Chapter 3

MATERIALS AND EXPERIMENTAL METHODS

MATERIALS AND EXPERIMENTAL METHODS

Purification of chemicals employed is an important aspect in any experimental work on the thermodynamic properties. The errors that arise in experimental measurements apart from other sources are due to the impurities such as water which is absorbed by chemicals, dissociation of chemicals on standing or exposure, association etc. In addition to initial purification, a continuous check of the purity of chemicals is an important aspect for any experiment.

3.1: Purification of Chemicals and Preparation of Mixtures:

All the chemicals were obtained in their highest purity state. However, some simple procedures have also been performed for their purifications prior to making the experimental measurements. All the liquids were used after double distillation and partially degassed with a vacuum pump under an inert atmosphere etc. A continuous check on the purity of the chemicals is an important step in any experiment on thermodynamics apart from initial purification. Further, all the samples were kept in tightly sealed dark bottles to minimize the absorption of atmospheric moisture and CO_2. Prior to actual measurements, all liquids were stored in contact with molecular sieves of size 0.4 nm to reduce the water content and were partially degassed under vacuum.

3.1.1: Amines

The amines used in the present work (n-butyl amine, sec-butyl amine, tert-butylamine, n-hexylamine, n-octylamine and cyclohexylamine) were of E. Merck make and were dried over potassium hydroxide pellets for several days. They were then distilled using a vacuum jacketed column packed with Pyrex glass rings. In each case the middle fraction of the distillate was retained and distilled two or three times to give a constant density.

3.1.2: Carbitols

Carbitols: 2- (2-methoxyethoxy) ethanol, 2- (2-ethoxy ethoxy) ethanol and 2-(2-butoxy ethoxy) ethanol) were of E. Merck make and were stored in dark bottles to prevent contamination from air and dried over $4A^o$ molecular sieves to reduce the water content and is then fractionally distilled under reduced pressure and used.

46

Chapter 3

Prior to the measurements, all liquids were partially degassed under vacuum. The purities of the liquids were checked by measuring the densities and ultrasonic velocities at 308.15 K and atmospheric pressure and compared with the available literature data and are given in Table: 3.1.

3.1.3: Water

Deionised water (permutite process) was distilled thrice. To each litre of the distillate was added 0.5 g of sodium hydroxide and 0.2 g of potassium permanganate. This alkaline solution was slowly distilled from an all Pyrex glass vessel and the middle fraction was collected. The purity of the sample was checked by measuring the conductance.

3.1.4: Mercury

Mercury was first run in a fine stream through a long length of 5 per cent nitric acid and the process was repeated a second time and the sample was finally passed through a column of water. It was then dried by means of filter paper and run through a hardened filter paper with a pinhole at the apex of the cone. The dried sample was finally distilled twice under reduced pressure using Gallen Kemp mercury distillation apparatus.

The chemicals used in the present study have been recorded in table 3.1 by comparing the experimental & literature values of density (p), viscoscity (η) and ultrasonic velocity (u) of the liquids.

Chapter 3

Table: 3.1: Comparison of experimental density (p) viscosity (η) and Ultrasonic velocity (u) of the pure liquids with literature values at 308.15 K.

Liquid	$\rho \times 10^{-3}$ Kg m^{-3}		$\eta \times 10^3$ Kg m^{-1} s^{-1}		u x m s^{-1}	
	Exptl.	**Lit.**	**Exptl.**	**Lit.**	**Exptl.**	**Lit.**
2-(2-Methoxyethoxy) ethanol	1.0073	1.0073 [1]	0.2546	0.2547 [2]	1413.5	1413 [2]
2-(2-Ethoxyethoxy) ethanol	0.9751	0.9748 [1]	4.510	4.500 [2]	1383.5	1384 [2]
2-(2-Butoxyethoxy) ethanol	0.9399	0.9397 [1]	3.4811	3.4323 [2]	1355.5	1355 [2]
n-Butyl amine	0.7241	0.7240 [1]	0.4248	0.4249 [2]	1230.0	1232.0 [2]
Sec.butyl amine	0.7084	0.7090 [1]	0.3994	0.3996 [2]	1151.0	1154.0 [2]
Tertiary butyl amine	0.6809	0.6837 [1]	0.4122	0.4126 [2]	1050.0	1050.0 [2]
n-Hexyl amine	0.7522	0.7507 [3]	0.6001	0.6000 [2]	1286.5	1288.0 [2]
n-Octyl amine	0.7704	0.7702 [3]	0.9263	0.9267 [2]	1324.0	1325.0 [2]
Cyclo hexyl amine	0.8525	0.8527 [3]	1.3245	1.3249 [2]	1385.5	1384.0 [2]

3.1.5: Preparation of mixtures

Mixtures with composition spanning the entire mole fraction ranges were prepared. All the binary liquid mixtures were prepared by weighing appropriate amounts of pure liquids on an electronic balance (Mettler balance – India) with a precision of ± 0.1 mg by syringing each component into airtight stopper bottles to minimize evaporation losses. The estimated uncertainty on the mole fraction composition is found to be less than ± 1x 10^{-4} .The mixtures are shaken in order to ensure complete homogeneity of the two liquids.

3.2: DENSITY MEASUREMENTS

Several methods are in use to measure the densities of pure liquids or solutions. These methods are briefly mentioned below.

Chapter 3

3.2.1: Magnetic Float Method

This is another form of buoyancy method. It has been used for very accurate measurements of densities and density changes. It makes use of submerged float containing an iron core, the buoyancy of which is delicately counter balanced by the pull of a current carrying solenoid. The method is particularly suitable for determination of excess volumes of mixtures. Benjamin[1] designed a magnetic float method, which gives an accuracy of ± 0.001 % in density measurements. Millero[2] surveyed the earlier literature on magnetic float densitometers and described a highly versatile new apparatus with a precision of ± 0.0002 %, a capacity of about 32 cm^3 and a useful temperature range greater than from 293.15 to 325.15 K. Drost-Hensen et al.[3] gave details on magnetic float densimeter and Masterton and Seiler[4] fully described their apparatus which has a reproducibility of 3 ppm. Further developments are proposed by Kupke and Beams[5], Keramati and Wolgemuth[6], Kupke and Crouch[7] and Haynes and co-workers[8-10].

3.2.2: Balancing Column Method

It is based on the principle that the hydrostatic pressure of a given weight of liquid is proportional to its density. This method is quite useful when small quantities of liquid are available. It is capable of yielding an accuracy of 2 in 10^4.

3.2.3: Buoyancy Method

It is based on Archimedes' principle. This method is rapid and useful for temperature variation studies. However, it is less widely used due to the difficulty of maintenance of temperature and due to surface tension effects. Under optimum conditions the method is capable of yielding an accuracy of 2 in 10^6.

3.2.4: Dilatometer Method

The density (ρ) of the mixtures is also evaluated using the molar volumes V_1 and V_2; molecular weights M_1 and M_2 of the component liquids and the excess molar volume V^E of the mixture using the relation

$$V = \frac{M_1 X_1 + M_2 X_2}{(V_1 X_1 + V_2 X_2) + V^E} \qquad \ldots \quad 3.1$$

where 'X$_1$' and 'X$_2$' are mole fractions of the component liquids. The measurement of V^E thus enables one to determine the density of the mixture. Neubeck[11] described a basic weight dilatometer where in a mixture or a pure liquid is maintained by mercury. Volume changes which occur on change of temperature are determined by the weight of mercury expelled or added to the dilatometer. Improved versions in this method are reported by Burlew[12], and Wirth and Losourdo[13]. Washington and Battino[14] described a dilatometer convenient to use in the temperature range 263.15 – 353.15 K and utilized for density measurements accurate to ± 0.003 cm^3 mol^{-1} in V^E. Continuous dilatometers which are capable of measuring the excess volumes with an accuracy of ± 0.0005 cm^3 mol^{-1} are developed[15, 16] and reported in recent times.

3.2.5: Digital Densimeter

Kratky and co-workers[17-19] designed a digital densimeter in which the density of a fluid is determined by measuring the resonant frequency of a mechanical oscillator in a 'U' shaped glass tube, filled with the fluid. The method assumes a linear relationship

$$\rho = AT^2 + B \qquad\qquad ... \qquad 3.2$$

between density 'ρ' of the fluid and the square of the time period 'T' of the oscillator. 'A' and 'B' are constants of the instrument and their values are determined by calibration with two fluids of known densities. Water and air are generally used for this purpose, Anton Parr K.G. manufactured a digital densimeter (Model DMA 020) based on this principle.

Kiyohara and Benson[20] employed this densimeter and found its usefulness for determination of densities accurately. Under favourable circumstances density can be determined, within ± 1.5 x 10^{-6} g cm^{-3}. However, this accuracy is limited to differences which are less than about 0.05 g cm^{-3}. It is especially useful for the study of dilute aqueous solutions and to the determination of partial molar volumes. Recently, it has been used to determine densities of mixtures of organic liquids. Other models are also used by Letcher[21] (Model PAAR DMA 601 which gives an accuracy of ± 0.007 cm^3 mol^{-1}), Picker[22] and Roux et al.[23]

3.2.6: Pycnometer Method (Present Method)

In this method the mass of a known volume of the liquid is weighed. Many types of pycnometers are described by Bauer and Lewin. Pycnometers are employed

to determine the densities of the mixtures and the excess volumes are derived from these densities provided the composition of the mixture is known. Battino[24] used an improved version of the single arm pycnometer described by Wood and Brusie[25] which can be used to determine the density accurate to fifth decimal place. Parker and Parker[26], Kohler and Rott[27] and Ruiter[28] reported other good designs of mixing bottles for the measurement of densities.

In the present investigations pycnometer of Parker and Parker type with minor modifications has been employed. The double stem pycnometer with a bulb of 20 cm^3 capacity on one of the stems used in the studies is shown in Fig. 3.1. The stems A and B are made of a Pyrex glass tube of a bore of about 3 mm and are bent to the sides making obtuse angle at the bend. The vertical part of the stem (A) containing the bulb is about 5 cm in length and carries a mark in the middle to read the liquid level. A mark is also made on the second stem (B) at the same level to read the difference between the liquid levels in the two stems. The inclined part of each of the stems is 4 cm. The enlargement of the stems serve as 'over flow cup' when liquids are raised to temperatures above the room temperature. The open ends of the stems are closed by suitable teflon caps in order to prevent the loss of liquids due to evaporation.

The well cleaned and dried pycnometer is weighed accurately with reference to another pycnometer of the same design, weight and surface area hung to the hook of the pan containing the weights. The liquid is introduced into the weighed pycnometer with the help of a syringe without air bubbles, approximately up to the marks on the stems. The pycnometer is kept immersed vertically in a thermostatic bath maintained at the desired temperature with the ends of the stems closed with the teflon caps for about half-an-hour. This permits the unfilled part of the stem to be drained completely and allows liquid-vapour equilibrium to be established. The amount of the liquid at initial filling is adjusted to allow for expansion (or contraction) due to the difference in temperature between the bath and the surroundings. The distance between the mark and the liquid meniscus in both the stems is determined with the aid of a travelling microscope, which reads, with a precision of 0.001 cm. The pycnometer is then removed from the bath, wiped with a cleaned damp cloth and is hung from the hook. After about 15 minutes, it is weighed with an analytical

balance which gives an accuracy of 0.1 mg. The reference pycnometer hung from the hook of the other pan is also subjected to the same treatment to eliminate the errors in weight due to moisture condensation on the surface of the pycnometer.

The volume of the pycnometer is calculated from the weight of the water filling the pycnometer up to the mark exactly. The weight of water at 308.15 K filling the volume of the pycnometer included between the marks is determined as described below. The capillary used to prepare the stems is filled with mercury and the length of the mercury thread is measured with the help of the travelling microscope. From this length and the weight of the mercury filling, the internal radius (r) of the capillary is calculated. The uniformity of the bore of the tube is also checked by a similar procedure. The volume (dV) of water enclosed between the mark and the liquid meniscus is calculated using the formula $\pi r^2 h$ (where 'r' is the radius of the capillary, 'h' is the distance between the mark and the meniscus). This volume (dV) gives the difference between the volume of water that fills exactly up to the mark at 308.15 K and the actual volume of the liquid taken. The weight (dW) of water of this volume (dV) is calculated by multiplying 'dV' with the density of water at 308.15 K (0.99406 g/cm^3). The mass (w) of water filling the pycnometer up to the mark is obtained by substracting or adding dW, as the case may be from or to the weight of water actually filling the pycnometer at 308.15 K. The experiment is repeated till reproducible results with accuracy of 1 in 10^4 parts are obtained. By dividing this weight 'W' by the density of water at 308.15 K, the exact volume of the pycnometer between the marks can be calculated.

To determine the density of any liquid, the pycnometer is filled with the liquid such that the liquid meniscus is as close as possible to the mark on the pycnometer. The pycnometer with liquid is kept immersed in the thermostat at 308.15 K for sufficient time such that the level of the liquid is constant in both the limbs. The height of the liquid level above or below the reference mark as the case may be is measured with a travelling microscope. The weight 'W' of the liquid in the pycnometer is determined by the procedure similar to that described earlier. This weight 'W' is divided by the volume of the liquid taken in the pycnometer to obtain the density of the liquid.

Chapter 3

A water thermostat is used in the studies whose temperature is controlled to better than ± 0.05 K. The temperature of the thermostatic liquid is measured using a thermister thermometer. The accuracy in density measurement is ± 1 in 10^4 parts.

3.3: VISCOSITY MEASUREMENTS

Viscosities of liquid and liquid mixtures can be determined by two methods: (1) absolute method and (ii) relative method. The determination of viscosity by absolute method requires the determination of the constants of the apparatus. Hence, viscosities of liquids and binary solutions were determined by the relative method. In this method, it is easy to measure the viscosity of a liquid by comparison with a reference standard liquid of known viscosity. Different types of viscometers and their modifications and conditions of viscosity measurements are given below.

1.   .Ostwald's Viscometer..

2.   Stokes falling Sphere Viscometer..

3.   Ubbelohde suspended level Viscometer.

3.3.1: Ostwald Viscometer Method

Viscosity measurements can also be made with a modified Ostwald type viscometer with a flow time of 300s for water at 298.15 K. The viscometer contains two bulbs A & B. A definite quantity of liquid is introduced into the bulb (B) of the viscometer and is kept immersed in the thermostat. After equilibration, the liquid is drawn up by suction into the bulb (A) until the liquid level is raised above the mark 'a'. The liquid is then allowed to drain and the flow time (t_1) taken by the liquid to drain from 'a' to 'b' is measured with a stop watch which reads 1/10 sec. The viscometer is cleaned, the reference liquid is added and the procedure is repeated to determine the flow time (t_2). The viscosity of the test liquid is calculated by using the equation (3.3)

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2} \qquad \qquad ... \quad 3.3$$

Where

η_1 =   viscosity of the test liquid

ρ_1 =   density of the test liquid

t_1 = flow time of the test liquid

and

η_2 = viscosity of the reference liquid (water)

ρ_2 = density of the reference liquid

t_2 = flow time of the reference liquid.

At least eight determinations are made for each liquid and difference of any two readings should not exceed 0.2 sec. The reported viscosity data are reproducible within $\pm$ 0.005 Cp.

3.3.2: Stokes falling sphere Viscometer Method

The viscometer consists of a vertical cylindrical tube filled with the liquid under test and immersed in a thermostat at the desired temperature. A steel ball, of density 'ρ' and the diameter suitable to give a slow rate of fall, is now dropped through the neck of the tube, and the time of fall between two marks is determined with a stop watch. If the process is repeated with a liquid of known density and viscosity then Eq. 3.4

$$\eta = \frac{2r^2 (\rho - \rho_m)g}{9\,V} \qquad \text{... 3.4}$$

r = radius of spherical body

ρ = density of spherical body

ρ_m = density of fluid

g = acceleration due to gravity

V = volume of the liquid taken in the tube.

Yields for the ratio of the viscosities of the two liquids

$$\frac{\eta_1}{\eta_2} = \frac{(\rho - \rho_{m1})\,t_1}{(\rho - \rho_{m2})\,t_2} \qquad \text{... 3.5}$$

Therefore, knowing the viscosities of one of the liquid, the density of the ball, and the densities of the two liquids, the viscosity of the liquid under study can be calculated by means of Eq. 3.4 from the observed values of t_1 and t_2.

54

Chapter 3

3.3.3: Ubbelohde suspended level Viscometer Method (Present Method)

Viscosity measurements were made with the help of Ubbelohde[29] suspended level viscometer using 10-15 cm^3 of solution. The design of this viscometer eliminates pressure corrections and minimises surface tension effects[30]. Choice of viscometer dimensions was limited by consideration of two factors.

(i)   Volume of solution required: Unlike Kaminsky's work on aqueous systems[31] preparation of purified solvent limited the volume to 10-15 cm^3.

(ii)   Capillary dimensions: The capillaries are too narrow which are found to be very sensitive to small quantities of impurities or dust, while broad capillaries require a large kinetic energy correction, together with introducing errors due to heating and drainage effects.

For absolute viscosities the viscometer is calibrated with water at temperature over the range 288.15 – 318.15 K and with 30% aqueous sucrose solution, using an equation of the form

$$\eta = \rho\,(at - b/t) \qquad\qquad ... \qquad 3.6$$

which may be derived for a capillary viscometer and where 'η' is the dynamic viscosity, 'ρ' is the density of the liquid,'t' is the reflux time and 'a' and 'b' are the viscometer constants. Majority of the work is concerned solely with relative viscosities, η/η_o and it is found sufficiently precise to take

$$\eta/\eta_o \;=\; \frac{\rho t}{\rho_o t_o} \qquad\qquad ... \qquad 3.7$$

where 'η', 't' and 'ρ' refer to the solution and 'η_o', 't_o' and 'ρ_o' to the solvent. 't_o' and 'ρ_o' are redetermined for every batch of solvent used.

The viscometers should be mounted in a thermostatic water bath of 45 dm^3 capacity by means of rigid metal clamps which can be individually leveled. Efflux times are measured with an accuracy of ± 0.001s by means of photocell/lamp assemblies firmly attached to the viscometers and coupled to an electronic timing and programming system. Eicher and Zwolonski[32] reported a heating effect due to lamps in a similar detector unit. When times of a continuous series of measurements on

water were plotted over a time scale of several hours, no trend was apparent and the efflux times varied by less than 0.02% from the mean. The first efflux time is generally discarded as subsequent measurements are usually slightly shorter due to an initial wetting effect.

In the present study, viscosities of liquids and liquid mixtures were determined by relative method using Ubbelohde viscometer (Fig. 3.2) as this is superior over the Ostwald type of viscometer. In the Ubbelohde viscometer the liquid is allowed to flow only down the walls of the bulb below the capillary, thus forming a suspended level in the form of a hallow hemisphere at the lower end of the capillary to discharge into an air space. This eliminates the uncertainty regarding the position of the lower liquid level and balance the effect of surface tension at the upper meniscus.

The viscometer is fabricated with a heavy walled glass so that the instrument is resistant to shock. The bulb between etched lines (Ubbelohde) has a capacity about 4 ml and the capillary tube had a length of about 90 mm and 0.5 mm internal diameter. The reservoir of the viscometer was filled with the sample liquid by tilting the viscometer to about 30° from the vertical and its limbs closed with teflon caps. The viscometer was kept for about 30 minutes in a thermostat maintained at 308.15 K temperature. This permits the contents of the viscometer to attain the experimental temperature. Then the teflon caps were removed and the sample was raised to the middle of the upper most bulb by applying suction, with the air vent being closed by the finger tip. The efflux time was measured between the marks on either side of the bulb with a stopwatch, which had an accuracy of ± 0.1 sec. The efflux time of each sample was measured at least eight times. The average efflux time and density at the same temperature were employed in the calculation of viscosity, the viscosities (η) of pure liquids and liquid mixtures were computed from the efflux time and densities using the relation

$$\eta \quad = \quad K_V \, \rho t \qquad \qquad \qquad ... \qquad 3.8$$

where 'K$_V$' is viscometer constant, 'ρ' and 't' are density and efflux time for a given sample respectively. At the experimental temperature (308.15 K) 'K$_V$' was evaluated using viscosity and density of pure water from the relation

Chapter 3

$$K_V = \frac{\eta_w}{\rho_w \, t_w} \qquad\qquad ... \quad 3.9$$

The calibration of viscometer was checked by measuring the viscosities of pure liquids like benzene, chlorobenzene and carbon tetrachloride at 298.15 K. The measured values of viscosity are accurate to $\pm 0.1\%$.

3.4: ULTRASONIC VELOCITY MEASUREMENTS

An ultrasonic interferometer is a simple and direct device to determine the ultrasonic velocity in liquids with a high degree of accuracy.

3.4.1: Principle

The principle used in the measurement of velocity (u) is based on the accurate determination of the wavelength (λ) in the medium. Ultrasonic waves of known frequency (f) (2 MHz in the present study) are produced by a quartz plate fixed at the bottom of the cell. The waves are reflected by a movable metallic plate (reflector) kept parallel to the quartz plate. If the separation between these two plates is exactly equal to the multiples of the sound wavelength, standing waves are formed in the medium. The acoustic resonance gives rise to an electrical reaction on the generator driving the quartz plate, and the anode current of the generator becomes maximum.

If the distance is now increased or decreased with the help of the micrometer screw, and the variation is exactly one half wave length ($\lambda/2$) or multiple of it, anode current again becomes maximum. From the knowledge of wavelength (λ), the velocity (u) can be calculated by the relation

Velocity = Wavelength x Frequency

$u = \lambda f$ $\qquad\qquad ... \quad 3.10$

In the present investigation, a single crystal variable path interferometer was used to measure the ultrasonic velocities.

3.4.2: Description

The ultrasonic interferometer consists of the following parts.

a. The High Frequency Generator

b. The Measuring Cell

Chapter 3

a. The High Frequency Generator

It is designed to excite the quartz plate fixed at the bottom of the measuring cell at its resonant frequency to generate ultrasonic waves in the experimental liquid filled in the measuring cell. A micrometer to observe the changes in current and two controls for the purpose of sensitivity regulation and initial adjustments of the micrometer are provided on the panel of the High Frequency Generator.

b. The Measuring Cell

This is a specially designed double walled cell for maintaining the constant temperature of the liquid during the experiment. A fine micrometer screw has been provided at the top, which can lower or raise the reflector plate in the cell through a known distance. It has quartz plate fixed at its bottom.

A sketch of the interferometer cell is shown in Fig. 3.3. The interferometer cell is made of stainless steel, which is in the form of tube of inner diameter 1.6 cm with capacity 12 cm^3. A gold plated quartz plate of diameter 0.9 cm is cemented at the bottom of the interferometer cell. The ultrasonic waves start from the quartz plate and they are reflected back from the movable reflector. The reflector used in this interferometer is a fused quartz rod with a reflecting surface of 1.4 cm in diameter. This is coupled to a micrometer screw assembly reading up to 0.01 cm using a teflon coupler via the steel rod. The micrometer screw activates the steel rod, which is spring loaded to take care of any backlash. The micrometer assembly along with the reflector is fixed to the liquid cell with the help of a threaded cup and teflon ring. The required stability for the measuring cell is given by a heavy pedestal provision has been made for circulating water at any desired temperature from a thermostatically controlled bath into a jacket around the measuring space to maintain the experimental liquid at a constant temperature during the experiment.

A crystal controlled r.f. Oscillator (Fig. 3.4) is used to excite the transducer at a fixed frequency of 2 MHz. The HT current to the output tube is fed through a bridge network including a micrometer in such a way that the changes in the plate current can be easily observed on the micro-ammeter. Two controls are provided for the purpose of sensitivity regulation and initial current adjustment of the meter. The interferometer consists of an electrically dried quartz plate coupled to the liquid column. The length of the liquid column can be varied by moving the quartz

reflector, keeping its plane parallel to the quartz plate. Whenever the distance between the transducer and the reflector corresponds to an integral number of half-wavelengths, the liquid column vibrates in resonance resulting in an increase of the motional impedance of the transducer. This leads to a sharp dip in the r.f. Voltage across the transducer. The current vibrations are noted in the micro ammeter. The distance covered by the reflector between any two consecutive dips corresponds to one-half wavelength, $\lambda/2$.

3.4.3: Measurement of Velocity:

In the present work, a single crystal variable path interferometer (model; M-8, Mittal Enterprises, New Delhi) cell is used and shown in Fig.3.3. The interferometer cell of capacity 12 cm^3 is made up a stainless steel tube of inner diameter 1.6 cm. A gold plated quartz crystal of diameter 0.9 cm is cemented to the bottom of the cell.

The ultrasonic waves move from this crystal till they are reflected back by the movable reflector. A fused quartz rod is used as a reflector. The reflecting surface is 1.5 cm in diameter. This is coupled to a micrometer screw assembly reading up to 0.001 mm using teflon coupler via the steel rod. The micrometer actuates the steel rod, which is spring loaded to take care of any backlash. The micrometer assembly along with the reflector is fixed to the liquid cell with the help of a threaded cup and teflon ring. A heavy pedestal gives the measuring cell the required the stability. Water is circulated, from a thermostatically controlled bath into the jacket around the measuring space to maintain the experimental liquid at a constant temperature during the experiment.

To excite the transducer (crystal) at a fixed frequency of 2 MH$_Z$ a crystal controlled r.f. oscillator (Fig 3.4.) was used. The H.T current to the out-put tube is fed through a bridge network including a micro-ammeter in such a way that the change in plate current can easily be observed on the micro – ammeter. Two controls to regulate sensitivity and to adjust initial current of the meter are also provided. The interferometer thus consists of an electrically driven quartz transducer coupled to the liquid column. The length of the liquid column can be varied by moving the quartz reflector, keeping its plane parallel to the quartz crystal. Whenever the distance between quartz transducer and the reflector corresponds to an integral multiple of half wave length, the liquid column vibrates in resonance. This leads to an increase in the

59

Chapter 3

motional impedance of the transducer. The current variations are noted in the micro- ammeter. The distance covered by the reflector between any two consecutive dips corresponds to one half wave length, $\lambda/2$.

Chapter 3

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Chapter 3

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Chapter 3