CHAPTER 2

EXPERIMENTAL TECHNIQUES

2.1 Measurement of ultrasonic velocity in liquids

Ultrasonic velocity in liquids can be determined using continuous wave techniques [97-107] and pulse techniques [108-113]. Optical techniques and interferometric techniques come under continuous wave techniques. Interferometric techniques are widely used in the present time over optical techniques and yield very high degree of accuracy in the measurement of ultrasonic velocity. It may be mentioned here that the NRL measurements on the speed of sound in pure water are accurate to $\pm$ 0.02 m s^{-1}.

In the pulse techniques [109,110], a transducer radiates a short train of periodically repeated pulses into the liquid. These pulses will get reflected by a plane parallel reflector which can be moved to and fro with respect to the transducer. The reflected pulses are received by the same transducer radiating the pulses. Direct determination of transit time of a given pulse enables one to evaluate the velocity of propagation of sound wave in the medium. Techniques capable of giving an accuracy of 1 in 30,000 have been developed [111-113]. Sing around technique of the type developed by Garnsey *et al.* [114] is capable of determining the difference between sound velocities in dilute solutions and water with an accuracy of $\pm$ 0.005 m s^{-1}.

The interferometric technique has certain advantages over pulse technique for the determination of ultrasonic velocities in liquids. In the interferometric technique, path length and frequency can be measured accurately with ease, as compared to the transit time determination in pulse technique. Interferometric technique has been found to be more suitable for the determination of temperature and pressure

coefficients of ultrasonic velocity [115,116]. Del Grosso [117] has discussed in detail the aspects which demonstrate the superiority of ultrasonic interferometer in the measurement of ultrasonic velocity. In the present investigation, ultrasonic velocity was determined using a single crystal variable path interferometer working at 3 MHz. It consists of X-cut quartz transducer coupled to the liquid column whose length can be varied by moving the reflector parallel to the quartz transducer. The liquid column vibrates in resonance whenever the distance between the transducer and the reflector corresponds to $\frac{n\lambda}{2}$, where n is an integer and 'λ' is the wavelength. The vibration of the liquid column increases the motional impedance of the quartz transducer, resulting in a sharp dip in the voltage across it. The voltage across the transducer measured as a function of reflector distance exhibits a series of minima separated by $\lambda/2$. The wavelength can be determined by counting the number of dips over a distance of 4 cms movement of the reflector towards the transducer. The experiment is repeated 6 to 8 times and the average values of λ is used to evaluate the velocity using the relation $u = f\lambda$, where 'f' is the frequency of the ultrasonic wave generated by the quartz transducer. The distance through which the reflector is moved, is maintained the same in all measurements so as to keep the diffraction effect constant.

The mechanical assembly of the interferometer, enables the evaluation of λ with an accuracy of $\pm$ 0.0025%. The mechanical assembly of the interferometer is described in Section 2.2 of this chapter.

The pitch of the screw used to move the reflector is 0.1 cm and the head scale consists of 200 divisions. The least movement of the screw that can be measured is one fifths of a head scale division giving an accuracy of 1 part in 10000. Since a distance of 4 cm was usually covered in wavelength measurement, the error in the evaluation of λ due to limitations of the screw works out to be $\pm$ 0.0025 per cent. The accuracy in λ is less for highly absorbing liquids owing to the reduction in sharpness of the voltage minima. Since the present work deals with dilute aqueous solutions of electrolytes errors due to this source are negligible.

The quartz transducer has been excited at 3 MHz using tri-tet crystal controlled r.f. oscillator. The frequency stability of the oscillator is better than $\pm$ 1 Hz. The voltage variations across the transducer were observed using a difference amplifier. The details of the electric circuitry are given in Section 2.3.

The interferometer was immersed in a water thermostat whose temperature was maintained at any desired temperature with an accuracy of $\pm$ 0.01°C. The temperature of the experimental liquid was measured using a bead type thermistor which forms one arm of a constant current Wheatstone bridge followed by Chopper-stabilized operational amplifier as a null detector. The temperature gradients inside the cell were eliminated by stirring the contents of the cell periodically while making the measurements. The temperature of the experimental liquid was found to be constant to be better than $\pm$ 0.005°C. The sound velocity measurements are accurate to $\pm$ 0.003 per cent. The block diagram of the interferometer assembly is shown in Fig. 2.1.1.

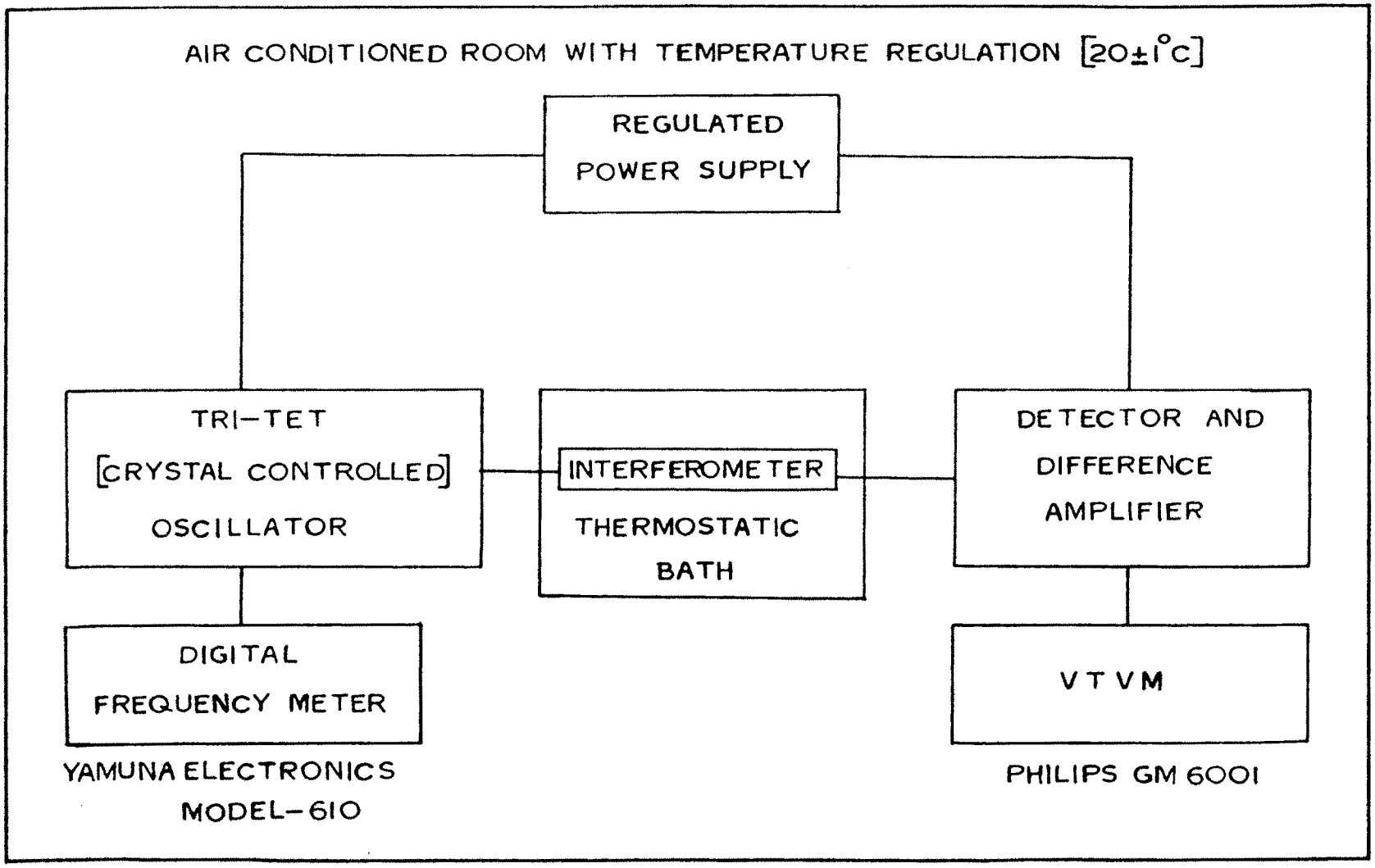


FIG. 2.1.1 . BLOCK DIAGRAM OF INTERFEROMETER ASSEMBLY

The thermostat and the electronic circuitry were housed in an air conditioned room, the temperature of which was maintained at 20 ± 1°C. This resulted in an improved stability of the oscillator and the efficiency of the temperature control.

The ultrasonic velocities evaluated have to be corrected for diffraction errors. The procedure developed by Subramanyam *et al.* [118] was followed to evaluate diffraction errors. The above said procedure is discussed in Section 2.4.

2.2. Mechanical assembly of the ultrasonic interferometer

In the present work a single crystal variable path interferometer was used to determine the ultrasonic velocities in liquids. The mechanical assembly of the interferometer is schematically shown in Fig. 2.2.1.

The experimental cell 'A' is made up of nickel coated brass tube of inner diamter 3.8 cms. The bottom portion of the tube is partially closed leaving an opening of 1.95 cms in diameter in which X-cut quartz crystal 'Q' is held firmly with a teflon O-ring. The crystal is kept pressed by a teflon ring 'D' held in position by three screws and tightened with optimum pressure by three nuts N. The inner surface of the crystal touching the bottom of the cell forms the grounded terminal. The phosphor bronze strip H fixed to the teflon ring presses against the outer face of the crystal and forms the second lead for electrical connections. The experimental cell is rigidly fixed to the nickel plated brass cup 'U' with a neoprene O-ring seal to prevent the outer surface of the crystal in contact with water when the interferometer is immersed in a thermostatic water bath.

The pitch of the screw used to move the reflector 'R' of diameter 2.5 cms is 1 mm and the head scale consists of 200 divisions. The bottom of the screw is coupled to the reflector rod through a steel ball B which eliminates the error due to nonaxiality between them besides making the reflector free to move axiality without rotating along with the screw. A pin 'P' screwed into a slot contained in the head C guides the reflector rod to move without any rotation. The reflector is spring loaded to eliminate back-lash in the screw. The head C is rigidly mounted on the cell A by three screws with a neoprene O-ring seal to prevent leakage of thermostat water into the cell. The stirrer 'I' helps to eliminate temperature gradients inside the liquid. 'T' is the thermistor used to measure the temperature of the experimental liquids.

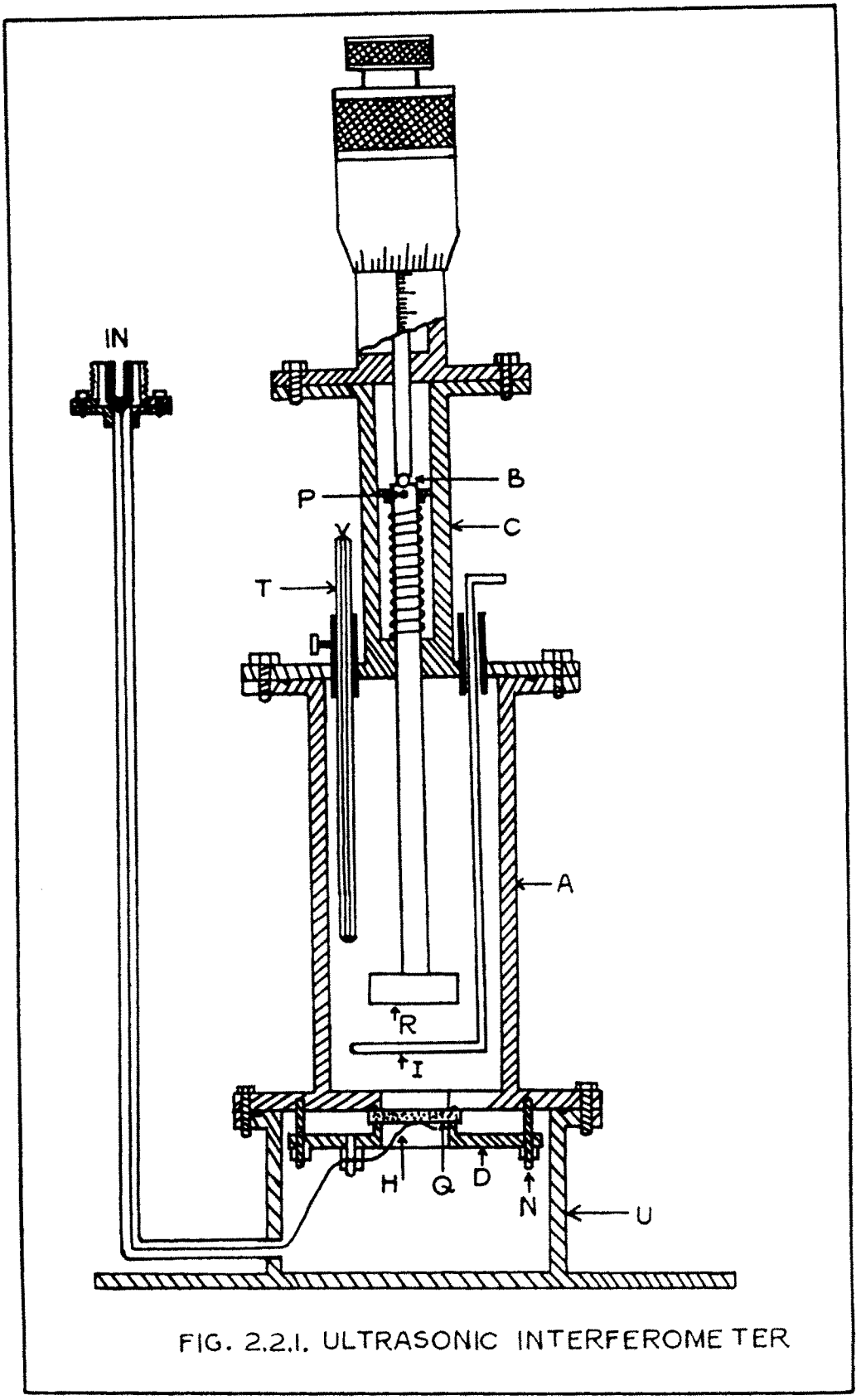
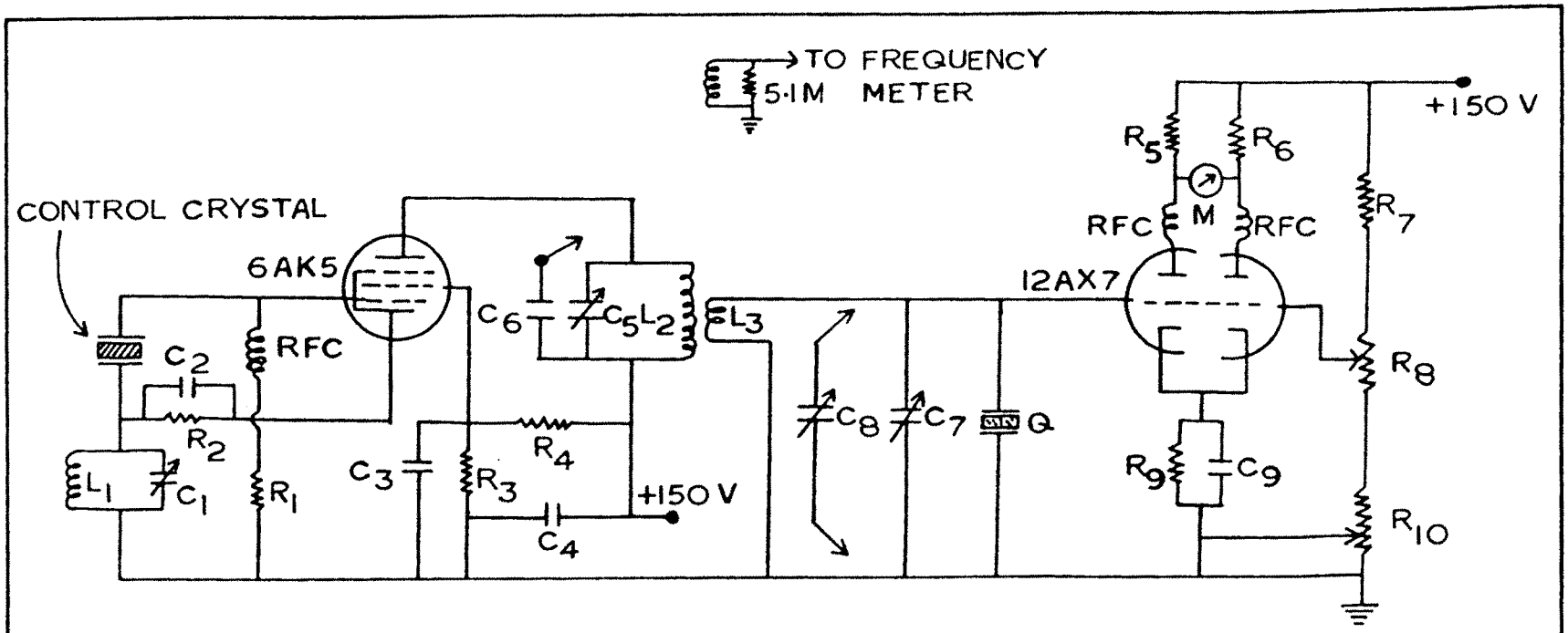


FIG. 2.2.1. ULTRASONIC INTERFEROMETER

2.3 Electronic circuitry

The electronic circuitry is shown in Fig. 2.3.1. The control crystal is an AT-cut quartz with 1 MHz natural frequency. The tank circuit in the plate of the oscillator valve can be tuned to 3 MHz. The output voltage of the oscillator is used to excite the quartz transducer 'Q' connected across the tuned circuit. The variation of the voltage across the crystal due to change in the motional impedance is detected using a different amplifier. One of the grids of the difference amplifier is fed with the r.f. voltage developed across the transducer, while the other is maintained at suitable d.c. potential which can be varied with the help of the potential division net work. This controls the sensitivity of the difference amplifier. When the currents through both the sections of the valve are nearly equal the voltage across the voltmeter terminals tends to a zero value. Variation of the radiator to reflector distance causes changes in the radiation impedance of the transducer leading to r.f. voltage variation across one of the grids. The variations after sufficient amplificiation are read using VT VM (Philips GM 6001) operated at the centre zero position.



R_1	27K	R_6	220K	C_1	50–500 pF	C_6	500 pF
R_2	15K	R_7	47K	C_2	50 pF	C_7	5 50 pF
R_3	150K	R_8	1K	C_3	0.05 μF	C_8	50 500 pF
R_4	47K	R_9	7.5K	C_4	0.05 μF	C_9	50 μF
R_5	220K	R_{10}	10K	C_5	20 - 200 pF	M	VTVM

Q TRANSDUCER

FIG. 2.3.1 .ELECTRONIC CIRCUIT OF THE TRI-TET OSCILLATOR AND DIFFERENCE AMPLIFIER-DETECTOR

2.4 Effect of diffraction on the measurement of ultrasonic velocity

Errors associated with the diffraction of the ultrasonic beam have to be taken into account for accurate determination of velocity in liquids. Del Gresso *et al.* [115] have considered this aspect in connection with the velocity determination with interferometric technique. Bass and Williams as cited by Mc Skimin [119] have shown theoretically that the correction due to diffraction in velocity, Δu, over the plane wave velocity, u, is given by

$$\frac{\Delta u}{u} = \frac{\lambda^{3/2}}{2\pi^2 r D^{1/2}} \qquad \ldots \qquad 2.4.1$$

where 'r' is the diameter of the ultrasonic beam and D is the distance from the source at which the measurements are made. Mc Skimin [119] used the above equation to evaluate the diffraction correction on the velocity measurements in solids.

Subrahmanyam *et al.* [118] studied the diffraction effects in liquids in order to test the applicability of the Eq. 2.4.1. The diffraction errors are found to be 3.2 times the value envisaged by the theory. Hence,

$$\frac{\Delta u}{u} = \frac{3.2\lambda^{3/2}}{2\pi^2 r D^{1/2}} \qquad \ldots \qquad 2.4.2$$

This relation was used in the present work to evaluate the diffraction errors and the experimental velocities are corrected suitably, since the mechanical assembly of the interferometer is similar to the one used by the above workers.

The set of values of ultrasonic velocity in pure water at 25°C, calculated from individual measurements of wavelength and after accounting for diffraction effects presented in Table 2.4.1 indicate the reproducibility of measurements.

TABLE 2.4.1

Ultrasonic velocity data in pure water at 25°C

Trial number	u ($m\ s^{-1}$)	$u - u_{mean}$ ($m\ s^{-1}$)
1	1496.68	-0.02
2	1496.68	-0.02
3	1496.76	+0.06
4	1496.72	+0.02
5	1496.64	-0.06
6	1496.68	-0.02
7	1496.72	+0.02
8	1496.72	+0.02
u_{mean} = 1496.70 $m\ s^{-1}$ Standard deviation = 0.04 $m\ s^{-1}$		

The mean value obtained in the present work is in good agreement with the data reported in literature as shown in Table 2.4.2.

TABLE 2.4.2

Ultrasonic velocity in pure water at 25°C - Comparison with literature data

u ($m\ s^{-1}$)	Reference
1496.70	Present work
1496.65	Mc Skimin [119]
1496.59	Ilgunas *et al.* [120]
1496.69	Del Grasso [121]
1496.76	Owen *et al.* [122]
1497.00	Greenspan [111]