3.1 Introduction

It is said that “seeing is believing”. Any search for a novel material or idea, therefore, relied on adequate theoretical background and most obviously, rigorous experimental validations of the conceived ideas. Present paradigms of experimental techniques, in all directions of modern science have undergone unpredicted advancements in past decades and have revealed hitherto in wondered insights of matter.

In the present course of investigation in order to pursue towards study of additive, intensive, constitutive properties of polar liquids studies of following parameters have been carried out.

1. Dielectric constant
2. Relaxation time
3. Density
4. Refractive Index
5. Viscosity

For the first two parameters, Time Domain Reflectometry technique has been adapted. Whereas the density, refractive index and viscosity have been measured by oscillating tube method, transparent detection method and cone and plate methods respectively. Following sections elucidate the necessary theories and the techniques and devices at actual.

Last few decades have experienced tremendous advancement in high frequency electronics and especially field effect devices that have asked for high demands of efficient dielectric materials. The need for study of dielectric properties of materials is aroused from practical requirements of high technical importance, thus creating a common forum for physicists, chemists and engineers to come together towards scrupulous investigations of molecular structure and dynamics in various phases of matter. Thus, the appropriateness of the use of dielectrics in modern technical application has become a factor of immense importance.

Whenever a microwave signal is incident on a matter some part of energy is transmitted through the material, some part is reflected and the rest of the energy is absorbed by the material. Such phenomenon has pronounced effect in case of dielectric materials due to the fact that dielectric materials are polarized when subject to such signal.

The term complex relative permittivity \( \varepsilon^* \) is given by following mathematical relation
\[ \varepsilon^* = \varepsilon' - j\varepsilon'' \]

where

\( \varepsilon' \) is dielectric dispersion (dielectric constant)

\( \varepsilon'' \) is dielectric absorption (dielectric loss).

The value of \( \varepsilon^* \) is a function of a varied rays of parameters i.e. frequency, temperature, pressure and intensity of the applied electric field. Among all the parameters, frequency has the highest impact on \( \varepsilon^* \). Hence, in order to obtain the complete information about the dielectric behaviour of the system, measurement of \( \varepsilon' \) and \( \varepsilon'' \) have to be studied over a wider range of frequencies. Relaxation time can be further derived from the study of frequency dependent permittivity. Finally, these parameters can be correlated to figure out the molecular structure or dynamics related information.

The traditional methods of dielectric permittivity were centered, around the measurements in the frequency domain. The common feature of all such methods being that the solution contained in the sample holder and its complex permittivity is measured at various discrete frequency points. It is, however not possible to design one piece of apparatus which can give the required frequency coverage. The experimental methods depend on range and type of the system under consideration. At low frequency, the bridge techniques [1-4] are used, with which one can obtain complex permittivity at frequency, as low as 1 KHz. The upper frequency limit of bridge tends to be 100 to 250 KHz. At higher frequencies, transmission lines are used. Transmission lines may be coaxial line or wave guide. The frequency range of a coaxial line, is around 50 MHz to 20 GHz. Above this and below 100 GHz, wave guides are employed. Above 100 GHz free space methods are used [5]. At microwave frequencies 0.1 GHz to 100 GHz, traveling wave method, described by Buchanan [6] is used. The reflectometer technique has been used by Surber [7] to measure dielectric parameters of liquid dielectrics. Heston, Hennelly and Smyth [8] have devised a method for the measurement at complex dielectric constant of high loss liquid dielectrics. The method is based upon the measurement of variation in the reflection coefficient of a uniform layer of dielectric, as the depth of the layer is changed by means of movable plunger. Smyth [9] has further improved this method for the measurement of dielectric parameters for low loss liquids.
Transmission measurement techniques such as Microwave Bridge [10-11], Heterodyne method [12, 13] and resonance method [14-16] have been used.

Complex permittivity can be measured by probing standing wave pattern at regular intervals [17]. A standing wave method has been developed by Bramin and Smyth [18], using a traveling probe immersed in the liquid filled slotted section. In the travelling wave method, simultaneous absolute measurement of guide wavelength in column and adsorption index can be made, which are then used to calculate $\varepsilon'$ and $\varepsilon''$. Poley [19] devised a technique to measure the dielectric constant $[\varepsilon']$ and loss factor $[\varepsilon'']$ by incorporating the measurements of V.S.W.R. at varying depths at liquid column and guide wavelength in the liquid dielectric. Dagg and Reesor [20] have developed a method for the measurement of complex dielectric by measuring standing wave ratio in a liquid filled guide. They have successfully used the technique to measure very low losses in the liquid dielectrics. Fatuzzo and Mason [21] have developed a method for the measurement of complex dielectric constant, using reflection measurements on a liquid filled line section terminated by a short circuit. This method is particularly suitable for the measurements on dilute solutions of polar compounds in non-polar solvents.

Recently sampling of the standing wave pattern at regular intervals has been improved by Jordan and Grant [22], Vanloon and Fincy [23], Rodriguezvidal [24]. Also the automated data processing Sheppard [25], Szwarowsui and Sheppard [26], essentially increased the experimental sensitivity. For that reason, precise data on a large variety of liquids can now be obtained by standing wave observations. Puranik et. al., [27] have used X-band setup to measure complex permittivity.

### 3.2 Time Domain Spectroscopy

Since we have adapted time domain spectroscopy for present investigation a bit detailed discussion should precede the actual experimental set up. The time domain methods rely on studying the nature of a step voltage pulse before and after impulse with the material of interest. This method is highly accurate since it offers fast polarization of dielectric material [28]. The present status of instrumentation in this sector provides resolution of nearly 10-11 ns [28-33] thus enabling time domain studies of even low viscosity polar liquids [34-36]. As compared to frequency domain microwave techniques, time domain spectroscopy (TDS) has number of advantages. TDS is
conceptually simple, precise method which requires a small sample and cover wide frequency range from 10 MHz to 10 GHz or even more in a solitary measurement. For this reason, during the last two decade, this new experimental technique has become pioneer to study the dielectric relaxation mechanism of liquids.

Initially this method was used for low frequency dispersion by Davidson et.al [37]. In mid 60’s the Hewlett-Packard introduced a time domain system with a frequency response up to 12 GHz [38]. Originally, the apparatus was used mostly by the engineers, for making measurements on the solid state devices [39-41].

TDS was introduced into the field of liquid dielectric by Fellner-Feldegg [42-44]. TDS methods can be classified as slow response and fast response techniques [45]. The slow response technique has been reviewed by Vaughan [46], Sugget [47] and Hyde [48]. Cole [49], Cahine and Bose [50] have modified the TDS technique to minimize error in measurement and increase the frequency range in the dielectric studies. Later on, Cole [51] suggested precision difference methods for evaluation of dielectric behavior in time domain. Dawkins [35] described a method of approximation. Gestblom [52] suggested the single reflection method in TDS. K. Baba [54] developed a lumped capacitor TDS technique. Recently Cole et.al [54] has developed a magnificent method of bilinear correction to eliminate the reflections at higher frequencies.

Time Domain Technique has been used to get complex permittivity spectra of various materials viz. Solids [54], organic liquids [53-57], aqueous solutions [58, 59], electrolytic solutions [60-63], biological materials [64], agricultural samples [65] and biological samples [66-68].

There are two methods of measurements in time domain spectroscopic techniques.

1) Time Domain Transmission method

2) Time Domain Reflectometry method.

3.2.1 **Time Domain Transmission Method (TDT)**

Time Domain technique based on transmission method is called as transmission dielectric time domain spectroscopy. In TDT method, the signal emerging from the sample is sampled with incident signal.

3.2.2 **Time Domain Reflectometry (TDR)**

TDR technique is used for observing the time dependent response of a sample of
interest after application of time dependent electromagnetic field. Steady state measurement of a complex quantity, expresses amplitude and phase relation to an alternating sinusoidal field. For systems with linear response characteristics, the two approaches are capable in principle of giving the same information, expressed differently, with the relation between the two forms, a Laplace Transform or its inverse. (The wave shape of reflected pulse from the sample is compared with the incident pulse).

Gestblom et.al [69-71] has used TDT method for low loss liquids. Cole [53] has used TDR method for the dielectric relaxation measurements. Sugget [72, 73] used TDR techniques to study amides and peptides. The advantage with TDS method is that one can get frequency response over a wide range with a single short measurement. The instrument setup is quite simple than that used in frequency domain technique.

3.3 Experimental Setup

The photograph of actual setup used for the present work is shown in figure 3.1. It consist of digitizing (sampling) oscilloscope HP 54750A [74], TDR module HP 54754A [75] a transmission line, sample cell and the temperature bath. All these components (in experimental setup) are discussed in the following sections. Block diagram of experimental setup is shown in figure 3.2.

3.3.1 HP 54750 A Digitizing oscilloscope

It is a high performance mainframe that contains digitizer, time base and display circuitry. The HP 54750A is the state-of-art instrument provided with TDR/TDT module and a 1.44 MB floppy disk drive [74, 75]. The working of the oscilloscope is controlled by the front panel keys. The oscilloscope also can be programmed by a controller or computer and communicated by HP-IB setup. The necessary ports and software is provided with the oscilloscope. At present oscilloscope is being controlled by the front panel control keys only.

Control Keys:

Control Keys are classified into two groups

i) Instant Action Keys:

Clear Display, Run, and Stop Signal are the examples of instant action keys (which are also called as HARD Keys), because as soon as you press them, the instrument changes its operating status.
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Figure 3.1 (c): Reflected waveform [with sample Rx(t)]

Figure 3.2: Block Diagram of Dual Channel TDR Unit.
ii) Menu keys:

Acquisition, Time base, Disk etc. are the examples of menu keys. Pressing them causes SOFT key menus to come on the right side of the display. To activate a function on soft key menu, a key next to the annotation on display is pressed. Some keys have two functions. The second alternative function is printed in BLUE. To activate these functions SHIFT key is pressed before pressing the key of interest.

Hardware of HP-54750A:

a) Interface Circuitry

The interface circuitry accepts the commands from system microprocessor to control the module functions such as IF gain, sampler band width and optical channel bandwidth. Non-volatile RAM within the module provides the system microprocessor with module CAL factors, as well as information on the type of plug-in installed.

b) A/D Converters

The A/D converters traces the peak value of the analog pulse at its input and converts it into a 12 bit digital word. These 12 bit words are put into a FIFO memory which is then read and processed by the CPU.

c) CAL Table

The CAL table automatically adds the calibration factors to the sampled data. The result is then transferred to system bus.

d) Microprocessors and Co-processors

There are two 32-bit microprocessors, one 32-bit co-processor and one 16 bit co-processor in the main frame.

Motorola 68020 is 32-bit microprocessor that controls system hardware and also acts as a traffic controller on the system bus. Motorola 68882 is a 32-bit co-processor that performs all floating point math. TMS 34010 is another 32 bit microprocessor that draws data on the display. HP custom graphics co-processor is a 16-bit co-processor that controls gray scale persistence mode and also writes blocks of data to the display.

e) Host RAM and Flash ROM

The host RAM is 4 MB of non-volatile RAM. This is where the waveform data is held and manipulated. In addition, this is the location of the current front-panel setup, setup memories and waveform memories.

The flash ROM contains the system firmware that controls the operation of the instrument. You can load new system firmware into the instrument by using the disk
drive.

f) **User Interface, FIFO and HP-IB hardware**

   It consists of keyboard and the hardware that interfaces the keyboard and knob with the system bus. FIFO is first-in-first-out memory that transfers waveforms through the HP-IB bus under hardware control.

g) **Ports**

   The centronics port is a parallel connector for printers compatible with centronics interface.

h) **Video RAM and Display**

   The system use 1 MB fast video RAM for storing the display image. The Video RAM also contains the pixel memory. The color display has high resolution. The display is divided in graticules, which helps in comparing any minute change in signal under study.

i) **Floppy Disk Drive**

   The system is provided with a 3.5”, high density, MS-DOS compatible disk drive. You can use the disk drive to load the system firmware into the flash ROM, Load applications, save waveforms and load waveforms.

3.3.2 **Plug-in Module: HP 54754A**

   The HP 54754A plug-in module provides two accurate oscilloscope measurement channels with user selectable bandwidth of 12.5 GHz or 18 GHz. The high bandwidth mode provides high-fidelity display and measurement of very high-speed waveforms. It is capable of performing both single ended TDR/TDT measurements and differential TDR/TDT measurements. The TDR rate can be selected manually or automatically for repetition rate of TDR step from 50 Hz to 250 KHz. When this function is set to automatic, the TDR step repetition rate varies as the time base scale is changed to keep multiple steps off screen.

   It has 3.5 mm connectors. The plug in module takes up two out of four mainframe slots. In single ended TDR measurement, a positive going step (a fast rising step voltage pulse of 200 mV with 39 ps rise time) is launched on one of the channels while the other channel is terminated using a short. In differential TDR measurement, a positive going step is launched on channel 1 and an effective negative going step is launched on channel 2. The response controls are provided which shows the single ended or differential mode response of a TDR system under test.
a) Pulse generator

The pulse generator unit consists of trigger input, trigger generator, sequential delay generator and pulse filter. The plug-in trigger input passes the trigger input directly into the mainframe. The trigger generator receives trigger input and sends a signal to sequential delay generator, whenever an edge meeting criteria is received the trigger is enabled. The sequential delay generator controls the time between when it receives trigger input from a trigger generator and when it sends a trigger output to the pulse filter. This delay time increase sequentially with each trigger event, allowing the trace to built up from left to right across the screen. The delay values depend upon the record length and time base setting and are programmed by the system microprocessor.

b) Sampling head

The sampling head or sampler consists of a diode gate and sampling capacitor. The incoming signal is applied to the input of the diode gate which is normally biased off. When the trigger event occurs, the pulse filter produces a pulse which momentarily forward biased the gate and allows the input signal to change the capacitor. It consists of an IF amplifier which senses the voltage on sampling capacitor and conditions it in preparation for digitizing the A/D converter.

Sampling gate closed for short amount of time and allowing the signal to charge shunt capacitor through source resistance does sampling. Sampling efficiency is given by,

$$\frac{V_{out}}{V_{in}} = \left[1 - e^{-\frac{t}{RC}}\right]$$

(3.1)

Shannon sampling theorem states that, to reliably extract all the information in signal, it must be sampled at the rate at least twice the signal highest frequency.

Thus to sample 20 GHz signal, sampling rate must be 40 GHz which is not possible with fastest A/D converter available. This difficulty has been overcome by using repetitive sequential sampling. In the sampling process, the input signal is sampled once per trigger event. The first trigger event in sequence causes the input signal to be sampled at an initial delay. On next trigger event (at a subsequent repetition of waveform), the sampling instant is delayed by small amount relative to initial delay. Each additional trigger event causes the sampling instant to be delayed by greater amount of time, so that after many triggers the input waveform builds on the screen form left to right.
c) The Sample Cell

The sample cell [76] holds the liquid under consideration. The impedance of the cell should be matched with coaxial transmission line to which cell is connected. If there is impedance mismatch then, unwanted reflections may disturb the wave, thereby causing some errors in the measurements. Thus physical dimensions (inner diameter of outer conductor, the length and outer diameter of inner conductor) of the cell are very important, so one must be careful while designing the sample cell. The length of the inner conductor is called as ‘pin length’ of the cell and is very important factor in the analysis. The sample length must be enough to avoid unwanted reflections.

In total reflection method, the sample length must be long enough to produce an adequate difference signal but short enough to keep less complication of resonance effects at frequencies above the range of interest. The characteristics impedance of a coaxial line is given by,

\[ Z = \frac{138.2}{\sqrt{\varepsilon'}} \log_{10}\left(\frac{b}{a}\right) \]  

(3.2)

where ‘a’ is the outer diameter of inner conductor, ‘b’ is the inner diameter of outer conductor and \( \varepsilon' \) is the relative permittivity of the dielectric medium placed between the conductors. This impedance for our transmission line is frequently 50Ω. Using Teflon and taking appropriate dimensions of inner and outer conductor, a cell can be designed to get a matching impedance of \( Z = 50 \Omega \) (for air \( \varepsilon' = 1 \) and for Teflon, \( \varepsilon' = 2.2 \)). The SMA type cell is having inner diameter 3.5 mm. The inner conductor of SMA connector itself considered as ‘inner conductor’ and hex-nut acts as an outer conductor. Since these SMA connectors have already designed for precise impedance of 50 Ω, a special design when used with high frequency is not required. The physical length of inner conductor can be changed. The geometrical construction of this open ended sample cell is given in figure. 3.3 (b) (The inner and outer diameters are not shown as per scale needed for 50 Ω transmission line).

d) Effective pin length

When cell is filled with sample, the fringing effect [77-79] takes place. Due to the fringing field the effective pin length [77] will not be equal to physical pin length. The effective electrical pin length will be more than the physical pin length. The accurate determination of effective pin length (d) is very important for the accurate evaluation of
dielectric parameters. Figure 3.3 (a) shows SMA cell dimensions with effective pin length. It is found that for SMA type cell effective pin length is greater than actual physical length by 0.1-0.2 mm.

![Figure 3.3 (a) Fringing field and SMA cell dimensions](image)

![Figure 3.3(b): Geometrical construction of SMA cell](image)

e) **Temperature bath**

Temperature dependent dielectric measurements were done by using temperature bath shown in figure 3.4. The temperature of sample was maintained at desired value within the accuracy limit of ±1ºC, by circulating constant temperature water through heat insulating jacket surrounding the sample cell.

![Figure 3.4: Constant Temperature bath.](image)
3.4 TDR Fundamentals

The quality of transmission line has been checked by measuring Standing Wave Ratio (SWR). But SWR measurement cannot figure out the discontinuities separately. SWR measurement must be done at various frequencies to understand broadband response of the transmission line.

In TDR technique, a voltage step is propagated through the transmission line under investigation and the reflected voltage waves are monitored by oscilloscope at particular point on the line.

TDR measurement can give characteristics impedance of line and it shows both position and nature (resistive, inductive or capacitive) of each discontinuity along the line. TDR also demonstrate whether losses in transmission line system are series losses or shunt losses. Furthermore, TDR measurements give meaningful information regarding broadband response of transmission line.

a) Propagation of signal along transmission line

The equivalent circuit for transmission line is shown in figure 3.5. If C, G, L and R are defined per unit length for infinite long transmission line. Then we can write,

\[ Z_{in} = Z_0 = \frac{R + j\omega L}{G + j\omega C} \]  

(3.3)

where \( Z_0 \) is characteristics impedance of transmission line.

A voltage pulse enter at the input of transmission line requires finite time to travel distance ‘x’ along the line. The phase of the voltage moving along the line lags behind the voltage introduced at input by amount \( \beta \) per unit length. Furthermore, voltage will be attenuated by an amount \( \alpha \) per unit length by series resistance and shunt conductance of the line. The phase shift and attenuation are defined by propagation constant \( \gamma \) as

\[ \gamma = \alpha + j\beta = \sqrt{(R + j\omega L)(G + j\omega C)} \]  

(3.4)

Velocity with which voltage propagates along the line can be written as

\[ V_p = \frac{\omega}{\beta} \text{ unit length per sec} \]  

(3.5)

The velocity of propagation approaches \( V_c \) for transmission line with air dielectric. For general case where \( \varepsilon_r \) is dielectric constant of medium

\[ V_p = \frac{V_c}{\sqrt{\varepsilon_r}} \]  

(3.6)
The voltage and the current at any distance ‘x’ along the transmission line can be written in terms of propagation constant $\gamma$ as,

$$ E = E_{in} e^{-\gamma x} \quad \text{and} \quad I = I_{in} e^{-\gamma x} \quad (3.7) $$

Since the voltage and current at any point ‘x’ is known, characteristics impedance of the line can be written as,

$$ Z_0 = \frac{E_x}{I_x} = \frac{E_{in} e^{-\gamma x}}{I_{in} e^{-\gamma x}} = \frac{E_{in}}{I_{in}} = Z_{in} \quad (3.8) $$

When transmission line is terminated in a load whose impedance matches the characteristic impedance of the line, then voltage and current relationships are satisfied by preceding equations (3.8).

If $Z_L$ is not equal to $Z_0$, the incident energy is not fully delivered to load and propagates back towards source. The ratio of amplitude of reflected wave to incident wave is called reflection coefficient ($\rho$).

$$ \rho = \frac{E_r}{E_i} = \frac{Z_L - Z_0}{Z_L + Z_0} \quad (3.9) $$

The magnitude of steady state sinusoidal voltage along the line terminated in load other than $Z_0$ varies periodically as a function of distance between minimum and maximum value. This variation is called standing wave. The ratio of maximum to minimum values of this voltage is called the Voltage Standing Wave Ratio (VSWR). It is related with reflection coefficient by equation

$$ \sigma = \frac{1 + |\rho|}{1 - |\rho|} \quad (3.10) $$

Figure 3.5: Equivalent circuit of transmission
b) Step reflection from purely resistive loads

The TDR technique uses reflected pulse from sample to determine dielectric parameters. The block diagram for step reflection from load is shown in figure 3.6. It is very interesting to observe response of different type of loads to the incident step. The shape of the reflected pulse is valuable since it reveals both the nature and magnitude of mismatch. The typical examples for different values of purely resistive load are shown in figure 3.7. The knowledge of $E_i$ and $E_r$ measured on oscilloscope allows $Z_L$ to be calculated in terms of $Z_0$. Assuming $Z_0$ to be real (as in precision cables), it is seen that, mismatch reflects a voltage of the same shape as the driving voltage with magnitude and polarity of $E_r$ determined by relative values of $Z_L$ and $Z_0$.

\[ Z_L = \frac{E_r}{E_i} \]

\[ \text{Figure 3.6: Response to step pulse.} \]

c) Step reflections from complex loads.

The response of complex loads of different types to incident step pulse is as shown in figure 3.8. The reflected voltage from complex loads is evaluated at $t=0$ and $t=\infty$ by assuming any transition between two points to be exponential.

i) Series R-L

At $t=0$, reflected voltage is $+E_i$. This is because inductor will not accept sudden changes in current and initially it looks like infinite impedance. Then current in $L$ builds up exponentially and its impedance drops down towards zero. At $t=\infty$, $E_r(t)$ is determined only by the value of $R$.

\[ E_r(t) = \frac{R - Z_0}{R + Z_0} \]

at $t=\infty$ \hspace{1cm} (3.11)
Figure 3.7 Reflection of step pulse from different purely resistive Loads
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Figure 3.8: Reflection of step pulse from different complex loads
The exponential transition of $E_r(t)$ has time constant determined by effective resistance seen by inductor. Since the output impedance is $Z_0$ in series with $R$

$$\tau = \frac{L}{R + Z_0} \quad (3.12)$$

The reflected voltage can be written as,

$$E_r = E_i \left[ \left(1 + \frac{R-Z_0}{R+Z_0}\right) \left(1 - \frac{R-Z_0}{R+Z_0}\right)e^{-t/\tau} \right] \quad (3.13)$$

ii) Shunt R-C

At $t=0$, load appears as a short circuit. Since capacitor will not accept sudden changes in voltage and thus $E_r = -E_i$. At $t=\infty$, capacitor is effectively an open circuit and $Z_L = R$.

$$\therefore \rho = \frac{R-Z_0}{R+Z_0} \quad (3.14)$$

Resistance seen by capacitor is $Z_0$ in parallel with $R$. Therefore the time constant of exponential transition is

$$\tau = \frac{Z_0R}{R+Z_0}C \quad (3.15)$$

The reflected voltage can be written as,

$$E_r = E_i \left[ \left(1 + \frac{R-Z_0}{R+Z_0}\right) \left(1 - e^{-t/\tau}\right) \right] \quad (3.16)$$

iii) Shunt R-L

At $t=0$, $Z_L = R$ (assuming $R > Z_0$) and at $t=\infty$, $Z_L = 0$. Impedance seen by inductor is parallel combination of $R$ and $Z_0$. Thus,

$$\tau = \frac{R+Z_0}{R.Z_0}L \quad (3.17)$$

The reflected voltage $E_r$ is given by equation

$$E_r = E_i \left[ \left(1 + \frac{R-Z_0}{R+Z_0}\right) \left(e^{-t/\tau}\right) \right] \quad (3.18)$$

iv) Series R-C

At $t=0$, $Z_L = R$ (assuming $R > Z_0$) and at $t=\infty$, $Z_L = \infty$. Impedance seen by capacitor is series combination of $R$ and $Z_0$. Thus

$$\tau = (R + Z_0).C \quad (3.19)$$
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The voltage $E_r$ is given by equation

$$E_r = E_i \left[ 2 - \left( 1 - \frac{R - Z_0}{R + Z_0} \right) e^{-\tau/\tau_0} \right]$$  \hspace{1cm} (3.20)

3.5 Sources of Errors

The primary sources of errors in TDR measurements are

(a) Step Generator       (b) The Oscilloscope
(c) Cables and connectors (d) Selection of Time Window

a) Step Generator

The shape of the step pulse is important for accurate TDR measurements. The Device under Test (DUT) responds not only to the step but also to aberrations on the step such as over short and non-flatness. Drift in step voltage with time cause remarkable change in frequency dependent permittivity spectra due to wrong time referencing. The use of proper warm up time reduces errors in measurement due to drift in pulse. The rise time of the step is very important. It decides the maximum frequency limit of frequency range in which complex permittivity spectra can be obtained. Measurement made at inappropriate rise time can yield invalid conclusions.

b) The Oscilloscope

Oscilloscope introduces errors in measurement in several ways. The finite bandwidth of oscilloscope translates to the limited rise time. Edge with a rise times that approach the minimum rise time of oscilloscope is measured slower than they actually are. When we need to measure how a device responds to a very fast edge, the oscilloscope’s limited rise time may distort or hide some of the device response.

The oscilloscope can also introduce small errors due to trigger coupling into channels and channels cross talk. These errors appear as ringing and other non-flatness in display of measured channel baseline which get superimposed on the measured waveform. These effects are generally small and only significant when measuring very small signals.

b) Cables and Connectors

Cables and Connectors between step generator and DUT can be significantly affecting the TDR measurements. The impedance mismatch and imperfect conductors add reflections to the actual signal being measured. These reflections can distort signal and make it difficult to determine actual reflection from DUT.
In addition, cables are imperfect conductors that become more imperfect as frequency of signal increases. Cable losses increase at higher frequencies, increase the rise time of edges and cause edges to droop as they approach their final value. Although the major sources of unwanted reflections are known in practice, it is extremely difficult to determine their exact contribution in the reflection coefficient. The effect of unavailable reflections is reduced by using numerical smoothening techniques in the portion in which they are present in the response signal.

c) Selection of the time window

The choice of time window through which the reflected signals are observed has to be related according to the frequency range of interest. The minimum frequency observable value is $f_{\text{min}} = 1/ (\text{Time Window})$ while the maximum frequency observable is $f_{\text{max}} = N/ (2 \times \text{Time Window})$, where $N$ is the number of points used to sample and digitize the signal. Smaller time window causes loss of signal while larger time window includes unwanted reflections. Thus proper selection of time window is important to minimize these effects. In present work time window of 5 ns is used.

For digitizing the signal, it is necessary to select number of points per waveform in the time window. To reduce the noise, an averaging of signals 16, 64 or 512 times can be done. If signal is averaged over 64 times or more, noise will be reduced significantly.

3.5.1 Error Minimization

Cables and connectors of good quality can significantly reduce the errors due to unwanted reflections in measurement. Precision in connections can also reduce errors in measurement. The error due to drift in incident step is significant when instrument is switched on. Give at least 30 minute warm up time for instrument to minimize these errors.

One of the ways to minimize error is to use method of waveform subtraction. Response of a known good reference device is measured and reference waveform is stored in memory. The reference waveform could then be subtracted from the waveform measured from DUT. The result shows how response differs from reference response. This technique removes error in terms common to both DUT and reference device such as trigger coupling, channels cross talk and reflections from cables and connectors. But subtracted waveform describes how DUT differs from reference device but not actual response of DUT, without errors introduced by test system. A digital error correction method called normalization can significantly reduce errors from TDR measurement.
Normalization can predict how the DUT will respond to an ideal step of used defined rise time. This method can also increase bandwidth (i.e. decrease rise time) of system by some amount depending on the noise floor. The normalization method is not used in the present work.

### 3.6 Experimental Procedures and Data Analysis

The actual experimental setup is shown in figure 3.1 while the block diagram of TDR is shown in figure 3.2. The TDR unit is used for measurements after warming up at least 30 min. This is necessary to minimize the time drift which occurs between two consecutive readings. The time window is kept 5 ns to keep the unwanted reflections off screen and for the desired frequency coverage. The reflected step pulse is digitized in 1024 points and waveform is averaged over 64 times for each measurement. A flexible coaxial cable of 1m length is connected between TDR unit and sample holder. The SMA cell with 1.35 mm effective pin length is used as sample holder. After connecting transmission line and sample holder to TDR unit, the reflected waveform is observed carefully. The unwanted reflections in reflected step at point of contact between transmission line and TDR unit as well as transmission line and sample cell are minimized by ensuring proper contact between these components. The reflected pulse without sample $R_1(t)$ is averaged 64 times and acquisition is stopped. This acquired waveform is then transferred to floppy disk in TDR mainframe. Reflected pulse with sample $R_s(t)$ is recorded by the same method by putting liquid sample in SMA cell. The data files for reflected pulse without sample $R_1(t)$ and with sample $R_s(t)$ stored on floppy disk and transferred to computer for further processing. The pulses $R_1(t)$ and $R_s(t)$ are subtracted to get $p(t) = R_1(t) - R_s(t)$ and added to get $q(t) = R_1(t) + R_s(t)$ by using computer program. The file containing data array $p(t)$ and $q(t)$ is termed as TDR file.

#### 3.6.1 Fourier Transformation

The time domain data is converted to frequency domain data using Fourier Transform. While performing Fourier Transform one should be careful about the nature of the curve which is to be obtained. Since nature of curve for $p(t)$ and $q(t)$ are not same, different methods of Fourier Transforms are used.

The Fourier Transform of $p(t)$ is obtained by summation method [80] using equation.

$$p(\omega) = T \sum_{n=0}^{N} \exp(-i\omega t) p(nT)$$  \hspace{1cm} (3.21)
The Fourier Transformation using summation methods have some limitations that for all the sampling intervals, the nature of pulse form must be known [75]. Furthermore, the transform $p(\omega)$ is simply the area under the curve $p(t)$ which has an initial peak followed by a decay to zero or a finite limiting value strictly to infinite time.

The pulse form of $q(t)$ is not known exactly. Therefore summation method of Fourier Transforms cannot be used for $q(t)$ curve. The Fourier Transform of such type of curves can be obtained with the Samulon method [81-84] as follows,

$$q(\omega) = \frac{T}{1 - \exp(-i\omega T)} \left[ \sum_{n=0}^{N} (q(nT) - q((n-1)T)) \exp(-i\omega nT) \right]$$

(3.22)

In equation 3.21 and 3.22, $\omega$ is angular frequency, $T$ is the sampling interval or time difference between two adjacent points and $N$ is number of points. In our experiment $N$ is 1024 points and $T$ depends on time window and number of points per waveform ($N$). For example, if time window is 5 ns then $T$ is 4.88 ps for 1024 points per waveform. Thus the time domain data is converted into frequency domain data in frequency range of 10 MHz to 20 GHz.

The frequency domain data obtained from Fourier Transform is further used to determine frequency dependent complex reflection coefficient $\rho^*(\omega)$ given by equation,

$$\rho^*(\omega) = \frac{C}{i\omega d} \frac{p(\omega)}{q(\omega)}$$

(3.23)

The single reflection method has an advantage of giving a reflection coefficient of magnitude $0.3 < |\rho| < 1$ over the whole frequency spectrum present in the incident pulse. Thus, it can be considered a true wide frequency method even reaching $> 20$ GHz, if sufficient accuracy in $\rho^*(\omega)$ is achieved. The demands on the accuracy in $\rho^*(\omega)$ are quite severe at high permittivity and high frequencies. This method has been mostly used for liquids dielectric medium.

The complex reflection coefficient spectra is called as ‘raw’ data, using this ‘raw’ data complex permittivity can be determined [29] as follows

The basic equation for determining relative complex permittivity $\varepsilon^*$ of the sample derived from transmission line theory is conveniently written in simple form as,

$$\varepsilon^*(\omega) = \frac{C}{i\omega d} \frac{r_0 - r_s}{r_0 + r_s} Z \cot(Z)$$

(3.24)
where \( Z = \frac{\varepsilon \omega d}{C} \sqrt{\varepsilon^*} \) and \( r_0, r_x \) are the Fourier Transform of the pulses from the cell without sample \([R_0(t)]\) and with sample \([R_x(t)]\) respectively.

If we consider only single reflection then \( z \cot z = 1 \) and equation (3.24) can be written as,

\[
\varepsilon^*(\omega) = \frac{C}{i \omega d} \frac{r_0 - r_x}{r_0 + r_x}
\]

Equation (3.25) indicates that the dielectric constants of unknown sample can be found if the time profile of the incidents \( r_0 \) and reflected \( r_x \) pulses are recorded within a frequency range determined by the time limits of \( r_0 \) and \( r_x \).

A working equation more convenient than equation (3.25) can be obtained as follows by rearranging it.

\[
r_0 = \frac{\left[ \varepsilon^* + \frac{C}{i \omega d} \right]}{\left[ \frac{C}{i \omega d} - \varepsilon^* \right]} r_x
\]

If the sample cell is without any liquid i.e. with air is considered the \( \varepsilon^* = 1 \) and we get

\[
r_0 = \frac{1 + \frac{C}{i \omega d}}{\frac{C}{i \omega d} - 1} r_1
\]

where \( r_1 \) is reflected pulse in case of air with \( \varepsilon^* = 1 \), eliminating \( r_0 \) from equation(3.26) and (3.27).

\[
\frac{r_1}{r_x} = \frac{\left[ \varepsilon^* + \frac{C}{i \omega d} \right]}{\left[ \frac{C}{i \omega d} - \varepsilon^* \right]} \times \frac{C}{\left[ 1 + \frac{C}{i \omega d} \right]} (3.28)
\]

The equation can be rearranged to get,

\[
\varepsilon^* - 1 = \frac{1 + \left[ \frac{\omega d}{c} \right]^2}{\left[ 1 - \left[ \frac{\omega d}{c} \right]^2 \right]} \rho^*
\]

or \( \varepsilon^* - 1 = \frac{(1 + A) \rho^*}{1 - B \rho^*} \)
where $A=B=\left[\frac{\omega d}{c}\right]^2$

Thus using equation (3.29) one can obtain complex permittivity spectrum in the desired frequency range.

Using this complex permittivity spectrum, static dielectric constants ($\varepsilon_s$), dielectric constants at infinite frequency ($\varepsilon_\infty$) and relaxation time ($\tau$) can be calculated by using Havriliak-Negami expression [81].

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + (i\omega\tau)^{1-\alpha}}$$

(3.31)

### 3.6.2 Bilinear Calibration Method

The problem in TDR experiment arises from the fact that the characteristic impedance of the transmission line connecting the measuring plane of the cell is generally not uniform along the propagation axis. This fact introduces reflections of the traveling pulses superimposed on the reflection from the sample cell. The above expression (3.31) is obtained by considering the transmission line as ideal, so that $A=B=(\omega d/c)^2$ but under experimental conditions transmission line can’t be an ideal i.e

$$A \neq B \neq \left[\frac{\omega d}{c}\right]^2$$

To minimize the effect of unwanted reflections, several workers [76-78] have suggested different methods. Giese and Tieman [85] described a method by which it is possible to take in to account the influence of unwanted reflections quantitatively. Cole et.al [48] has described bilinear calibration unwanted reflections at high frequencies. In the low frequency limit

$$\lim_{\omega \to 0} \rho^* = \varepsilon^* - 1$$

So, the permittivity at low frequency can be obtained directly by determining $\rho^*$. Corrections in raw data are necessary to get accurate values of permittivity at high frequencies. The unwanted reflection also depends on pin length of the sample cell used. For different pin lengths the position of randomness of data shifts either to low frequency side or to high frequency side in the spectrum.

The calibration process suggested by Cole et.al [54] involves experimental determination of values of $A^*$ and $B^*$ for getting reliable values of $\varepsilon^*$ at higher
frequencies from $\rho^*$. The experimental permittivity is given by.

$$\varepsilon^* - 1 = \frac{(1 + A \rho^*)}{1 - B \rho^*} \tag{3.32}$$

The frequency dependant values of $A^*$ and $B^*$ are determined using two or more standard liquids. We have $A^* = A' - jA''$ and $B^* = B' - jB''$. For standard liquids, the values of $\varepsilon^* = \varepsilon' - j\varepsilon''$ can be obtained theoretically by using suitable model such as Debye, Davidson-Cole etc.

Writing equation (3.32) for two standard liquids

$$\varepsilon_{1}^* - 1 = \frac{(1 + A \rho_{1}^*)}{1 - B \rho_{1}^*} \tag{3.33}$$

$$\varepsilon_{2}^* - 1 = \frac{(1 + A \rho_{2}^*)}{1 - B \rho_{2}^*} \tag{3.34}$$

Knowing the values of $\varepsilon_{1}^*$, $\rho_{1}^*$ and $\varepsilon_{2}^*$, $\rho_{2}^*$, equation(3.32) and (3.34) can be solved to determined the values of $A^*$ and $B^*$. At least two standard liquids are needed in order to evaluate four unknowns $A'$, $A''$, $B'$ and $B''$. Using two standard liquids gives four linear equations which can be solved to get these four unknowns at all frequencies.

It is observed that, the accuracy of calculated permittivity depends on the choice of the calibrating liquids. It is important to use proper calibrating liquids. In order to improve the accuracy, the raw spectra of calibrating liquid should be closer to that of liquid under investigation. Cole et.al [54] has suggested many calibrating liquids with different kinds of dielectric spectra for determination of values of $A^*$ and $B^*$.

After getting the frequency dependant values of A’s and B’s, these values are considered as constant and using equation (3.32). $\varepsilon^*(\omega)$ of unknown samples can be determined experimentally. Thus using bilinear calibration process one can eliminate non-ideal configuration of transmission line and imperfections of the system including spurious reflections.

The frequency dependant permittivity spectrum obtained by calibration process is called as ‘cor’ data. An example of corrected spectra along with raw spectrum is shown in figure 3.9. Using ‘cor’ data, dielectric parameters can be obtained using equation 3.31. The dielectric parameters viz. static permittivity ($\varepsilon_s$), relaxation time ($\tau$) and high frequency limiting dielectric constant ($\varepsilon_\infty$) are calculated by fitting ‘cor’ data to the
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suitable dielectric model using nonlinear least squares fit method [82].

![Graph showing reflection coefficient spectrum (ρ*) and complex permittivity spectrum (ε*)](image)

Figure 3.9 Typical reflection coefficient spectrum (ρ*) and complex permittivity spectrum (ε*) obtained by bilinear calibration method.

### 3.7 Determination of Effective Pin Length

An effective pin length (d) can be determined by considering equation (3.23) in the limit \( \omega \to 0 \)

\[
\text{Limit } \omega \to 0 \rho^* = \lim_{\omega \to 0} \left( \frac{c}{j \omega d} \right) \left[ \frac{p(\omega)}{q(\omega)} \right]
\]

This equation reduces to

\[
\varepsilon_s - 1 = \left( \frac{c}{d} \right) \left[ \frac{p_{\text{area}}}{q_{\infty} - q_0} \right] \tag{3.35}
\]

or

\[
\left( \frac{d}{c} \right) = \left[ \frac{p_{\text{area}}}{(q_{\infty} - q_0)} \right] \left( \varepsilon_s - 1 \right) \tag{3.36}
\]

where \( p_{\text{area}} \) is the area under \( p(t) \) curve. The \( q_{\infty} \) and \( q_0 \) are the values of \( q(t) \) at \( t = \infty \) and \( t = 0 \) respectively. Figure 3.10 illustrates \( p_{\text{area}}, q_{\infty} \) and \( q_0 \). The values of \( p_{\text{area}}, q \) and \( q_0 \) can be determined experimentally by using liquids of known static permittivity \( \varepsilon_s \). Therefore for different known liquids, we can plot a graph of \( p_{\text{area}}/(q_{\infty} - q_0) \) verses \( (\varepsilon_s - 1) \) as shown in figure 3.10. The slope of the curve gives right hand side of equation (3.36). \( (d/c) = \text{slope} \).

Thus, effective pin length is given by \( d = c \times \text{slope} \).
3.8 Density

The density is defined as the property of a body, given by the ratio of its mass to its volume (kg/m³). The fluid density is a major key in the control of most industrial process, because it allows not only, a better management of the process but also the accurate determination of the quantity and quality of the product. The measurement of the density is used in drinks industry, to control the alcohol content in binary mixtures or sugar content in soft drinks and fruit juices. Density is widely used in the characterization of material, both pure and mixtures. The density of liquids is necessary to study the excess molar volume, viscosity, isentropic compressibility, specific acoustic impedance etc.

3.8.1 Experimental Method

Density can be determined using several measuring instruments, such as pycnometers, hydrometers and vibration-type density meters and also using the method of hydrostatic weighing. Recently the vibration type density meters have shown great versatility and are being used in various branches of industry. The advantages of using this instrument are its response time, its simplicity and the small volume of the (needed) sample.

**DENSITYMETER**

Density ($\rho$) of all samples, were determined by vibrating tube density meter (Anton Paar DMA 5000 system) having an accuracy (density) 0.000005 g/cm³. The temperature was kept constant to ± 0.001°C. The Repeatability, s.d. Density: 0.000001

![Figure 3.10: Determination of effective pin length](image-url)
g/cm$^3$. Temperature: 0.001$^\circ$C. The densitymeter was calibrated using double distilled water and air as references. It is having large measuring temperature range 0 to 90$^\circ$C. DMA 5000 requires a small amount of sample less than 1ml. Measuring time per sample is also very short i.e. less than 1 minute.

**Measuring principle**

The working principle of vibration type densitymeter is based on the law of harmonic oscillation, in which a U tube is completely filled with the sample to be analyzed, and subjected to an electromagnetic force. The measurement of the frequency and duration of vibration of the tube with the sample, allows the determination of the density value of the sample. This measuring principle is based on the Mass Spring Model.

The measuring cell consists of an oscillator formed by hallow U shaped borosilicate glass tube that comprises about 0.7ml of the sample. This tube has double walls and the space between them is filled with a gas with high coefficient of thermal conductivity. A platinum resistance thermometer is placed in that space which allows the temperature measurement of the fluid during the density measurement. The remaining instrumentation consists of system of electronic excitement and electrical components that provides a signal transmission of the period for the processor unit, free of interferences.

The U tube oscillates at its fundamental frequency, which is a function of the system mass. If we assume that the sample volume inside the cell is constant, it can be seen that the oscillation frequency is therefore a function of the sample density. The oscillation period $\tau$, is given by

$$\tau = \sqrt{\frac{\rho v + m}{C}}$$

(3.37)

Where $\rho$ is the density, $v$ the cell volume, $m$ the mass cell and $C$ the spring constant.

Applying the square of equation 3.37 and substituting $G = \frac{4\pi^2 v}{C}$ and $H = \frac{4\pi^2 m}{C}$ we obtain
\[ \rho = \frac{\tau^2 - H}{G} \]  

(3.38)

The internal software of the instrument includes the assumption that

\[ \rho = A\tau^2 - B \]  

(3.39)

Where \( \tau \) is period, \( \rho \) is density and A, B are coefficients. These coefficients are determined in the instrument calibration using two substances of known densities, typically air and distilled water.

Photograph of densitymeter is shown in fig. 3.12 (a), fig 3.12(b).

Photograph 3.12 (a) DMA 5000 Anton Paar densitymeter

Photograph 3.12 (b) DMA 5000 Anton Paar densitymeter
3.9 Refractive Index

The refractive index is an important optical constant used to determine purity of sample, molar refraction. The small change in molar refraction reflects the influence of molecular packing and molecular interaction [45, 84, 85, 86] on the polarizability of molecules.

3.9.1 Theory

The speed of light in a vacuum is always the same, but when light travel 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 (c) in a vacuum to the speed of light (v) in another substance is defined as the refractive index (n) of the substance.

\[
\text{Refractive index of substance } n = \frac{c}{v}
\]  

Whenever beam of light crosses a boundary from one medium into another its speed as well as direction of travel changes i.e. it is refracted (figure 3.12). (Except of the
light traveling perpendicular to the boundary of two medium there is no change in direction.) The relationship between speed light in the two mediums \((v_A \text{ and } v_B)\), the angles of incidence \((\theta_A)\), angle of refraction \((\theta_B)\) and the refractive indexes of the two mediums \((n_A \text{ and } n_B)\) is given as,

\[
\frac{v_A}{v_B} = \frac{\sin \theta_A}{\sin \theta_B} = \frac{n_B}{n_A}
\]  

\(3.41\)

Thus, it is possible to determine the refractive index of the sample quite accurately by measuring the angle of refraction and knowing the refractive index of the layer that is in contact with the sample, instead of measuring the speed of light in a sample. Nearly all refractometers utilize this principle, but may differ in their optical design.

### 3.9.2 Abbe’s Refractometer

Refractometer was first devised by German scientist Ernst Abbe. He was working for the Zeiss Company in Jena, Germany in the late 19th century. There are two detection systems for refractive index, transparent systems and reflection systems. Hand-held refractometer and Abbe refractometer use transparent detection systems and digital refractometer use reflection detection systems.

An Abbe refractometer is a bench-top refractometer with the highest precision of the different types of refractometer. In the Abbe’s refractometer, the liquid sample is sandwiched into a thin layer between an illuminating prism and a refracting prism (figure 3.13). The refracting prism is made by glass with a high refractive index (e.g., 1.75). It
should be noted that the refractive index of the liquid should be smaller than that of
refracting prism so as to allow for the critical angle phenomenon. A beam of light from
suitable source (sodium source) is reflected by mirror and passed 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 and scattered in liquid film at different angle of incidence.

Inspection of figure 3.13 shows that light traveling from point A to point B will have
the largest angle of incidence ($\theta_i$), hence the largest possible angle of refraction ($\theta_r$) for
that sample. All rays of light entering the refracting prism will have smaller $\theta_i$ and hence
lie to the left of point C. The rest of ray suffer the total internal reflection and will not
pass through refracting prism. Thus, when viewed through telescope, the field of view is
divided into two region light region to the left of point C and a dark region to the right.

Figure 3.13. Cross section part of the optical path of an Abbe refractometer.

Samples with different refractive indices will produce different angles of
refraction (see equation 3.41 above and recall that the angle of incidence and the
refractive index of the prism are fixed) 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. The refractive index varies significantly with wavelength
of light. Thus, for the most accurate measurement, it is necessary to use monochromatic
light. The most widely used wavelength of light for refractometry is the sodium D line at
589 nm.

They are based on the principle that as the density of a substance increases its
refractive index rises proportionately. 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 refractive index normally decreases as the temperature of liquid increases.

### 3.9.3 Experimental method

![Figure 3.14: Actual setup of Abbe’s Refractometer](image)

The photograph of actual used Abbe’s refractometer set up as shown in figure 3.14. The surface of the refractometer prism was cleaned using acetone and a lens wiper. This ensured that no stains or air bubbles were left on the prism surface. The lower prism was lifted and locked with the upper prism. Next, several drops of distilled water were placed on the prism surface using a syringe. Moreover, special care was taken to avoid water absorption in the chemicals, since this contamination has an important effect on refractive index measurements. The bottom-reflecting mirror was adjusted to get the maximum illumination through the telescope. The milled drum was rotated till sharp critical line (boundary line) coincides with the junction of the crossed graticule index line. Keeping the telescope in the above-mentioned position the corresponding reading of scale was taken against the index mark. This gives the refractive index of specimen within accuracy of ±0.001 in our experimental setup. The refractive index of the distilled water was then measured at 293 K is 1.333, which agrees well with the value reported in the literature [87-90].

Then measurement of refractive index of pure liquids and their binary mixtures at different temperature were taken. The measured values of refractive indices shows repeatability and accuracy compared to literature values.
3.10 Viscosity

Viscosity is an internal property of a fluid that offers resistance to flow. Viscosity is also defined as the measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress. It describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction.

Viscosity can be determined by using U-tube viscometers, Falling sphere viscometers Oscillating Piston Viscometer, Vibrational viscometers and Rotational viscometers. Among all the types of viscometer rotational viscometer is having utmost importance due to its accuracy and precision. Rotational viscometers use the idea that the torque required to turn an object in a fluid is a function of the viscosity of that fluid. They measure the torque required to rotate a disk or bob in a fluid at a known speed. 'Cup and bob', 'viscometers work by defining the exact volume of a sample which is to be sheared within a test cell; the torque required to achieve a certain rotational speed is measured and plotted. There are two classical geometries in "cup and bob" viscometers, known as either the "Couette" or "Searle" systems - distinguished by whether the cup or bob rotates. The rotating cup is preferred in some cases because it reduces the onset of Taylor vortices, but is more difficult to measure accurately.

'Cone and Plate' (rotational viscometer) viscometers use a cone of very shallow angle in bare contact with a flat plate. With this system the shear rate beneath the plate is constant to a modest degree of precision and deconvolution of a flow curve; a graph of shear stress (torque) against shear rate (angular velocity) yields the viscosity in a straightforward manner.

3.10.1 Experimental Details

Viscosity is measured by Brookfield CAP 2000+ Viscometer. Brookfield Cone and Plate (CAP) 2000+ is having a large range of viscosity 0.2–15,000 cP. It requires Small sample size, < 1 mL, simplifies testing. It is also having an integrated temperature control consisting of temperature range 5-75°C.
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