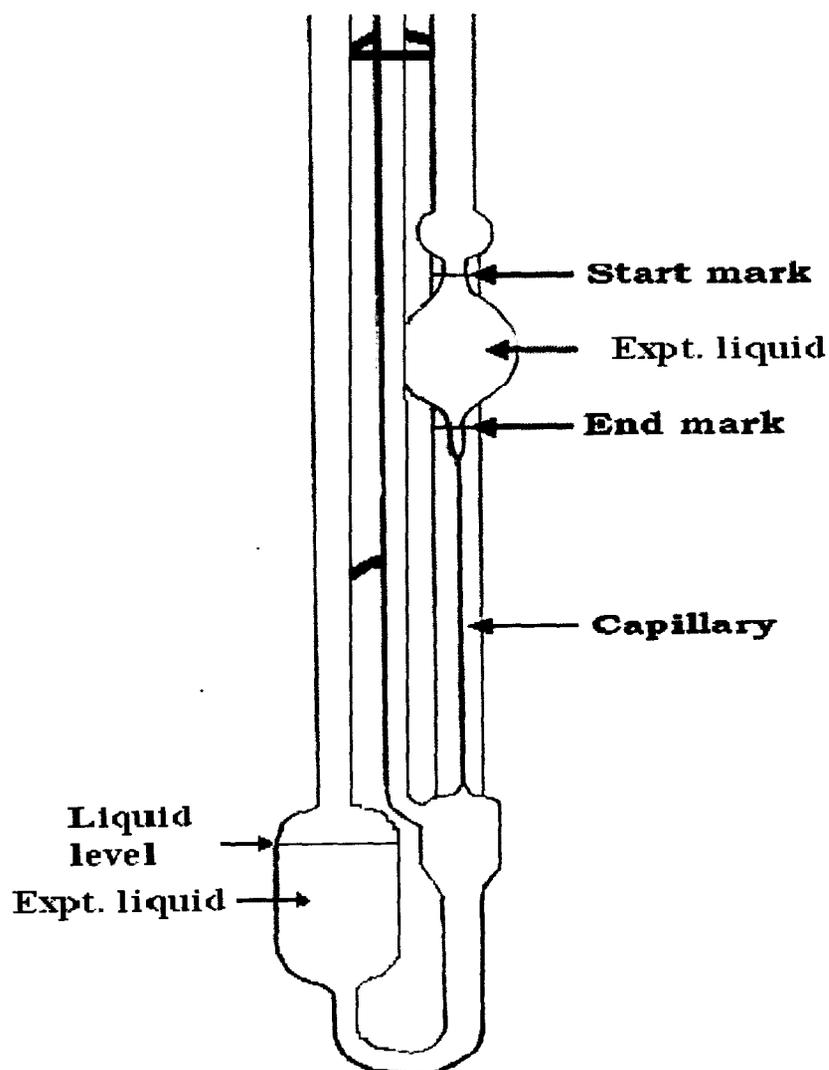


Figure 2: A suspended-level Ubbelohde viscometer.

3.2.3 Conductance Measurements

Systronic Conductivity meter- 308 was 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 Lindand co-workers [38]. 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. In order to minimize moisture contamination, all solutions were prepared in a dehumidified room with utmost care.

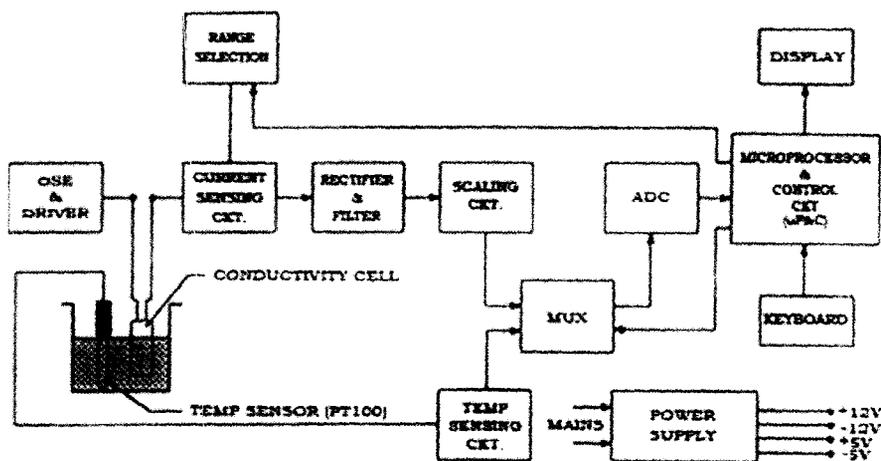


Figure 3: Block diagram of the Systronic Conductivity meter- 308.

3.2.4 Ultrasonic Sound Speeds Measurements

The speeds of sound (u) in pure liquids and solutions were measured with multi-frequency ultrasonic interferometer (supplied by Mittal Enterprise, New Delhi) with an accuracy of $\pm 0.3\%$. In the present work, a steel cell fitted with a quartz crystal of 2 MHz⁽³⁵⁾ - frequency was employed. This was calibrated with water, methanol, and benzene at the desired temperatures. The temperature stability was maintained within $\pm 0.01^\circ\text{C}$ by circulating thermostated water around the cell by a circulating pump.

The principle used in the measurement of the sound velocity (u) is based on the accurate determination of the wavelength (λ) in the medium. Ultrasonic waves of known frequency (f) are produced by a quartz crystal fixed at the bottom of the cell. A movable metallic plate kept parallel to the quartz crystal reflects these waves. 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 gives rise to an electrical reaction on the generator driving the quartz crystal and the anode current of the generator becomes maximum. If the distance is now increased or decreased and the variation is exactly one half wavelengths ($\lambda/2$) or integral multiple of it, anode current again becomes maximum. From the knowledge of the wavelength (λ), the velocity (u) can be obtained by the relation:

$$\text{Velocity } (u) = \text{Wavelength } (\lambda) \times \text{Frequency } (f) \quad (4)$$

Isentropic compressibility (κ_s) can then be calculated by the following relation:

$$\kappa_s = \frac{1}{u^2 \rho} \quad (5)$$

where ρ is the density of the experimental liquid.

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

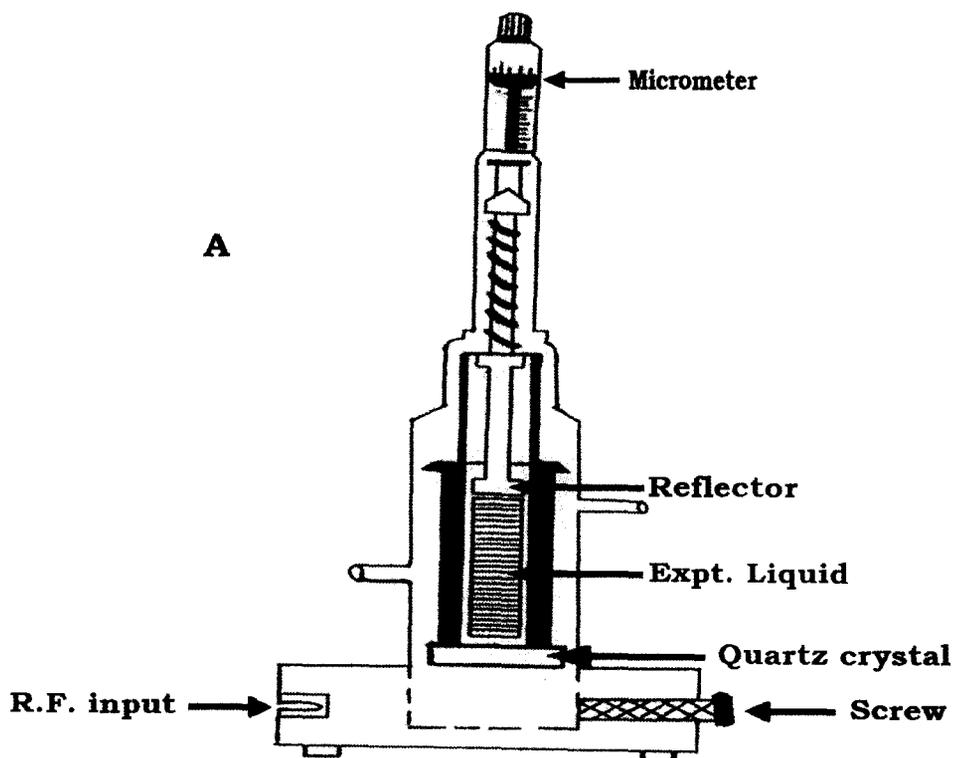
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shielded cable. The cell is filled with an experimental liquid before switching on the generator. The ultrasonic waves move normal from the quartz crystal till they are reflected back from the movable plate and the standing waves are formed in the liquid in between the reflector plate and the quartz crystal.

The micrometer is slowly moved till the anode current on the high frequency generator shows a maximum. A number of maximum readings of anode current are passed on and their number (n) is counted. The total distance (d) thus moved by the micrometer gives the value of wavelength (λ) with the following relation:

$$d = n \times \frac{\lambda}{2} \quad (6)$$

In Figure 3(A) Cross-section of the measuring cell of a Multifrequency Ultrasonic Interferometer and (B) Position of reflector vs. crystal current are depicted. 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 $\lambda / 2$.



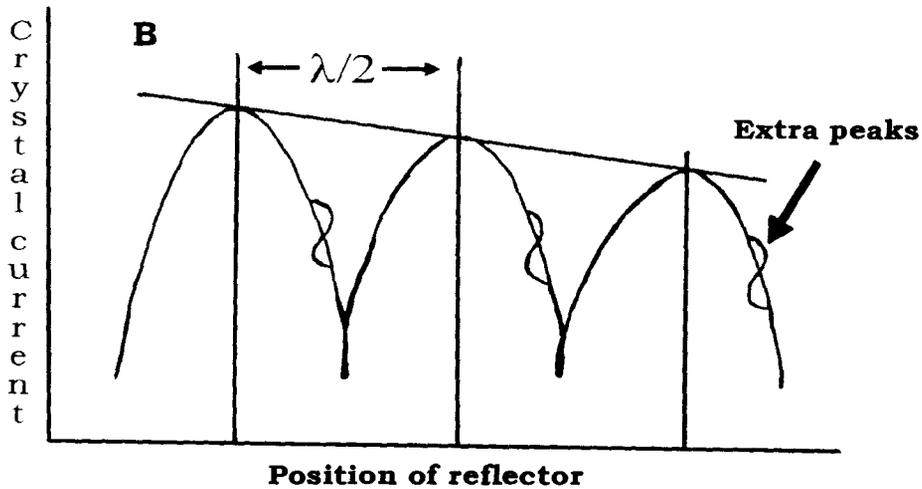


Figure 3: (A) Cross-section of the measuring cell of a Multifrequency Ultrasonic Interferometer and (B) Position of reflector vs. crystal current.

3.2.5 Refractive Index Measurements

Refractive index was measured with the help of Abbe-Refractometer (U.S.A). The accuracy of refractive index measurement was ± 0.0002 units. The refractometer was calibrated using twice distilled and deionized water, and calibration was checked after every few measurements. The temperature stability was maintained within $\pm 0.01\text{K}$ 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 refractive index is based on the difference of velocity of light in two different media.

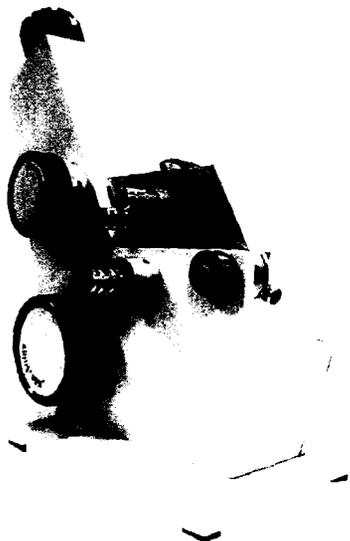


Figure 4: Abbe refractometer

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The speed of light in a vacuum is always the same, but when light moves through any other medium it travels more slowly since it is constantly being absorbed and reemitted by the atoms in the material. The ratio of the speed of light in a vacuum to the speed of light in another substance is defined as the index of refraction (n_D) for the substance.

$$\text{Refractive index } (n_D) \text{ of substance} = \frac{\text{Speed of light in vacuum}}{\text{Speed of light in substance}}$$

Whenever light changes speed as it crosses a boundary from one medium into another, its direction of travel also changes, i.e., it is refracted. The relationship between light's speed in the two mediums (V_A and V_B), the angles of incidence ($\sin \theta_A$) and refraction ($\sin \theta_B$) and the refractive indexes of the two mediums (n_A and n_B) is shown below:

$$\frac{V_A}{V_B} = \frac{\sin \theta_A}{\sin \theta_B} = \frac{n_B}{n_A} \quad (7)$$

Thus, it is not necessary to measure the speed of light in a sample in order to determine its index of refraction. Instead, by measuring the angle of refraction, and knowing the index of refraction of the layer that is in contact with the sample, it is possible to determine the refractive index of the sample quite accurately. Nearly all refractometers utilize this principle, but may differ in their optical design. In the Abbe' refractometer the liquid sample is sandwiched into a thin layer between an illuminating prism and a refracting prism (Figure 2).

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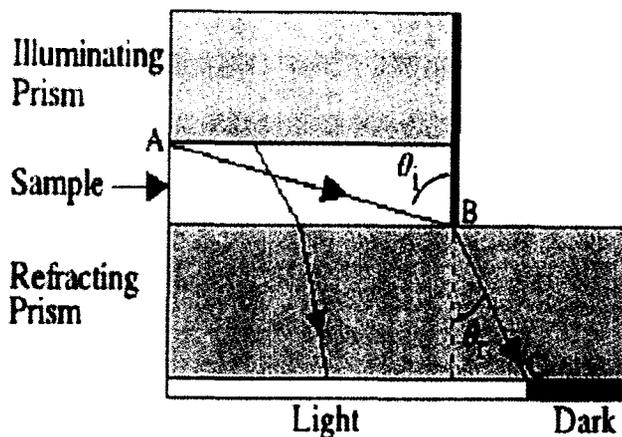


Figure 5: Cross-section of part of the optical path of an Abbe refractometer. The sample thickness has been exaggerated for clarity

The refracting prism is made of a glass with a high refractive index (e.g., 1.75) and the refractometer is designed to be used with samples having a refractive index smaller than that of the refracting prism. A light source is projected through the illuminating prism, the bottom surface of which is ground (i.e., roughened like a ground-glass joint), so each point on this surface can be thought of as generating light rays traveling in all directions. Inspection of Figure 2 shows that light traveling from point A to point B will have the largest angle of incidence (θ_i) and hence the largest possible angle of refraction (θ_r) for that sample. All other rays of light entering the refracting prism will have smaller θ_r and hence lie to the left of point C. Thus, a detector placed on the back side of the refracting prism would show a light region to the left and a dark region to the right.

Samples with different refractive indexes will produce different angles of refraction (equation 7) and this will be reflected in a change in the position of the borderline between the light and dark regions. By appropriately calibrating the scale, the position of the borderline can be used to determine the refractive index of any sample. In an actual Abbe' refractometer there is not a detector on the back of the refracting prism, and there are additional optics, but this is the essential principle.

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As mentioned earlier, the speed of light in a substance is slower than in a vacuum since the light is being absorbed and reemitted by the atoms in the sample. Since the density of a liquid usually decreases with temperature, it is not surprising that the speed of light in a liquid will normally increase as the temperature increases. Thus, the index of refraction normally decreases as the temperature increases for a liquid. For many organic liquids the index of refraction decreases by approximately 0.0005 for every 1 °C increase in temperature. However for water the variation is only about -0.0001/°C. Most of the refractive index measurements reported in the text are determined at 298.15K and 303.15K.

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