Chapter 3

Experimental
Section A

3.1 INTRODUCTION:

The dielectric constant is a property of major concern in understanding acid-base behaviour in various solvents. A “dielectric” is a substance that can sustain an electric field and acts as an insulator. Some liquids and gases can serve as good dielectric materials, having a special property of storing and dissipating electrical energy when subjected to electromagnetic fields. Dry air is an excellent dielectric. Dielectric measurements are useful for detecting explosives, plastic and metal weapons, drugs, chemical agents, and biological agents.

A new temperature dependant dielectric constant measurement technique for pure liquid and their binary mixture has been presented. The technique presented has very convincing advantages. The technique does not require post-manufacturing compensation of individual units under operating conditions, and no need of external components. Finally, the technique reported here is suitable for a very wide dielectric constant range, is very effective, and gives reasonable results. These features make this technique very attractive. The thesis provides both the definitions and the mathematical theory on which the method is based.

The idea has been demonstrated with a practical circuit that incorporates a new integrated circuit.

Principle:

In the present study, the technique utilizes pulse width measurement for determination of dielectric constant using the TDS2024 Digital Storage Oscilloscope as a tool, while Monostable multivibrator (IC 74121), which is triggered by Astable multivibrator is designed for generation of pulse width and external cylindrical capacitor used as a sample cell.

3.2 Definition

A one-shot multivibrator integrated circuit is a device that, when triggered, produces an output pulse width that is independent of the input pulse width, and can be programmed by an external Resistor-Capacitor (RC) network. The output pulse width will be a function of the RC time constant. There are various one-shots manufactured by Fairchild Semiconductor that have diverse features, although, all one-shots have the basic property of producing a programmable output pulse width.
All Fairchild one-shots have True and Complementary output, and both positive and negative edge-triggered inputs.

3.2.1 Monostable Multivibrator (74121):

Shown below is the pin out diagram for the IC 74121. There are three trigger inputs for the device, two negative edge-triggering (A) inputs, one positive edge Schmitt-triggering (B) input. If $A_1$ or $A_2$ is LOW and B goes HIGH, or if B is HIGH and $A_1$ or $A_2$ go LOW, the one-shot will fire.

![Pin Out Diagram](image1)

3.2.2 Output Pulse Width vs. Timing Components (DM74121):

Timing equations hold for all combinations of $R_{\text{EXT}}$ and $C_{\text{EXT}}$ for all cases of $C_{\text{EXT}} > 1000$ pF. For cases where $C_{\text{EXT}} < 1000$ pF, use the graphs shown below.

![Graph](image2)

Figure 3.2: Variation of pulse width vs. timing components
3.2.3 Connection Diagrams of 74121:

![Connection Diagram](image)

**Figure 3.3: Connection Diagram Top View**

3.2.4 Functional Description:

The basic output pulse width is determined by selection of an internal resistor $R_{\text{INT}}$ and capacitor ($C_x$). When monostable multivibrator is triggered then the output pulse width remain independent of further transitions of the inputs and function of the timing components. The pulse width can vary from a few nano-seconds to 28 seconds by choosing appropriate $R_x$ and $C_x$ combinations. There are three triggered inputs from the device, two negative edge-triggering (A) inputs, one positive edge Schmitt-triggering (B) input. The working of the device can be summaries from the given truth table.
Table 3.1: Truth Table of 74121:

<table>
<thead>
<tr>
<th>Inputs</th>
<th>Outputs</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>A2</td>
</tr>
<tr>
<td>L</td>
<td>X</td>
</tr>
<tr>
<td>X</td>
<td>L</td>
</tr>
<tr>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>H</td>
<td>H</td>
</tr>
<tr>
<td>H</td>
<td>$\downarrow$</td>
</tr>
<tr>
<td>$\uparrow$</td>
<td>H</td>
</tr>
<tr>
<td>$\uparrow$</td>
<td>$\downarrow$</td>
</tr>
<tr>
<td>L</td>
<td>X</td>
</tr>
<tr>
<td>X</td>
<td>L</td>
</tr>
</tbody>
</table>

3.3 Theory related to the estimation of Static dielectric constant using Monostable multivibrator:

Monostable multivibrator is an electronic device used to get an output pulse of desire interval, and the output pulse duration is depend on resistor and capacitor used in the timing branch (charging path) of the circuit. For IC-74121 version Monostable multivibrator, the expression for output pulse is as follows,

$$T = 0.33 \times R \times C$$

(1)

where, equation (1) is an equation for output pulse width. From this equation it is clear that interval of output pulse depends on values of resistor and capacitor used in
the timing circuit. Usually values of R and C are remain constant in Multivibrator circuit, where as if R having fixed value, and value of C depends on various dielectric media placed in it, then the expression for T can be written as,

\[ T = \text{Constant} \times C \quad (2) \]

where, Constant = 0.33 R and we can write,

\[ T \propto C \quad (3) \]

For cylindrical capacitor,

\[ C = \frac{2\pi \varepsilon_r \varepsilon_0 L}{\ln(b/a)} \quad (4) \]

where, L is the capacitor length, a is the inner electrode radius, b is the outer electrode radius, \( \varepsilon_r \) is the relative permittivity of dielectric medium and \( \varepsilon_0 \) is the absolute permittivity of free space. From equation (4) it is clear that if the physical dimensions of capacitor remain fixed then value of capacity of the capacitor for given dimensions will be directly proportional to the dielectric constant of the liquid used in the cylindrical capacitor.

Thus we can have,

\[ T \propto \varepsilon_r \quad (5) \]

From equation (3) and (5), we can write,

\[ T \propto \varepsilon_r \quad (6) \]

Let \( T_1 \) be the pulse width of Monostable multivibrator without liquid. Therefore,

\[ T_1 = \frac{2\lambda \pi \varepsilon_0 L}{\ln(b/a)} \quad (7) \]

Where, \( \lambda \) is proportional constant and for air \( \varepsilon_r = 1 \)

\( T_2 \) is the pulse width of Monostable multivibrator with liquid. Therefore,

\[ T_2 = \frac{2\lambda \pi \varepsilon_r \varepsilon_0 L}{\ln(b/a)} \quad (8) \]
Subtracting equation (7) from (8), we get

\[ T_2 - T_1 = A(\varepsilon_r - 1) \]  

(9)

where; \( A = \frac{2\lambda \pi \varepsilon_0 L}{\ln(b/a)} \) called sample holding device constant (SHD) to be determined experimentally over large range for the dielectric constants which has the dimension of time. Hence knowing SHD, and shift in pulse width for unknown sample the dielectric constant in principle can be easily determined.

From equation (9), we get

\[ A = \frac{T_2 - T_1}{\varepsilon_r - 1} \]

or

\[ \varepsilon_r = \frac{T_2 - T_1}{A} + 1 \]  

(10)

where, \( T_1 \) is the pulse width of Monostable multivibrator without liquid and \( T_2 \) is the pulse width of Monostable multivibrator with liquid

From equation (10) it is clear that the permittivity of dielectric medium can be easily estimated in terms of pulse width.

3.4 Dielectric constant measurements:

![Block diagram of experimental setup of Dielectric constant measurement](image)

Figure 3.4. Block diagram of experimental setup of Dielectric constant measurement.

**Experimental setup:**

The experimental setup for static dielectric constant measurement consists of various devices:

- DC regulated power supply
- Astable multivibrator
- Monostable multivibrator
- Sample cell
- Digital storage oscilloscope

Figure 3.4 shows the block diagram of the experimental setup used for the measurement of dielectric constant.

Power supply provides constant voltage to astable and monostable multivibrator for its proper operation. In this case astable multivibrator plays a role of triggering source for monostable multivibrator. Monostable multivibrator generates the pulse, and width of the pulse is determined by internal resistor and external capacitor i.e. sample cell. Initially measurement of pulse width, it is necessary to warm up the device near about 20-25 minutes for its proper operation. The change in pulse width is depends on value of external cylindrical capacitor used in the timing component, and it is the characteristic of the sample inside the cell. We have measured pulse width without sample and pulse width with sample with the help of TDS2024 digital storage oscilloscope. The difference between it, used for the calculation of the dielectric constant of the sample at the corresponding temperature which is determined by using the equation10.
Figure 3.5: Actual photograph of Experimental setup

Above photograph is the actual experimental set up for the dielectric constant measurement.
Experimental setup of static dielectric constant is calibrated by number of liquids. For different liquids, first measure the pulse width without liquid and then with liquid sample. We know the literature value of dielectric constant of various standard liquids at 25°C, by putting the values of it in equation 10, the device constant ‘A’ can be determined. Once ‘A’ is determined it is used for the estimation of dielectric constant of different liquids. In this way the device calibrated and used for the determination of dielectric constant of liquids. Experimental values obtained from the present setup and literature values are shown in give table 3.2.
Table 3.2: Experimental and literature values of dielectric constant:

<table>
<thead>
<tr>
<th>Name of liquids</th>
<th>Pulse width without liquids (ns)</th>
<th>Pulse width with liquids (ns)</th>
<th>Difference of Pulse width (ns)</th>
<th>Lit. values of $\varepsilon_a$</th>
<th>Cell Constant (A) ns</th>
<th>Observed value of $\varepsilon_a$</th>
<th>% error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heptane</td>
<td>48</td>
<td>48.6</td>
<td>0.6</td>
<td>1.92*</td>
<td>0.65</td>
<td>1.94</td>
<td>0.91</td>
</tr>
<tr>
<td>Dipentyl ether</td>
<td>48</td>
<td>49.1</td>
<td>1.1</td>
<td>2.798*</td>
<td>0.61</td>
<td>2.72</td>
<td>-2.83</td>
</tr>
<tr>
<td>Bromobenzene</td>
<td>48</td>
<td>50.8</td>
<td>2.8</td>
<td>5.4*</td>
<td>0.64</td>
<td>5.38</td>
<td>-0.46</td>
</tr>
<tr>
<td>2-Butoxyethanol</td>
<td>48</td>
<td>52.7</td>
<td>4.7</td>
<td>-</td>
<td>-</td>
<td>8.34</td>
<td>-</td>
</tr>
<tr>
<td>2-Ethoxyethanol</td>
<td>48</td>
<td>55.45</td>
<td>7.45</td>
<td>-</td>
<td>-</td>
<td>12.64</td>
<td>-</td>
</tr>
<tr>
<td>1-Hexanol</td>
<td>48</td>
<td>55.7</td>
<td>7.7</td>
<td>13.3*</td>
<td>0.63</td>
<td>13.03</td>
<td>-2.02</td>
</tr>
<tr>
<td>2-Methoxyethanol</td>
<td>48</td>
<td>58.2</td>
<td>10.2</td>
<td>-</td>
<td>-</td>
<td>16.94</td>
<td>-</td>
</tr>
<tr>
<td>1-Butanol</td>
<td>48</td>
<td>58.4</td>
<td>10.4</td>
<td>17.1*</td>
<td>0.65</td>
<td>17.25</td>
<td>0.88</td>
</tr>
<tr>
<td>2-Propanol</td>
<td>48</td>
<td>58.9</td>
<td>10.9</td>
<td>18.3*</td>
<td>0.63</td>
<td>18.03</td>
<td>-1.47</td>
</tr>
<tr>
<td>1-Propanol</td>
<td>48</td>
<td>60.2</td>
<td>12.2</td>
<td>20.1*</td>
<td>0.64</td>
<td>20.06</td>
<td>-0.19</td>
</tr>
<tr>
<td>Acetone</td>
<td>48</td>
<td>60.3</td>
<td>12.3</td>
<td>20.7*</td>
<td>0.62</td>
<td>20.22</td>
<td>-2.32</td>
</tr>
<tr>
<td>Ethanol</td>
<td>48</td>
<td>62.9</td>
<td>14.9</td>
<td>24.3*</td>
<td>0.64</td>
<td>24.28</td>
<td>-0.08</td>
</tr>
<tr>
<td>Ethanol amine</td>
<td>48</td>
<td>64</td>
<td>16</td>
<td>-</td>
<td>-</td>
<td>26.00</td>
<td>-</td>
</tr>
<tr>
<td>Methanol</td>
<td>48</td>
<td>68</td>
<td>20</td>
<td>32.63*</td>
<td>0.63</td>
<td>32.25</td>
<td>-1.16</td>
</tr>
<tr>
<td>Nitrobenzene</td>
<td>48</td>
<td>69.5</td>
<td>21.5</td>
<td>34.8*</td>
<td>0.64</td>
<td>34.59</td>
<td>-0.59</td>
</tr>
<tr>
<td>N,N-Dimethylformamide</td>
<td>48</td>
<td>71.5</td>
<td>23.5</td>
<td>37.06*</td>
<td>0.65</td>
<td>37.72</td>
<td>1.78</td>
</tr>
<tr>
<td>Ethylene glycol</td>
<td>48</td>
<td>72.8</td>
<td>24.8</td>
<td>-</td>
<td>-</td>
<td>39.75</td>
<td>-</td>
</tr>
</tbody>
</table>

* Hand Book of chemistry and physics, 64\textsuperscript{th} ed., CRC Press Florida 1993.
Digest of Literature on dielectrics VOL.40 1976, national academy of science
The graph of measured pulse widths versus literature values of dielectric constant is shown below.

Figure 3.6: Variation of dielectric constant with pulse width difference
The spectra of measured pulse widths with and without sample for 2-Butoxyethanol and N, N- Dimethylformamide is shown in the figure.
3.5 Digital storage oscilloscope: TDS2024 digital storage oscilloscope is used in this experimental setup, because of its excellent features.

Features:
- 60 MHz, 100 MHz and 200 MHz Bandwidths
- Sample Rates up to 2 GS/s
- 2 or 4 channels
- 2.5 k Points Record Length
- Color or Monochrome LCD Display
- Auto-set Menu with Waveform Selection
- Probe Check Wizard to Ensure Correct Probe Usage
- Advanced Triggering
- 11 Automatic Measurements
- Multi-language User Interface
- Waveform and Setup Memories
- Optional RS232, GPIB and Centronics Printer Interfaces with TDS2CMAX Module
- Optional Compact Flash Memory Storage, RS232 and Centronics Printer Interfaces with TDS2MEM Module.

The TDS1000 and TDS2000 Series digital storage oscilloscopes deliver an unbeatable combination of superior performance, unmatched ease-of-use, and affordability in an ultra lightweight, portable package. These new products extend the performance and ease-of-use features in the former TDS200 Series, the benchmark for low-cost oscilloscopes. A TDS2024 digital storage oscilloscope is shown in figure 3.7.
3.5.1 Digital performance:

With up to 200 MHz bandwidth and 2 GS/s maximum sample rate, no other color digital storage oscilloscope offers as much bandwidth and sample rate for the price. The TDS1000 and TDS2000 Series oscilloscopes provide accurate real-time acquisition up to their full bandwidth. These instruments offer advanced triggering, such as pulse width triggering and line-selectable video triggering, and 11 standard automatic measurements on all models. The Fast Fourier Transform (FFT) math function allows the user to analyze, characterize and troubleshoot circuits by viewing frequency and signal strength (standard).

3.5.2 Setup and use:

The simple user interface with classic, analog-style controls makes these instruments easy to use, reducing learning time and increasing efficiency. Innovative features such as the auto set menu, probe check wizard, context-sensitive help menu and color LCD display (TDS2000 Series) optimize instrument setup and operation.
Open choice solutions deliver simple, seamless integration between the oscilloscope and the personal computer, providing you with multiple choices to easily document and analyze your measurement results. Choose from optional communication modules, Compact flash mass storage capability, Open choice software or integration with third-party software.

3.6 Power Supply:

The 78xx family is a very popular choice for many electronic circuits which require a regulated power supply, due to their ease of use and relative cheapness. When specifying individual ICs within this family, the xx is replaced with a two-digit number, which indicates the output voltage. The particular device is designed to provide (for example, the 7805 has a 5 volt output, while the 7812 produces 12 volts). The +5 volt supply is useful for both analog and digital circuits. DTL, TTL, and CMOS ICs will all operate nicely from a +5 volt supply.

3.6.1 Schematic Diagram of power supply:

The +5 volt power supply is based on the commercial 7805 voltage regulator IC. This IC contains all the circuitry needed to accept any input voltage from 8 to 18 volts and produce a steady +5 volt output, accurate to within 5% (0.25 volt). It also contains current-limiting circuitry and thermal overload protection, so that the IC won't be damaged in case of excessive load current; it will reduce its output voltage instead.

The 1000µf capacitor serves as a "reservoir" which maintains a reasonable input voltage to the 7805 throughout the entire cycle of the ac line voltage. The two rectifier diodes keep recharging the reservoir capacitor on alternate half-cycles of the line.
voltage, and the capacitor is quite capable of sustaining any reasonable load in between charging pulses.

The 10µf and 0.01µf capacitors serve to help keep the power supply output voltage constant when load conditions change. The electrolytic capacitor smooths out any long-term or low frequency variations. Therefore, the 0.01µf is included to bypass high-frequency changes, such as digital IC switching effects, to ground.

3.6.2 Advantages:

The 78xx series has several key advantages over many other voltage regulator circuits

- 78xx series ICs do not require any additional components to provide a constant, regulated source of power, making them easy to use, as well as economical, and also efficient uses of circuit board real estate. By contrast, most other voltage regulators require several additional components to set the output voltage level, or to assist in the regulation process.

- 78xx series ICs have built-in protection against a circuit drawing too much power. They also have protection against overheating and short-circuits, making them quite robust in most applications. In some cases, the current-limiting features of the 78xx devices can provide protection not only for the 78xx itself, but also for other parts of the circuit it is used in, preventing other components from being damaged as well.
3.7 Temperature controller: Temperature dependent dielectric measurements can be done using temperature bath shown in figure 3.9. The temperature of sample was maintained at desired value, within accuracy limit of ±1°C, by circulating constant temperature water through heat insulating jacket surrounding sample cell.
Section B

3.8 Density Measurement:

The density of a material is defined as its mass per unit volume. In some countries, density is also defined as its weight per unit volume. It is a physical property of matter, as each element and compound has a unique density associated with it. Density defined in a qualitative manner as the measure of the relative "heaviness" of objects with a constant volume. The mass is normally measured with an appropriate balance; the volume may be measured directly (from the geometry of the object) or by the displacement of a fluid. Hydrostatic weighing is a method that combines these two. Density may also refer to how closely "packed" or "crowded" the material appears to be. Usually the density is expressed in grams per ml or cc.

Measuring Principle:

Densities of pure liquids and their binary mixtures were measured with a 3 ml volume pyknometer (Density bottle), for each temperature, mass of the liquid was kept constant. Mass is recorded with mechanical balance with an accuracy of 0.00001gm and the densities were calculated using the relation of mass and volume. Double distilled water was used for the calibration volume of the pycknometer. Pycknometer which is used for the measurement of the density is shown in figure 3.8 given below. Temperature was maintained constant by circulating water through thermostat water bath, maintaining constant temperature (± 0.10 C). The volume (V) of pyknometer to the mark in the capillary is determined at 298.15 K, by using measured mass (M) of water and literature value of density (ρ) at 298.15 K, by using equation of density of liquids in terms of mass and volume given by

\[
\rho = \frac{M}{V}
\]
Again by considering the molecule as sphere and volume it is

\[ V = \pi r^2 h \]

where, ‘h’ is height of the liquid and ‘r’ is radius of capillary of pyknometer.

By recording change in height of expanded liquid in capillary of pyknometer at different temperature density was determined, keeping mass of liquid constant. And change in height is measured using travelling microscope which has least count 0.001 cm. Mass of the liquids is measured by using the single pan balance with an accuracy of 0.00001 gm. The densities of binary mixtures were determined by taking mass at 288.15 K, using mass and volume (V) of the pyknometer as determined above at 298.15 K, increase in volume of liquids for next temperatures can be determined as mentioned above.

Percentage error in the measurement of density is calculated by using the relation

\[ \% \text{ error} = \left( \frac{\text{observed} - \text{literature}}{\text{literature}} \right) \times 100 \]

We have compared of density of some pure liquids at 298.15 K with literature value which gives accuracy of the present work.
3.9 Refractive Index Measurement:

Dispersion forces are caused by induced dipoles formed as a result of oscillations of the electron clouds about the core of the molecules. Refractive index measurements furnish information about these interactions (1–3) since Refractive index at optical wavelengths is related to the mean polarizability $P$.

Measurement Principle:

Above figure shows the actual photograph of Abbe’s refractometer. The refractive index ($n_D$) of all samples was obtained using Abbe’s refractometer with a sodium D line. The refractometer was initially calibrated with distilled water. The surface of the refractometer prism was cleaned using ethanol and a lens wiper. This ensured that no stains or air bubbles were left on the prism surface. Next, several drops of distilled water were placed on the prism surface using a plastic syringe and were covered with a cap. Moreover, special care was taken to avoid water absorption in the chemicals, since this contamination has an important effect on refractive index measurements. The refractive index of the distilled water was then measured at 293 K and was set at 1.333, which agrees well with the value reported in the literature [4-6].
Then measurements refractive index of other pure and their binary mixtures at different temperature were taken. The measured values of refractive indices shows admirable repeatability and accuracy compared to literature values.
References:


