CHAPTER II

EXPERIMENTAL TECHNIQUES
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EXPERIMENTAL PROCEDURE

This chapter describes the experimental techniques employed in the present study to determine the ultrasonic velocity, ultrasonic attenuation, density, and viscosity. All the chemicals used in this work are of AR/BDH quality. Measurement of ultrasonic velocity is generally made either by continuous wave methods, or by pulse methods. Continuous waves are used in optical diffraction, reverberation and interferometry methods. In pulse echo or pulse superposition methods, RF pulses of short duration are used. Measurements are made at the temperatures of 303K, 313K, and 323K.

2.1 CONTINUOUS WAVE METHOD

In the fixed frequency variable path ultrasonic interferometer, the acoustic wave length is measured which, in turn, is used to compute the ultrasonic velocity. The ultrasonic cell is a double walled brass cell with chromium plated surfaces. The capacity of the cell is 10 ml. The schematic diagram is shown in figure 2.1. In the figure 2.1, A is the crystal holder from which the leads are taken out. B is the metallic reflector attached to the shaft C which moves up or down by the action of a spring and a micrometer screw. The micrometer screw has a least count of 0.001 cm, and the length of the pitch scale is 25 mm. The cell has an outer shell through which water is circulated. The temperature of the circulating water is maintained at a fixed temperature by a thermostatically controlled water bath. The temperature is maintained constant within ± 0.1°C.
ULTRASONIC INTERFEROMETER CELL

FIG. 2.1

A - CRYSTAL
B - REFLECTOR
At the bottom of the cell, an X-cut quartz crystal of resonant frequency 2 MHz is fixed. The crystal is excited at 2 MHz by an RF oscillator. With liquid sample in the cell, the standing waves are formed between the reflector and the crystal. This alters the anode current, and the changes in the anode current is measured in a differential amplifier configuration using a micro ammeter. The complete experimental set up is shown in figure 2.2.

The principle used in the measurement of velocity (C) is based on the accurate determination of the wavelength (λ) in the medium. If the separation between the quartz plate and reflector is exactly a whole multiple of the sound wavelength (λ), standing waves are formed in the medium. This 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, and the variation is exactly one half wavelength (λ/2) or multiple of it, anode current again becomes maximum. From the knowledge of wavelength (λ), the velocity (C) can be obtained from the relation $C = \lambda f$, where $f$ is the frequency of the ultrasonic wave.

When the solution attains the required temperature, the micrometer is operated, and the distance $d$ (mm) is measured and $d = n\lambda/2$. Therefore, by knowing ($\lambda$) from the above, C the ultrasonic velocity can be calculated. In practice, $d$ for 20 maxima is measured so that $f=2$ MHz and $n=20$, $C=200$ m/s. Thus $C$ in metres/second can be readily obtained by multiplying the measured values of $d$ for 20 maxima by 200. The velocity is measured to an accuracy of $\pm 0.001\%$. 

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FIG. 2.2 EXPERIMENTAL SET-UP FOR CONTINUOUS WAVE ULTRASONIC INTERFEROMETER
2.2 PULSE ECHO TECHNIQUE

The ultrasonic velocity and absorption can be determined with the pulse echo technique. A cell is designed and fabricated in this laboratory for the measurement of absorption and velocity.

The cell fabricated in this laboratory is a double walled stainless steel cell with a central uniform bore of 18 mm diameter and a depth of 25 mm with a perfectly plane, highly polished bottom surface F (floor). The section along A-B with details of measurement are shown in the figure 2.3. A stainless steel shaft of 16 mm diameter and 40 mm length with a circular head on one side and a provision to mount the quartz crystal on the other side C (Fig. 2.4) moves up and down in the bore of the cell by the action of three adjustable 8 mm allen bolts and springs. A bore of 5 mm diameter at the centre of the shaft helps in taking the leads from the quartz crystal to pulsed RF of pulse echo interferometer.

The length of the liquid cell can be suitably altered by the movement of the shaft inside the bore. Through the fine adjustment of the allen bolts with a key, parallelism can be achieved between the floor of the cell 'F' which acts as a reflector and the quartz crystal 'C'. Through a side provision 'D', the liquid under investigation can be poured in, or out, to the cell without disturbing the parallelism between the crystal and the floor of the cell. Temperature of the sample can also be recorded by inserting a thermometer in this side provision 'D'. The cell has an outer jacket through which water can be circulated. EE represents the inlet and outlet of the water circulating jacket. The temperature of the circulating water is maintained at a fixed temperature by a thermostatically controlled
water-bath. The temperature is maintained constant within ± 0.1°C. An X-cut quartz crystal of 10 MHz fundamental frequency (supplied by Bharat Electronics Limited, Bangalore) is rigidly fixed by a suitable adhesive in the crystal mounting space 'C' of the shaft. An overall view of the cell is shown in figure 2.5.

A sharp RF electrical pulse with variable pulse duration from 2 μs to 20 μs is applied to the crystal with a suitable repetition frequency. The reflected wave returns to the transducer, and a part of its energy is converted into electrical signal. This signal is amplified and displayed on the oscilloscope. The trace of the oscilloscope is a series of echoes with decreasing amplitude. The decrease in the amplitude is a measure of absorption. The photograph of the experimental set up is shown in figure 2.6, and the oscilloscope trace is shown in figure 2.7.

The schematic block diagram of the pulse echo interferometer is shown in the figure 2.8. The transmitter signal starts from a gated video oscillator connected in Wien's bridge configuration. It generates RF pulse centred around 10 MHz. The repetition frequency of the RF generation is variable from 40 KHz to 400 KHz. The pulsed RF signal is amplified by means of an RF power amplifier, and then fed to the quartz transducer through the transmission gate. The transmission gate is used to isolate the input of the receiver amplifier circuit from the high voltage transmitter signal. The typical RF signal is 20V peak to peak across the transducer through 50 ohm coaxial cable.

The receiver amplifier has four stages, and the overall voltage gain is more than 60 db. The output of the amplifier is fed to the triggered oscilloscope (Philips, dual beam
FIG. 2.5
ULTRASONIC CELL FOR LIQUID MIXTURES
FIG. 2.6 EXPERIMENTAL SET-UP FOR PULSE ECHO ULTRASONIC INTERFEROMETER.
FIG. 2.7 OSCILLOSCOPE TRACE FOR PULSE ECHO PATTERN.
The echo heights are fitted to an exponentially decaying wave of the form \( Ae^{-\alpha x} \), and the value of \( \alpha \) is evaluated and expressed in nepers. The distance between crystal and reflector is measured, and the absorption per unit length is calculated and expressed in nepers/m. It is assumed that the reflector reflects all the sound energy incident upon it. The acoustic impedance of the solution and the reflector are approximately 1,42,500 gm cm\(^{-2}\) s\(^{-1}\), and 41,92,400 gm cm\(^{-2}\) s\(^{-1}\). Hence, the reflection coefficient estimated using the relation

\[
R = \left( \frac{\delta_1 - \delta_2}{\delta_1 + \delta_2} \right)^2
\]

(2.1) where \( \delta \) represents the impedance, and suffixes 1 and 2 represent the medium and the reflector. The calculated value is 0.87 which shows that only a negligible fraction is transmitted. A further check on the accuracy of the measured absorption was also made, using carbon tetrachloride as a standard liquid. The measured absorption \((\alpha/f^2)_{\text{obs}}\) for carbon tetrachloride at a frequency of 10 MHz is \(0.56 \times 10^{-14}\) Np/cm which compares well with the earlier value of \(0.54 \times 10^{-14}\) Np/cm. The accuracy in the measurement of velocity and absorption is 0.01% and 5% respectively.

### 2.3 Measurement of Density

To measure the density of the solution at different temperatures (303K, 313K, and 323K), a dilatometer is used. The dilatometer consists of a long graduated tube. The capacity of the bulb is 3 ml. The dilatometer was filled with the solution up to a fixed marking at the temperature of 303K. The mass of the solution was found using a single pan electronic
Fig. 2.8. Ultrasonic Pulse Echo Interferometer

- Video Amp with Gain Control
- Transmission Gate
- Power Amplifier
- Gated Video
- Transmitter Pulse Width Control
- Gate Control Circuit
- Digital Frequency Counter
- Specimen
- Pulseshaping
- Rep. Rate Oscillator
- Power Supply
- Ext. Mode
- Int. Mode to all Modules
- Measure
- Operate
balance (Sortorius) with an accuracy of ±0.001 gm. The dilatometer was kept in a constant temperature-bath. The volume expansions were measured using a travelling microscope which was previously calibrated to measure the volume to an accuracy of 0.001%. From the measurement of volumes at the temperatures of 303K, 313K, and 323K, the density of the solution at these temperatures were determined. The buoyancy correction has been applied in determining the density of the solutions. The density measurement at the room temperature of 303K for certain liquids were made using a specific gravity bottle of 5 ml. capacity.

2.4 MEASUREMENT OF SHEAR VISCOSITY

The shear viscosities of the solutions are determined using an Ostwald's viscometer kept in a constant temperature water-bath at 303K, using the value of viscosity of water at 303K from literature.

The formula used for the computation of viscosity is

\[ \eta_s = \left( \frac{\rho_1 t_1}{\rho_0 t_0} \right) \eta_0 \] (2.2)

where \( \rho_1 \) and \( \rho_0 \) are the densities of the solution and water, \( t_1 \) and \( t_0 \) are the corresponding flow times, and \( \eta_0 \) is the shear viscosity of water at the temperature of 303K.