Chapter II

Experimental Techniques for Determination of Physical Properties and Thermodynamic Parameters

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2.1 Introduction

Coulomb stated that the force $F$ between changes $q_1$ and $q_2$ separated by a distance $d$ in a medium is given by $F=q_1q_2/4\pi r^2$ where the constant of the medium, that measures efficiency of transfer of electric charge, is known as dielectric constant of the medium. Later on, Maxwell, in order to study propagation of electromagnetic waves in the medium, introduced dielectric constant in his famous equation as

$$\nabla \times H = J + \frac{\partial D}{\partial t} \quad \text{Where } D = E$$

The magneto motive force ($\nabla \times H$) around a closed path is equal to the sum of conduction current ($J = \sigma E$) and the displacement current $\partial D/\partial t; D$ being electric displacement in the medium, is a function of dielectric properties through any surface bounded by the path. Further, Maxwell showed that the velocity $c$ of an EM wave in a given medium, is a function of medium parameters as $c = 1/\sqrt{\mu \varepsilon}$, where $\mu$ is permeability and $\varepsilon$ is the permittivity of the medium.

Taking time-dependence of the fields as $e^{jwt}$, we have

$$\nabla \times H = \sigma E + jw\varepsilon E$$

or

$$\nabla \times H = j\omega \left( \varepsilon - \frac{j\sigma}{\omega} \right) E = j\omega \varepsilon^* E$$

(2.1)

Where

$$\varepsilon^* = \left( \varepsilon - \frac{j\sigma}{\omega} \right)$$

(2.2)

Is known as complex dielectric constant. Let $\varepsilon_0$ be the dielectric constant of vacuum (free space) which has a value $(36\pi \times 10^9)^{-1}$ farad/meter. Dividing Eq. (2.2) by $\varepsilon_0$ we have

$$\frac{\varepsilon^*}{\varepsilon_0} = \varepsilon_r = \left( \frac{\varepsilon}{\varepsilon_0} - \frac{j\sigma}{\omega \varepsilon_0} \right) = \varepsilon' - \varepsilon''$$

(2.3)

where $\varepsilon_r$, the ratio of the complex dielectric constant to the dielectric constant of vacuum, is known as relative or normalized complex dielectric constant Further, separating Eq. (2.3) in real and imaginary parts we find that real part $\varepsilon' = \text{Re} (\varepsilon^*/\varepsilon_0)$ is associated with the ability of the dielectric substance to store electric energy, while the imaginary part $\varepsilon'' = -\text{Im} (\varepsilon^*/\varepsilon_0)$ is associated with the losses occurring in the dielectric. Also rewriting Eq. (2.3) as

$$\varepsilon_r = \varepsilon(1 + \varepsilon''/\varepsilon') = \varepsilon'(1 + \tan \delta)$$

(2.4)

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tanδ which represents the ratio of power lost in the dielectric ($\varepsilon''$) to the energy stored per cycle ($\varepsilon'$) in the dielectric is known as loss tangent. Dielectric constant depends upon frequency but its variation is quite gradual and may be taken to be constant over a narrow band of frequencies. The variation of tanδ with frequency is more pronounced than that of $\varepsilon'$. The dielectric constant is also a function of temperature and humidity so these parameters should be kept constant during measurement and has to be recorded along with the result.

The advantage of the methods employing wave-guides is that, the radiation is enclosed and there is no loss of energy outside the system. The experimental method [1, 4, and 5] for the measurements of complex permittivity of liquids as a function of frequency depends on the frequency range as well as the type of system under consideration. A convenient method of measuring the complex permittivity of liquids is electromagnetic standing wave within the test liquid or in front of the liquid. When the sample extends over an appreciable part of a wavelength, the electric and magnetic fields vary over the sample dimensions. In such cases an analysis based on Maxwell’s equations of the waves traveling along a transmission line, containing the sample is required. Apparatus employing co-axial line may be used over a frequency range of some MHz to 5 GHz. Hallow pipe (wave-guide) is useful over the range 2 to 60 GHz. The experimental determination of dielectric permittivity and loss rests on the observation of the waves transmitted through or reflected from length “L” of dielectric. The procedure followed depends mainly on whether the sample is liquid or solid and on whether the dielectric loss is large or small.

If solids are to be measured then solids may be machined or dip-pressed to fit the wave-guide and some adjustment of length is necessary. But measurements as a function of length in not practicable solids have the real advantage that, a window is not required for sample holder and the disadvantage is that they may not fit the sample holder snugly.

Recent improvements like sampling of the standing wave pattern at regular intervals and automated data processing improves the experimental sensitivity. In our work we have employed X-band microwave bench to measure dielectric constant and loss factor. In this method the position of the shorting plunger inside the cell is moved up or down slowly with the help of micrometer screw gauge. When the plunger moves up or down slowly in steps, the length of the liquid column in the cell changes
in steps as well as the corresponding output from the crystal detector is recorded. The data obtained is used to determine the values of propagation constants in the liquid. The values of $\varepsilon'$ and $\varepsilon''$ can be obtained from these propagation constants.

2.2 Dielectric Parameters from VSWR Measurements

Roberts and Von Hippel [13, 14] worked out a method of determining permittivity from the measurements of the parameters of the standing wave. The method consists, reflection of microwaves at normal incidence in transverse electric from a dielectric medium placed against a perfectly reflecting surface. The reflection sets up standing waves in the space in front of the sample. The separation of the first minimum from the face of the sample will depend upon wavelength of electromagnetic waves in the sample and on sample dimensions; hence on dielectric constant. So, and in turn a change in half power width of the standing wave form. Also losses in the dielectric shall decreases VSWR and $\tan \delta$ is related to these decreases in VSWR.

To get the expression for dielectric constant $(\varepsilon')$ and dielectric loss $(\varepsilon'')$, consider an E.M. wave traveling through air medium and strikes normally to the dielectric medium. A part of it is reflected and the rest of the part gets transmitted. A standing wave pattern is thus produced in air medium, as shown in Figure. 2.1 below. The transverse electric field component in this partial reflection case is given by [15].

![Diagram](image.png)
Figure 2.1: Standing waves in a wave guide (a) without dielectric (b) Loaded with dielectric of any length (c) Loaded with dielectric of length $\lambda_d/2$. $\lambda_d$ is wavelength of microwaves in the dielectric.

$$E_y = [E_0 \exp(j\omega t - \gamma_1 x)] \left[1 + \Gamma_0 \exp(2\gamma_1 x)\right]$$

(2.5)

Where $\gamma_1$ is propagation constant in air medium and is the sum of attenuation ($\alpha_i$) and phase shift constant ($\beta_i$). That is $\gamma_1 = \alpha_i + j\beta_i$.

The reflection co-efficient $r_0$ is given by

$$\Gamma_0 = |\Gamma_0| \exp(-2j\psi)$$

(2.6)

Where $\psi$ is the phase of the reflection coefficient. Thus input impedance $Z_0$ at the boundary is given by

$$Z_0 = Z_1 \frac{1 + \Gamma_0}{1 - \Gamma_0}$$

(2.7)

$Z_1$ is the impedance of air medium.

If the attenuation in air medium be negligible, then the inverse voltage standing wave ratio in air medium $\rho = \left(\frac{E_{\text{max}}}{E_{\text{min}}}\right)^{-1}$ is written as

$$\rho = \left(\frac{E_{\text{min}}}{E_{\text{max}}}\right) = \left(\frac{1 - \Gamma_0}{1 + \Gamma_0}\right)$$

(2.8)

Using $\Gamma_0 = |\Gamma_0| \exp(-2j\phi)$ and $\Phi = \rho_1 + j\phi$, equation (2.3) becomes

$$Z_0 = Z_1 \frac{1 + \Gamma_0}{1 - \Gamma_0} = Z_1 \coth \phi$$

(2.9)
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\[ \rho = \frac{E_{\text{in}}}{E_{\text{in}}} = \frac{1 - \exp(-2\rho_1)}{1 + \exp(-2\rho_1)} = \tan h \rho_1 \]

(2.10)

If \( x_0 \) be the position of the first minimum where incident and reflected waves combine out of 180° phase then

\[ \frac{2\pi x_0}{\lambda_g} = (2\pi) \left[ \frac{1}{4} - \left( \frac{x_0}{\lambda_g} \right) \right] \]

(2.11)

\( \lambda_g \) is the guide wavelength, using equations (2.7) and (2.11) we have,

\[ Z_0 = Z_1 \left( \frac{\tanh \rho_1 - j \cot \psi}{1 - j \tanh \rho_1 \cot \psi} \right) \]

(2.12)

Using equation 2.10 and 2.12 we get

\[ Z_0 = Z_1 \left( \frac{\rho_1 - j \tan \frac{2\pi x_0}{\lambda_g}}{1 - j \rho \tan \frac{2\pi x_0}{\lambda_g}} \right) \]

(2.13)

If the dielectric medium is terminated in a short circuit, the input impedance at the interface.

\[ Z_{(0)s} = Z_2 \tan h \gamma_z d \]

(2.14)

Where \( Z_2, \gamma_z \) and \( d \) are the characteristic impedance, propagation constant and length of dielectric medium respectively. Equating the two impedance at the interface \( (x = 0) \), we find \( Z_1 \) is given by

\[ Z_1 \left( \frac{\rho_1 - j \tan \frac{2\pi x_0}{\lambda_g}}{1 - j \rho \tan \frac{2\pi x_0}{\lambda_g}} \right) = Z_2 \tan h \gamma_z d \]

(2.15)

Since \( \frac{Z_1}{Z_2} = \frac{\gamma_z}{\gamma_1} \) so we have,
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\[
\frac{(\tanh \gamma d)}{(\gamma d)} = \frac{1}{\gamma_1} \begin{pmatrix}
\frac{p - j \tan \frac{2\pi x_0}{\lambda_e}}{1 - j \rho \tan \frac{2\pi x_0}{\lambda_g}}
\end{pmatrix}
\] (2.16)

\[
\frac{(\tan h \gamma d)}{(\gamma d)} = \begin{pmatrix}
\frac{\rho - j \frac{2\pi x_0}{\lambda_e}}{1 - j \rho \frac{2\pi x_0}{\lambda_g}}
\end{pmatrix}
\] (2.17)

Now characteristic propagation constant \( \gamma_2 \) of E.M. waves in dielectrics is related to intrinsic propagation constant of the waves in air medium as

\[
\gamma_2^2 = \gamma_1^2 + \left( \frac{m\pi}{a} \right)^2 + \left( \frac{n\pi}{b} \right)^2
\] (2.18)

and for TE\(_{10}\) mode as

\[
\gamma_2^2 = \gamma_1^2 + \left( \frac{\pi}{a} \right)^2
\] (2.19)

\( a \) and \( b \) are broad and narrow dimensions of the waveguide.

The complex dielectric constant of dielectric medium is given by

\[
\varepsilon = \left( \frac{-\gamma_1^2}{\omega^2 \mu_0} \right) = \varepsilon_0 \left[ \left( \frac{1}{\lambda_e} \right)^2 - \left( \frac{\gamma_2}{2\pi} \right)^2 \right] - \left( \frac{1}{\lambda_s} \right)^2 + \left( \frac{1}{\lambda_s} \right)^2
\] (2.20)

Where \( \lambda_e = 2a \) cut off wavelength \( \gamma_1 \) is propagation constant in the dielectric and \( \lambda_s \) is guide wavelength.

Knowing \( \lambda_e, \varepsilon_0, \gamma_2 \) and \( \lambda_s \varepsilon \) can be calculated.

Separating \( \varepsilon \) in to imaginary and real parts one can obtained the value of \( \varepsilon' \) and \( \varepsilon'' \) and loss tangent \( \tan \delta \).

The measurement of \( \gamma_2 \) is effected by using equation 2.16. The values of \( \rho = \frac{1}{VSWR} \), \( \lambda_0 \) and \( \lambda_s \) can be experimentally measure to give a complex values of
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\[ \frac{\tan h \gamma_2 d}{\gamma_2 d} \]

To find \( \gamma_2 \) from this values put \( \gamma_2 d = Te^{i\tau} \), then

\[ \frac{\tan h T e^{i\tau}}{Te^{i\tau}} = Ce^{i\xi} \]  

(2.21)

Knowing C and \( \xi \) values of T and \( \tau \) are seen from the standard table and hence \( \gamma_2 \) is determined.

The calculation part is simplified, if we take the length of dielectric sample in a multiple of quarter or half wavelength of E.M waves in dielectrics. Thus if the short circuiting plunger is placed at a distance of \( \frac{\lambda_d}{4} \) from dielectric medium, it corresponds to an open circuit termination and consequently the input impedance is given by

\[ Z_{(0)\infty} = Z_0 \text{Coth} \gamma_2 d \]  

(2.22)

The propagation constant along the axis of a hollow wave-guide of uniform cross section and highly conducting walls is given by

\[ \gamma_2 = \left( \frac{2\pi j}{\lambda_0} \right) \left[ \epsilon' \left( \frac{\lambda_0}{\lambda_d} \right)^2 - \omega j \right]^{1/2} \]  

(2.23)

or

\[ \gamma_2 = \alpha_d + j \left( \frac{2\pi}{\lambda_d} \right) \]

\( \alpha_d \) is the attenuation in the dielectric while \( \lambda_d \) is the wavelength of electromagnetic wave in dielectric. Separating equation (2.23) in to real and imaginary parts we get

\[ \epsilon' = \left( \frac{\lambda_0}{\lambda_d} \right)^2 + \left( \frac{\lambda_0}{\lambda_d} \right)^2 \left[ 1 + \frac{\alpha_d \lambda_d}{2\pi} \right] \]  

(2.24)

and

\[ \epsilon'' = \left( \frac{1}{\pi} \left( \frac{\lambda_0}{\lambda_d} \right)^2 \right) \alpha_d \lambda_d \]  

(2.25)

Since \( \lambda_0, \lambda_c \) and \( \lambda_d \) are known \( \alpha_d, \epsilon', \epsilon'' \) and \( \tan \delta \) can be computed.

2.3 Heston and Poly’s Method

i) Low loss liquids

For low loss liquids, the standing wave method is advantageous. W.M. Heston
et.al [16] has considered the case of low loss liquids. That is dilute solution of polar solute in non-polar solvents. For short circuit termination they used a liquid length in the steps of \( \left( \frac{n\lambda_d}{2} \right) \) and measured VSWR. Thus for liquid sample of length equal to \( \left( \frac{n\lambda_d}{2} \right) \), placed in contact with short circuit, then the value of \( x \), the distance of first minima for the interface is zero. Hence equation (2.13) gives,

\[
Z_{(0)sc} = \rho_n \quad \therefore Z_i = 1
\]

\( \rho_n \) denotes inverse VSWR when liquid length is \( \left( \frac{n\lambda_d}{2} \right) \), n being integer.

Again from equation (2.14), we get

\[
Z_{(0)sc} = \rho_n = Z_{\gamma_2} \tanh \gamma_2 d
\]

Since \( \gamma_2 = (\alpha_d + j\beta_d) ; d = \left( \frac{n\lambda_d}{2} \right) \)

\[
\rho_n = Z_{\gamma_2} + \frac{mh}{2} \left( \frac{n\alpha_d\lambda_d + 2j\pi}{2} \right)
\]

\[
\rho_n = Z_{\gamma_2} \tanh \left( \frac{n\alpha_d\lambda_d}{2} \right)
\]

For low loss substance, \( \tanh x \approx x \), and \( Z_{\gamma_2} = \left( \frac{\lambda_i}{\lambda_g} \right) \) therefore we have,

\[
\rho_n = Z_{\gamma_2} \left( \frac{n\alpha_d\lambda_d}{2} \right)
\]

\[
\rho_n = \frac{n\alpha_d\lambda_d}{2\lambda_g} \quad (2.26)
\]

\( \alpha_d \) is the sum of two parts. One due to dielectric attenuation and the other is due to plunger resistance and other losses. To eliminate latter losses, a graph is plotted between several mean values of \( \rho_n \) and \( n \). The slope of this graph gives the value of \( \alpha_d \).

\[
\alpha_d = \frac{\left( \frac{2\lambda_g}{\lambda_i} \right) \left( d \rho_{mean} \right)}{(dn)} \quad (2.27)
\]

The values of the desired parameters are then found from the relation given below.
ii) High loss liquids

Poly’s considered the case of high loss liquids [15] the method requires the
determination of standing wave ratios using slotted line for various liquid lengths,
which are integral multiples of $\left(\frac{\lambda_d}{2}\right)$.

If $\rho_m, \rho_n$ and $\rho_\infty$ be the inverse voltage standing wave ratios for liquid length
equal to $\left(\frac{m\lambda_d}{2}\right)$, $\left(\frac{n\lambda_d}{2}\right)$ and infinite length respectively, then to a very close
approximation one can write.

$$
\left(\frac{\rho_m}{\rho_\infty}\right) = \tanh \left(\frac{m\pi \tan \frac{\Delta}{2}}{n\pi \tan \frac{\Delta}{2}}\right) \quad (2.30)
$$

and

$$
\left(\frac{\rho_m}{\rho_n}\right) = \tanh \left(m\pi \tan \frac{\Delta}{2}\right) \quad (2.31)
$$

Where \( \tan \frac{\Delta}{2} = \left(\frac{\alpha_d\lambda_d}{2\pi}\right) \)

These relations can be used to determine the set of curves for $\left(\frac{\rho_n}{\rho_\infty}\right)$ and
$\left(\frac{\rho_m}{\rho_n}\right)$ as a function of $\tan \frac{\Delta}{2}$. From these curves $\tan \frac{\Delta}{2}$ can be determined and
parameters are computed from

$$
\varepsilon' = \left(\frac{\lambda_0}{\lambda_c}\right)^2 + \left(\frac{\lambda_0}{\lambda_d}\right)^2 \times \left(1 - \tan^2 \frac{\Delta}{2}\right) \quad (2.32)
$$
2.4 Surber’s Method

In case of Surber’s [17] method, \( \alpha_d \cdot \lambda_d \) is found by computing variation in the reflected power when the length of the sample is varied by moving a shorting plunger. The reflection co-efficient \( |\Gamma| \) at the face of the dielectric sample of length \( L \) enclosed in a wave guide cell and terminated by a perfectly reflecting short circuit, is given by

\[
|\Gamma| = \frac{Z_d \tanh \gamma_d L - 1}{Z_d \tanh \gamma_d L + 1}
\]  

(2.34)

If incident power happens to be constant, the output of the square law detector is proportional to \( |\Gamma|^2 \). This power may be recorded using a unidirectional coupler. If the liquid length chosen is an integral multiple of \( \left( \frac{\lambda_d}{2} \right) \), then the reflection co-efficient at the input of the liquid column, terminated in a short circuit, it given by [15-17]

\[
|\Gamma_n|^2 = T + 2F \exp\left(-2n\alpha_d \lambda_d\right) + \exp\left(-4n\alpha_d \lambda_d\right)
\]  

(2.35)

Where,

\[
T = |\Gamma_\infty|^2 = \left(\frac{(Z_d - 1)}{(Z_d + 1)}\right)^2
\]

and

\[
F = \left\{ \frac{1 - |Z_d|^2}{1 + |Z_d|^2} \right\}
\]

\( L = n\lambda_d \)

where \( n = \left\{ \frac{1}{2}, 1, \frac{3}{2} \right\} \)

For large values of \( n \), equation (2.35) reduces to

\[
|\Gamma_n|^2 = |\Gamma_\infty|^2 + 2F \left(1 - |\Gamma_\infty|^2\right) \exp\left(-n\alpha_d \lambda_d\right)
\]  

(2.36)
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\[ M_n = \left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2, \quad (M_n - 1) = 2F \left\{ \frac{1}{\left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2} - 1 \right\} e^{-\eta_d \lambda_d} \]  
(2.37)

or defining \( M_n \) such that \( M_n = \left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2 \)

\[ M_n = 1 + \left( \frac{2F}{\left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2} \right) \left( 1 + \left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2 \right) \exp(-n\alpha_d\lambda_d) \]  
(2.38)

or \[ (M_n - 1) = 2F \left( \frac{1}{\left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2} - 1 \right) \exp(-n\alpha_d\lambda_d) \]  
(2.39)

or \[ \log(M_n - 1) = \log 2F \left( \frac{1}{\left| \frac{\Gamma_n}{\Gamma_\infty} \right|^2} - 1 \right) - n\alpha_d\lambda_d \]  
(2.40)

A plot of \( \log(M_n - 1) \) versus \( n \) gives a straight line with slope \( \alpha_d\lambda_d \). Thus determining \( (\alpha_d\lambda_d) \), \( \varepsilon' \) and \( \varepsilon'' \) may be computed from the following relations.

\[ \varepsilon' = \left( \frac{\lambda_0}{\lambda_c} \right)^2 + \left( \frac{\lambda_0}{\lambda_d} \right)^2 \left\{ 1 - \left( \frac{\alpha_d\lambda_d}{2\pi} \right) \right\}^2 \]  
(2.41)

and \[ \varepsilon'' = \left( \frac{1}{\pi} \right) \left( \frac{\lambda_0}{\lambda_d} \right)^2 \alpha_d\lambda_d \]  
(2.42)

Where \[ \alpha_d\lambda_d = \frac{d}{dn} \log(M_n - 1) \]

2.5 Slot Line Method

The electromagnetic field at any point of a transmission line may be considered as the sum of two traveling waves. A wave from the generator incident on the load (which is not characteristic impedance) is reflected towards the generator due to mismatch, and so maximum power transfer will not occur. The reflected wave will combine with the forward wave to give a standing wave pattern [15-18]. The maximum field strength is found where the two waves add in phase and the minimum occurs where the two waves add in opposite phase, the distant between two successive minima or maxima is half the wavelength in a transmission line. The ratio of the amplitude of the maxima to the minima field strength of the wave is called the
standing wave ratio or ‘voltage standing wave ratio’ and is given by

$$VSWR = \frac{E_{max}}{E_{min}} = \frac{E_i + E_r}{E_i - E_r}$$

or

$$VSWR = \frac{\left(1 + \frac{E_r}{E_i}\right)}{\left(1 - \frac{E_r}{E_i}\right)}$$

(2.43)

Where \(E_1\) and \(E_2\) are respectively the amplitudes of the incident and reflected electric field strengths standing wave ratio is frequency expressed in decibels VSWR,

$$\text{db} = 20 \log_{10} \frac{E_{max}}{E_{min}}$$

Further, the ratio of the reflected to the incident electric field intensities is defined as reflected coefficient, i.e. \(\Gamma_r = \frac{E_r}{E_1}\)

Hence, \(VSWR = S = \frac{1 + \Gamma_r}{1 - \Gamma_r}\)

When \(E_{min} = E_{max}\), i.e. there is no reflection \([E_r = 0]\), the resulting VSWR=1, this is the requirement of a ‘matched circuit’.

Where \(\Gamma_r = \frac{E_r}{E_1}\) and is called reflection co-efficient.

The standing wave patterns are usually studied by means of the traveling detector \([6, 9]\), which probes the electric field intensity along the axis of propagation. We give brief description of the slotted wave guide technique for the measurement of \(\alpha, \lambda_d\) in dielectrics.

To sample the standing waves within a wave guide, a narrow longitudinal slot with ends tapered to give smoother impedance transformation and thereby giving minimum mismatch, is milled on the top of the broader dimension of the wave guide. Such section is called as slotted wave guide section. The slot is generally so many wavelengths long to allow many minima of standing wave pattern to be covered. The slot location is such that its presence does not influence the field configurations to any great degree. On this section, a probe inserted within a holder is mounted on a movable carriage Figure (2.2). The output is connected to a detector and indicating
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The necessary experimental setup is shown in Figure (2.2) to measure the dielectric parameters of a liquid sample. When the set up is assembled, the microwave source is energized and the output power is adjusted to required level. Setting the probe carriage for peak, meter reading, and the stub is adjusted until maximum output is indicated in the meter. With no sample in the cell, shorting plunger is moved downward carefully till it touches 90° bend. Now moving the probe carriage along the slot line the output meter reading are measured. Plotting curve between meter readings versus distance along the slot line we get standing wave pattern. From the plot we can obtain the guide wavelength (λg) of the exciting wave in the line.

Now removing the short circuit and then inserting the dielectric sample in the cell, short circuit plunger is introduced such that it touches the end of the sample. Moving probe carriage along the slot line the meter readings are recorded. The
standing wave pattern is obtained when cell is filled with dielectric by plotting meter reading versus distance along slot line.

From the standing wave pattern we can find out double the minimum width. If spacing between adjacent minima is \( \lambda_0 \) of the standing wave pattern then \( \lambda_d \) is given by \( \lambda_d = 2\lambda_0 \). VSWR can be obtained by using relation \( VSWR = S_n = \frac{(\lambda_d)}{\pi \Delta \lambda_n} \) for every minima; and from this can be computed the inverse \( VSWR = (\rho_n) = \frac{1}{S_n} \) where \( n = 1, 2, 3, \ldots \).

The graph between \( \rho_n \) versus \( n \) gives straight line and the slope of this line gives \( \alpha_d \).

\[
\alpha_d = \left( \frac{2\lambda_d}{\lambda^2_d} \right) \frac{(d \rho_{mean})}{(d \rho_n)} \tag{2.44}
\]

Now we can compute the values of loss factor \( (\varepsilon^\prime) \) using relations.

\[
\varepsilon'' = \left( \frac{1}{\pi} \right) \left( \frac{\lambda_n}{\lambda_d} \right)^2 \alpha_d \lambda_d \tag{2.45}
\]

and \( \tan \delta = \frac{\varepsilon''}{\varepsilon'} \tag{2.46} \)

2.6 Reflectometer Method

Reflectometer [15] is a special system, which permits rapid measurement of VSWR. Since there is a direct relationship between the VSWR and reflection coefficient \( (\Gamma_r) \) of a wave-guide, by measuring the ratio of relative amplitudes of reflected and incident signals, VSWR can be measured. The Figure (2.3) shows a basic reflectometer system. Components used to sample the incident and reflected powers are two directional couplers connected in opposite directions. Auxiliary arms of the two directional couplers are connected to the incident and reflected channels of a ratio meter. The \( (\Gamma_r) \) is determined and display it on the recorder. The forward directional coupler samples the incident power. The power coupled to the reverse directional coupler impedance on the load. If the load is not a matched one, then a portion of the incident power proportional to the degree of mismatch is reflected and travels towards the generator. The reverse directional coupler samples the reflected
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power which is applied to the reflected channel to the ratio meter measures and display the ratio of the magnitudes of reflected and incident power.

1) X-Y recorder or oscilloscope  
2) Sweep oscillator  
3) Modulator  
4) Variable attenuator  
5) & 6) Directional couplers  
7) Load  
8) Calibrated short circuit  
9) & 10) Tunned detectors  
11) Incident channel  
12) Reflected channel

**FIGURE 2.3** : Basic Reflectometer set-up

In principle, the reflectometer method is superior to slotted the measurement, because it measures the two dissimilar magnitudes while as the latter measures the ratio of nearly equal magnitudes. Further the slot line measuring system is insensitive to power fluctuations and slot errors. In the reflectometer method, the heart of the measurement centers around the dual three arm directional couplers, which are placed back to back. The most significant accuracy factor that contributes to the reflectometer method is the directivity of the reverse directional coupler. It must allow only an insignificant amount of forward energy to appear in the reverse direction.

After assembling the equipment, microwave power source is energized and the detector is tuned for maximum power output in power meter. The input power from the detector output, of forward directional coupler is noted. Similarly the output power from the detector reverse directional coupler is record. Suppose \( P_F \) and \( P_R \) are the forward and reverse powers measured by directional couplers then the power reflection co-efficient is obtained by

\[
\Gamma_p = \left( \frac{P_R}{P_F} \right)
\]
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Then,

\[ \Gamma_v = \sqrt{R_P} = \sqrt{\frac{P_R}{P_F}} \]

\[ VSWR (S) = \frac{1 + \Gamma_v}{1 - \Gamma_v} = \frac{1 + \sqrt{R_P}}{1 - \sqrt{R_P}} \]

\[= \frac{P_F + P_R + 2\sqrt{P_F P_R}}{P_F - P_R} \quad (2.47) \]

The above equation forms the basis for measuring VSWR using directional couplers. The above equation can also be written as

\[ S = \frac{I_F + I_R + 2\sqrt{I_F I_R}}{I_F - I_R} \quad (2.48) \]

Where \( I_F \) and \( I_R \) are respectively the currents corresponding to forward and reflected powers. Thus using the above equation (2.45) one can compute reflection coefficient and VSWR of the load under test using directional couplers. Further reflection coefficient \( \Gamma \) can be determined using relation \( \Gamma = VSWR - 1/VSWR + 1 \) for \( n = 1, 2, \ldots \) these values are used to find attenuation \( \alpha, \lambda \).

To find the VSWR by reflectometer of a given load the necessary experimental arrangement is shown in the Figure (2.4).

**FIGURE 2.4** : Setup for measuring dielectric constant of a material using reflectometer principle

1. Power supply
2. Microwave source
3 & 6. Isolators
4. Variable attenuator
5 & 7. Directional couplers
8. 90° - waveguide bend
9. Mica Joint
10. Short circuiting plunger
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(11) Dielectric cell (12) Micrometer screw to drive plunger
(13) & (15) Microwave power adaptor (14) & (16) Indicating power meter

2.7 Comparison of surber technique and slot line technique

So far we have discussed different techniques for the measurement of dielectric properties of binary liquid mixture using wave guides. In all the cases accurate measurements of wavelength in dielectric and VSWR is more important. Therefore in the present study the dielectric constant and dielectric loss were measured using Surber’s method [19] of measuring the reflection co-efficient from the air dielectric boundary of the liquid sample in the microwave X-band setup at 10.75 GHz microwave frequency.

Some remarks have to be made here in connection with the Surber’s technique employed in the present work. The slot line technique described above is time consuming even if low accuracy is desired. Further the slot line measuring system is insensitive to power fluctuations and slot errors. In principle the Surber’s method is superior to slotted line measurements because it measures the ratio of two dissimilar magnitudes of voltages while the latter measures the ratio of nearly equal magnitudes. The Surber’s technique permits rapid measurements of VSWR and is more convenient for practical purposes. Hence in the present work we have adopted Surber’s technique. Many researcher have been used this technique for measuring dielectric properties of binary liquid mixtures.

2.8. Instrumentations

2.8.1 DMA 35 Portable density meter

Definition of density

The density $d$ of a sample is defined as its mass divided by its volume.

$$d = \frac{m}{v}$$

Density is a temperature dependent measuring unit.

DMA 35 – an Overview

The moveable density meter DMA 35 measures the density of liquid in g/cm$^3$. oscillating U-tube principle is used in this density meter. We can measure density of binary liquid mixture at different temperatures from 0°C to 40°C. A temperature sensor measures the temperature of binary liquid mixture right at the measuring cell.
The density and temperature of binary liquid mixture is displayed on the screen.

Samples are filled into the measuring cell using the built-in pipette-style pump or a syringe. DMA -35 portable density meter was used to determine the densities of binary systems. This instrument was calibrated with distilled water after that we measured the samples.

DMA 45 portable density meter

**Specification**

- Manufacturer: Antan paar GMBH, Austria (Europe)
- Accuracy: 0.001 g/Cm$^3$
- Measuring range density: 0 to 3 g/Cm$^3$
- Temperature: 0 to 40°C.

### 2.8.2 Abbe’s Refractometer

Abbe’s refractometer was used to determine the refractive indices of...
binary systems. This instrument was calibrated with distilled water after that we measured the samples.

**Application and Specifications**

a. Allows measurement of refractive indices of liquids, transparent and translucent solutions and solids.

b. Measurable range can be increase from 1.300 to 1.700 with the help of sodium D line.

c. Accurateness 0.001 by direct reading and 0.0001 by evaluation.

d. Finely marked glass translucent arc scale.

e. Permits measurement of percentage of sugar.

f. Allows measurement of dispersion.

g. Water jacked prism box: allows measurement of index of refraction at the preferred temperature.

h. Compensator consisting of amice prisms: allows to compensate for dispersion thus making it possible to have a fine critical line.

i. Right side on RI is use bricks or sugar cone.

Abbe’s Refractometer manufactured by Mittal Enterprise New Delhi-110000, India.

**2.8.3 The Brook field DV-II + Pro Viscometer**

The Brook field DV-II + Pro Viscometer measures fluid viscosity at a given shear rates. Viscosity is a measure of fluid’s resistance to flow.
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Principle

The principle of operation of the DV-II + Pro is to drive a spindle (which is immersed in the test fluid) through calibrated spring the viscous drag of the fluid against the spindle, is measured by the spring deflection. Spring deflection is measured by using a rotary transducer. The measurement range of a DV-II + Pro (in centipoises or mPsec) is calculated by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, as well as the full scale torque of the calibrated spring.

The higher the torque calibration, the higher the measurement range

- Viscosity range is inversely proportional to the size of the spindle.
- Viscosity range is α (1 / rotational speed)
- To measure high viscosity, choose a small spindle or a slow speed.
- Satisfactory result between 10-100% torque scales.
- Spindle must rotate at least five times readings are taken.

This Brook field DV-II + Pro viscometer was used to determine the viscosities of binary systems.

This instrument was calibrated with distilled water after that we measured the samples.

Manufacturer: Brook field DV-II + Pro model LVDV- II + Pro Brook field engineering laboratories INC USA.

Specifications

Speeds: Choice of three options.

Instrument has “Interleaved” speed when manufactured.

Standalone: Interleaved: L/RV (18 speeds) 8LV speeds followed by 10 RV speeds.

Sequential: LV/RV (18 speeds) 8 LV speeds and 10 RV speeds arranged in sequential order from 0.3 rpm to 100 rpm.

Custom : 54 speeds user selectable temperature sensing range -100°C to 300°C(-148°F 572°F)

Analog torque output 0-1 vol+ Dc (0-100% torque)

Analog temperature output 0-3.75 to DC (-100°C to +275°C)

Viscosity accuracy: + 1% of full scale
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- Viscosity repeatability: + 0.2%
- Temperatures: + 1°C - 100°C to 149°C
- Accuracy: + 2°C \ (+150°C to +300°C
- Operating environment: 0°C to 40°C temperatures range (32°F to 104°F) 20%-80% R.H. non-condensing atmosphere.

2.9 Determination of Microwave Power Meter

PM-437 is a cost high performance microwave power meter for microwave power measurements. It can measure the frequency range from 50 MHz to 12.4 GHz (AT11801). It can measure power levels 0.1 µW ~ 100 mW. It’s a portable design keeping in mind the bench and field service application PM-437 power indicator from its power sensors and components.

The high performance and accuracy is obtained by the power sensor by chopper and by the linear correction circuit components. Chopper is using high-sensitivity, low noise MOS-FET switch components series chopper. Its main role is to transform the probe by the power of DC input signal modulation signal into equivalent exchanges, and through the CPU to carry out the processing.

Microwave power meter have preamplifier as the high-gain transistor and the high impedance low-barrier comprising operational amplifier with a gain amplifier exchanges about 600 hybrid amplifiers.

System specifications

- Frequency range : 50 MHz ~ 12.4 GHz
- Power Range : 0.1 µW ~ 100 mW
- Readout accuracy : Data ± 8%
- VSWR : ≤ 1.4
- Power Reference : 50 MHz 1.00 mW ± 1.5%
- Display Resolution : 0.01 dB
- Recorder output : 0 ~ 1 voltage, output impedance 1KΩ
- Display : W, dBm, dB
- Line Voltage Range : 220V AC ± 10%, 50Hz ~ 60Hz, 10W
- Size : 260mm (L) * 250mm (W) * 110mm (H)
- Weight : about 3.7 Kg

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Steps to use Power Meter

1. Connect the power sensor to the instrument.
2. Switch on the unit and let it warm up for at least 20 minutes.
3. Press ZERO key and ZERO will be displayed on the instrument. Press Enter key immediately. This will ensure zero calibration for the power meter.
4. Now connect the power sensor carefully to the power reference output.
5. Press CAL key and these after press enter key. This will ensure 1mW/0 dbm calibration point for the power meter.
6. Now the meter will display 1mW or 0 dbm depending on the unit of measurement.
7. Now press PWR REF key to disconnect the reference power. Also disconnect physically the power sensor from the instrument.
8. Press SET key more than once to get frequency of the key is for attenuation setting. The range of at setting is 0 to 30 db and frequency setting range is between 50MHz to 18 GHz.
9. In the microwave lab if the frequency used is 10 GHz then use ARROW key to get 10 on the display followed by ENTER key. This will ensure the power meter calibration for frequency. Now the meter is ready to use.

2.10 Low Temperature Circular Bath

NIVITECH Low Temperature Circulating Bath is used for maintaining the required constant temperature (Range – 40°C to + 40°C) of the sample in the corresponding instruments by connecting to it.

Cooling mechanism of low temperature circulating bath consist of hermetically sealed cascade refrigeration system. Compressor make of Kirloskar
Copeland. Forced air cool condenser and immersion type cooling coil copper plated make.

2.11 X–Band

Description

Figure 2.4 of this chapter II shows the actual arrangement used. The rectangular wave guide bench consists of a Microwave source Klystron power supply (model KPS- 151) connected to a Klystron (2k25) mounted on the Klystron mount (X 2051) capable of generating oscillations at about 9,000 Mc/s. The setup also comprises the following: an isolator (X 6021), a variable attenuator (X 5020), frequency meter (X 4155), directional coupler (X 6000), wave guide section (X4051), detector mount (X4155), E-plane (X3061), E-plane bend (X7071), precision movable short (X4181) and the dielectric liquid cell (X910). The slotted section in the broad face to accommodate a tunable probe-carriage is connected to the liquid dielectric cell and 90 ° E - plane wave guide bend. The liquid cell is fitted with the Teflon ended movable plunger driven by a micrometer screw gauge (0.01 x 10³m). The cell is provided with an outer water circulation jacket for maintaining constant temperatures. The sample length of solution taken in the cell could be adjusted using the movable plunger.
2.11.1 Operational specification of X-band microwave bench

1. The power supply is switched on and the system is allowed to stabilize. When the beam is turned ON the voltmeter reads 300V. Then the beam is turned OFF and the power is switched off. The Klystron is connected to the power supply. An electric fan is used to keep the Klystron air cooled.

2. The power supply is switched ON and the modulation switch is turned to CW Position.

3. Variable attenuator is set to a maximum.

4. Power meter is connected to the crystal O/P.

5. The beam is turned ON Beam voltage of 250V beam current of 20-25mA indicate the proper working of the bench.

6. The deflection in the Power meter is checked.

2.12 To determine the wave length of the wave guide and frequency

1. For every maximum and minimum deflection of Power meter, obtained by moving the plunger in dielectric liquid cell, the readings are noted.

2. The difference between the readings corresponding to two consecutive either maxima or minima gives the value of $\lambda_g/2$.

3. The free space wavelength $\lambda_0$ is calculated from

$$\frac{1}{\lambda_0^2} = \frac{1}{\lambda_g^2} + \frac{1}{\lambda_c^2}$$

Where $\lambda_c$ is the cut off wavelength equal to twice the broader dimensions of the wave guide ($a = 2.248 \times 10^{-2}$ m).

4. The frequency ($\nu$) of the microwave setup is obtained from the relation, $\nu = C/\lambda$, where C is the velocity of light.

2.13 Determination of dielectric constant and dielectric loss

1. The tunable probe is positioned in the maximum of the standing wave pattern and fixed. The dielectric liquid under study is taken in the closed dielectric cell and the plunger is introduced until it touches the 90° bend.

2. The position of the first minimum is noted by slowly moving the plunger up through the dielectric medium. Also the Power meter reading is noted ($S_1$).

3. By slowly moving the plunger up in the dielectric the position of the second minimum along with the Power meter reading is noted ($S_2$).

4. This process is repeated by moving the plunger up in the dielectric and noting...
the successive minima until the plunger is about to come out of the liquid.

5. The radiation wavelength in the dielectric $\lambda_d$ can be measured by calculating the distance between the successive minima $(\lambda_d)/2$.

6. The value of $\rho_n$ for each minimum setting which is nothing but the inverse of the VSWR meter reading ($S_n$) can be calculated, when $n$ represents the successive minima.

7. A graph is plotted between $\rho$ and $n$ whose slope value

   i. $(d\rho_{\text{mean}}/dn)$ can be found.

8. The value of $\varepsilon'$ and $\varepsilon''$ are then computed.