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# Dielectric Studies using Cavity Perturbation Technique in X-Band at Room Temperature on Nonyl *P*- Hydroxy Benzoate

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**ABSTRACT:** Cavity perturbation technique in microwave frequency region, LCR bridge for static dielectric constant and Abbe's refractometer instruments for measurement of dielectric constant were used. For dipole moments Higasi method was used in the dielectric study of the various concentrations of the binary system of nonyl *p*-hydroxy benzoates (9HB) and Isopropanol and benzene as solvent. There is a decrease in the dipole moment as the concentration of the benzoate increased in solute system in non-polar solvent benzene and they are attributed to the formation of hydrogen bond between the solute systems.

**KEYWORDS:** Cavity perturbation technique, Dielectric constant, Higasi method, Dipole moment, Hydrogen bond.

## I. INTRODUCTION

Studies of liquid mixtures have gained importance [1-3] because it provides one way of bringing together the molecules of different compounds and allow them to interact with one another. The physical forces responsible for such interactions cannot be studied when such molecules are isolated. Molecular mixtures bring about changes in thermodynamic properties like entropy, free energy and heat of mixing. Changes in physical properties like density, molar volume, viscosity, surface tension, refractive index, dielectric constant and speed of acoustic waves are manifestations of intermolecular interactions in liquid mixtures. Dielectric investigations of solutions containing varying amounts of interacting molecules help detect the formation and composition of complexes in them. The nature of complex formation in binary mixtures is still far from clear. The present trend is of liquid mixtures leading to formation of hydrogen bonding in the system due to solute-solvent interactions. Hydrogen bonding is a complex in the liquid state because of the uncertainty in identifying the particular bonds and the number of molecules involved. One of the physical parameters used for conformational analysis of any resultant structure is the net dipole moment, relaxation time and thermodynamic parameters [4-6]. This investigation is aimed at studying the dielectric behaviour of binary mixtures of nonyl *p*-hydroxy benzoate (9HB) and isopropanol in benzene, which will manifest useful information about the formation of complex in the mixture. Nonyl *p*-hydroxy benzoate (9HB) is used in various fields viz. food preservatives, cosmetic preservatives [7-9].

## II. RELATED WORK

A resonant cavity method intermediate between the Hartshorn-wartd method and the micro wave resonance methods suited for measurement on low loss materials and described by Works, Dakin and Boggs [10]. Birnbaum *et al* [11] studied the microwave resonance method to determine the dielectric constant and loss of liquids, solids and very lossy gases. A microwave resonance technique suited for measurement on low loss liquids has been described by Pitt and Smyth [12]. Cavity perturbation method is widely used in the characterization of dielectric [13], magnetic [14, 15] and super conducting materials [16]. The different microwave cavity perturbation techniques are discussed by Raman [17]. The application of microwave cavity perturbation technique for study of transients in semiconductors is becoming popular due to its simplicity in measurement procedure and high sensitivity. Effects of quality factor, sample size, and coupling factor of the sensitivity of the measurement are discussed. [18]. The complex dielectric constant for different

liquids are measured using waveguide transmission-line techniques at four frequency ranges, 8-12.4GHz, 12.4 -18 GHz, 18-26.5GHz and 26.5-40GHz.. The results are discussed and compared with the literature and the calculated data using debye's method [19]. In this paper, the measurements of dielectric constant of some liquids carried out in the X-band of the microwave spectrum are reported. A Gunn flange together with three cavities has been constructed with suitable holes for sample insertion. The theoretical analysis of the perturbation theory concerning the modified cavity-controlled oscillator is given and experimental measurements of the frequency shift for each designed cavity is presented. A comparison is achieved between the four positions of the cavity from which the optimum cavity position is selected. Finally, limitations of this method are represented [20].

### III. EXPERIMENTAL

M/s Frinton Laboratories Inc, USA supplied 99.5% purity Nonyl *p*-hydroxy benzoate (9HB). ExcelaR grade Isopropanol and Benzene were obtained from Qualigens, India and double distilled before use. Benzene is used as solvent, the binary system of nonyl *p*-hydroxy benzoates (9HB) and Isopropanol is used as solute in the preparation of solution. 9HB is dissolved in Isopropanol in various concentrations, the various weights of 9HB for each concentration is given for 26.5 ml of Isopropanol, the mole fraction of 9HB in isopropanol from 0.01, 0.02, 0.03, 0.04 and 0.05 %, the weight of 9HB of 0.18077, 0.36524, 0.55350, and 0.74571 grams respectively. All the weights were measured using a single pan electronic balance Dhona make, model 100 DS with an accuracy of 0.01 mg. Each concentration of solute is diluted with benzene in volume fractions of 90% benzene and 10% solute, 80% benzene and 20% solute, 70% benzene and 30% solute, and 60% benzene and 40% solute. The dielectric studies were carried out using LCR bridge for static dielectric constant at 1kHz., the microwave bench adopting cavity perturbation technique [1,10-14] method (in X-band) Resonant frequencies  $f_0$ ,  $f_1$  and  $f_2$  also correspond to empty cavity, cavity with empty holder alone and lastly cavity containing holder with the sample within it.

$$\epsilon^1 = 1 + \frac{V_c}{4V_s} \left[ f_0^2 \left( \frac{1}{f_2^2} - \frac{1}{f_1^2} \right) \right]$$

where  $V_c$ ,  $V_s$ =Volume of the cavity and sample respectively. The error in the real part of the dielectric constant is 2% .

Abbe's refractometer used for refractive index (high frequency dielectric constant  $\epsilon_\infty$ ) at room temperature (300 °K). The error in the estimation of  $\epsilon_\infty$  is +0.04%.

### IV. RESULTS AND DISCUSSION

The experimental results obtained from the static, microwave and optical frequencies for different concentrations of 9HB in isopropyl alcohol, diluted in a non polar solvent medium as benzene are discussed. The solute (9HB+ Isopropanol) is a highly polar compound. Hence highly saturated wave pattern is obtained in the measurement of complex dielectric constant at microwave frequency by using the Cavity technique. Due to the high loss nature of the sample, measurement of the dielectric constant at high concentrations of 9HB in isopropyl alcohol systems is not possible. In order to study the properties of these systems, we diluted the above system for different concentrations of non polar solvent medium benzene. Diluting the system in non polar solvent medium it is useful for studying the interaction between the 9HB and Isopropanol. From the standing wave pattern in the microwave frequency region, dilution studies of different concentrations of solute (9HB and Isopropanol) in non polar solvent medium is easy to obtain the dielectric constant. The measurements are done at room temperature (300°K). The dielectric investigations are carried out at low frequency using LCR meter, at microwave frequency using cavity technique and at optical frequency using Abbe refractometer. The data obtained from different concentrations of the solute in a nonpolar solvent medium at static and optical frequency are used to evaluate the dipole moments of the solute using Higasi's method and this method is applicable only for dilute solutions.

The signals obtained in C.R.O when connected to the tuned detector without and with sample in cavity perturbation technique are shown in Plate 1. and 2 respectively. The dielectric constant is measured by using the equation given in the experimental section.



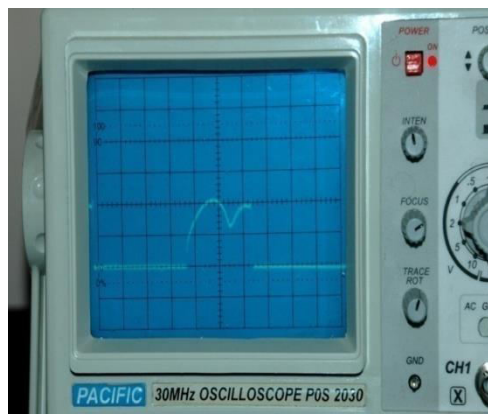


Plate 1 cavity with empty holder

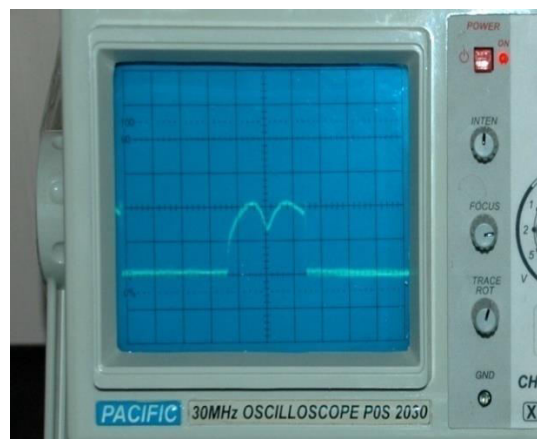


Plate 2 cavity containing holder with the sample

### Standardization of the cavity technique:

Using the cavity perturbation technique, the dielectric constant of some standard liquids like carbon tetra chloride ( $\text{CCl}_4$ ) and chlorobenzene have been measured and tabulated in table 1. These parameters are compared with the reported values in the literature [21]. From the table 1 it can be observed that the values of  $\epsilon'$  and  $\epsilon''$  are similar to the reported values within the accuracy of the experiment.

Table 1 The dielectric constant and dielectric loss of  $\text{CCl}_4$  and chlorobenzene using the fixed cavity resonator in X-band.

| Sample         | Dielectric constant ( $\epsilon'$ ) |               | Dielectric loss ( $\epsilon''$ ) |               |
|----------------|-------------------------------------|---------------|----------------------------------|---------------|
|                | Observed                            | Reported [55] | Observed                         | Reported [21] |
| $\text{CCl}_4$ | 2.25                                | 2.23          | ---                              | ---           |
| Chlorobenzene  | 4.67                                | 4.73          | 1.65                             | 1.50          |

Table 2. Dielectric data for various concentrations of (4HB + Isopropanol) in Benzene using cavity technique

| % of 4HB in isopropanol | Volume fraction of solvent + solute | $\epsilon_{\text{microwave}}$<br>$\epsilon^* = \epsilon' - j\epsilon''$ [59] |             |
|-------------------------|-------------------------------------|------------------------------------------------------------------------------|-------------|
|                         |                                     | Observed                                                                     | Reported    |
| 0.02%                   | 90+10                               | 2.53-j0.22                                                                   | 2.39-j0.25  |
|                         | 80+20                               | 3.11-j0.47                                                                   | 3.37-j0.41  |
|                         | 70+30                               | 3.23-j0.61                                                                   | 3.59-j0.75  |
|                         | 60+40                               | 3.57-j0.73                                                                   | 3.71-j0.80  |
| 0.03%                   | 90+10                               | 2.88-j0.33                                                                   | 2.81-j0.27  |
|                         | 80+20                               | 3.26-j0.46                                                                   | 3.34-j0.41  |
|                         | 70+30                               | 3.32-j0.69                                                                   | 3.39-j0.81  |
|                         | 60+40                               | 3.64-j0.87                                                                   | 3.72-j0.96  |
| 0.04%                   | 90+10                               | 2.95-j0.32                                                                   | 2.79-j0.21  |
|                         | 80+20                               | 3.46-j 0.34                                                                  | 3.65-j 0.41 |
|                         | 70+30                               | 3.68-j 0.77                                                                  | 3.81-j 0.62 |
|                         | 60+40                               | 4.03-j 0.87                                                                  | 4.03-j 0.98 |

Now the experiment is repeated with the present investigating compound 9HB using cavity perturbation technique for different concentrations at room temperature. Because 9HB is a high lossy compound, it becomes difficult to observe the frequency shift. Consequently dielectric loss can't be calculated due to lossy nature though diluted with benzene. Hence only dielectric constant is measured with 9HB.

Further, the dielectric constant is measured in different concentrations at different frequencies at room temperature. The results are tabulated in the table 3.

Table 3. Comprehensive dielectric data at different frequencies for Various concentrations of (9HB+Isopropanol) in Benzene

| % of 9HB in isopropanol | Volume fraction of solvent + solute | $\epsilon_{\text{static}}$ | $\epsilon_{\text{microwave}} (\epsilon')$<br>(Cavity) | $n^2$<br>(Optical frequency) |
|-------------------------|-------------------------------------|----------------------------|-------------------------------------------------------|------------------------------|
| 0.01%                   | 90+10                               | 2.000                      | 2.7273                                                | 2.226                        |
|                         | 85+15                               | 2.915                      | 2.8187                                                | 2.205                        |
|                         | 80+20                               | 3.070                      | 2.9102                                                | 2.184                        |
|                         | 75+25                               | 3.685                      | 3.0017                                                | 2.164                        |
| 0.02%                   | 90+10                               | 2.640                      | 2.7273                                                | 2.223                        |
|                         | 85+15                               | 3.030                      | 2.9102                                                | 2.208                        |
|                         | 80+20                               | 3.420                      | 3.0017                                                | 2.187                        |
|                         | 75+25                               | 4.170                      | 3.1851                                                | 2.167                        |
| 0.03%                   | 90+10                               | 2.710                      | 2.6351                                                | 2.232                        |
|                         | 85+15                               | 3.180                      | 2.8187                                                | 2.215                        |
|                         | 80+20                               | 3.620                      | 2.9102                                                | 2.190                        |
|                         | 75+25                               | 4.170                      | 2.9102                                                | 2.185                        |
| 0.04%                   | 90+10                               | 2.770                      | 2.7273                                                | 2.229                        |
|                         | 85+15                               | 3.230                      | 2.8187                                                | 2.208                        |
|                         | 80+20                               | 3.730                      | 2.8187                                                | 2.187                        |
|                         | 75+25                               | 3.930                      | 2.8187                                                | 2.177                        |

### Dipole moment:

The dipole moments are calculated from Higasi method (refer chapter 1).

$$\mu^2 = \frac{27 k T M_2}{4 \pi N d_1} \cdot \frac{(a_0 - a_\infty)}{(\epsilon_1 + 2)^2}$$

where  $M_2$  is molecular weight of solute,  $d_1$  is density of solvent,  $\epsilon_1$  is the dielectric constant of solvent (benzene).  $a_0$  and  $a_\infty$  are the respective slopes of the graphs  $\epsilon_0$  and  $\epsilon_\infty$  with respect to the concentration. The other constants have the usual meaning.

The data required for calculating dipole moment are given along with the dipole moment at each concentration in Table 4.

The values of  $a_0$  and  $a_\infty$  are found from the graphs of  $\epsilon_0$  and  $\epsilon_\infty$  against concentration respectively which are shown representative Figs. 1 and 2.  $\epsilon_0$  (static dielectric constant) is measured at 1 kHz using LCR meter.  $\epsilon_\infty$  is the dielectric constant measured at optical frequency.

The dipole moment values decrease with increase of 9HB in Isopropanol which is probably due to the hydrogen bonding in the system.

However from the present study the dipole moment is in between 5.8144 to 4.8880 for various concentrations of 9HB in Isopropanol.

From the Table 4 it can be observed that, the static dielectric constant ( $\epsilon_{\text{static}}$ ) increases with the increase of concentration of solute.

Table 4: Values of parameters for dipole moment

| in % of 9HB in Isoprop anol | Weight of solute (W <sub>1</sub> ) | Weight of solvent (W <sub>2</sub> ) | Weight fraction of solute (W <sub>1</sub> /W <sub>1</sub> +W <sub>2</sub> ) | $\epsilon_{static}$ | n <sup>2</sup> (Optical frequency) | a <sub>0</sub> | a <sub><math>\alpha</math></sub> | Dipole moment ( $\mu$ ) in Debye |
|-----------------------------|------------------------------------|-------------------------------------|-----------------------------------------------------------------------------|---------------------|------------------------------------|----------------|----------------------------------|----------------------------------|
| <b>0.01%</b>                | 0.4106                             | 3.9465                              | 0.0942                                                                      | 2.000               | 2.226                              | 10.873         | -0.432                           | 5.81                             |
|                             | 0.6159                             | 3.7273                              | 0.1418                                                                      | 2.915               | 2.205                              |                |                                  |                                  |
|                             | 0.8212                             | 3.5080                              | 0.1897                                                                      | 3.07                | 2.184                              |                |                                  |                                  |
|                             | 1.0264                             | 3.2888                              | 0.2379                                                                      | 3.685               | 2.164                              |                |                                  |                                  |
| <b>0.02%</b>                | 0.4290                             | 3.9465                              | 0.0981                                                                      | 2.64                | 2.223                              | 10.110         | -0.384                           | 5.60                             |
|                             | 0.6435                             | 3.7273                              | 0.1472                                                                      | 3.03                | 2.208                              |                |                                  |                                  |
|                             | 0.8580                             | 3.5080                              | 0.1965                                                                      | 3.42                | 2.187                              |                |                                  |                                  |
|                             | 1.0726                             | 3.2888                              | 0.2459                                                                      | 4.17                | 2.167                              |                |                                  |                                  |
| <b>0.03%</b>                | 0.4479                             | 3.9465                              | 0.1020                                                                      | 2.71                | 2.232                              | 9.125          | -0.328                           | 5.32                             |
|                             | 0.6718                             | 3.7273                              | 0.1527                                                                      | 3.18                | 2.215                              |                |                                  |                                  |
|                             | 0.8957                             | 3.5080                              | 0.2033                                                                      | 3.62                | 2.19                               |                |                                  |                                  |
|                             | 1.1197                             | 3.2888                              | 0.2539                                                                      | 4.17                | 2.185                              |                |                                  |                                  |
| <b>0.04%</b>                | 0.4671                             | 3.9465                              | 0.1059                                                                      | 2.77                | 2.229                              | 7.651          | -0.341                           | 4.89                             |
|                             | 0.7006                             | 3.7273                              | 0.1582                                                                      | 3.23                | 2.208                              |                |                                  |                                  |
|                             | 0.9341                             | 3.5080                              | 0.2103                                                                      | 3.73                | 2.187                              |                |                                  |                                  |
|                             | 1.1677                             | 3.2888                              | 0.2620                                                                      | 3.93                | 2.177                              |                |                                  |                                  |

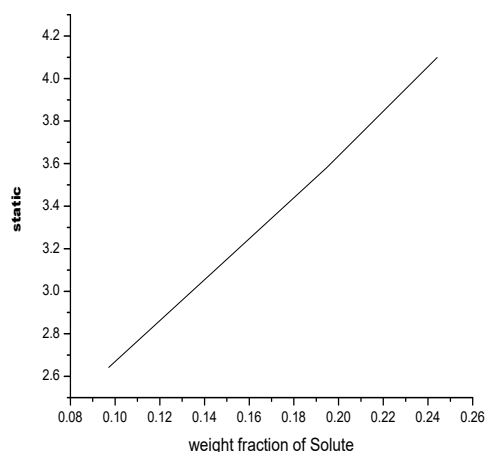


Fig.1. Weight fraction of Solute Versus Static dielectric Constant For 0.01MF of 9HB in Isopropanol

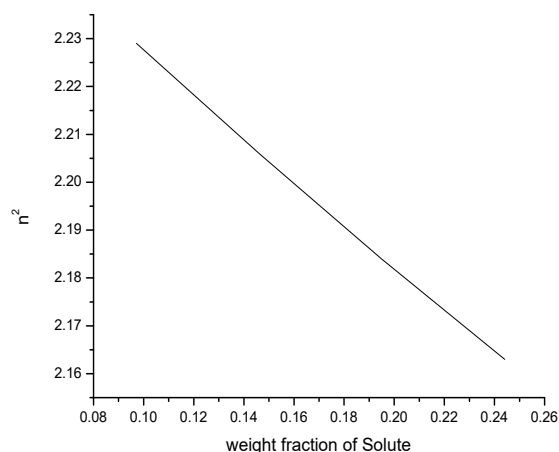


Fig.2. Weight fraction of Solute Versus Optic dielectric constant For 0.01MF of 9HB in Isopropanol

**Table 5. Comprehensive data on dielectric constant, dipole moment for various concentrations of (9HB + Isopropanol) in Benzene**

| % of 9HB in isopropanol | Volume fraction of solvent + solute | $\epsilon_{\text{static}}$ | $\epsilon_{\text{microwave}}$<br>$\epsilon''$ | $n^2$<br>(Optical frequency) | Dipole moment $\mu$ (Debye) |
|-------------------------|-------------------------------------|----------------------------|-----------------------------------------------|------------------------------|-----------------------------|
| 0.01%                   | 90+10                               | 2.000                      | 2.7273                                        | 2.226                        | 5.81                        |
|                         | 85+15                               | 2.915                      | 2.8187                                        | 2.205                        |                             |
|                         | 80+20                               | 3.070                      | 2.9102                                        | 2.184                        |                             |
|                         | 75+25                               | 3.685                      | 3.0017                                        | 2.164                        |                             |
| 0.02%                   | 90+10                               | 2.64                       | 2.7273                                        | 2.223                        | 5.60                        |
|                         | 85+15                               | 3.03                       | 2.9102                                        | 2.208                        |                             |
|                         | 80+20                               | 3.42                       | 3.0017                                        | 2.187                        |                             |
|                         | 75+25                               | 4.17                       | 3.1851                                        | 2.167                        |                             |
| 0.03%                   | 90+10                               | 2.71                       | 2.6351                                        | 2.232                        | 5.32                        |
|                         | 85+15                               | 3.18                       | 2.8187                                        | 2.215                        |                             |
|                         | 80+20                               | 3.62                       | 2.9102                                        | 2.190                        |                             |
|                         | 75+25                               | 4.17                       | 2.9102                                        | 2.185                        |                             |
| 0.04%                   | 90+10                               | 2.77                       | 2.7273                                        | 2.229                        | 4.89                        |
|                         | 85+15                               | 3.23                       | 2.8187                                        | 2.208                        |                             |
|                         | 80+20                               | 3.73                       | 2.8187                                        | 2.187                        |                             |
|                         | 75+25                               | 3.93                       | 2.8187                                        | 2.177                        |                             |

Comparing the results obtained from cavity perturbation technique the dielectric constant at microwave Frequency with the plunger technique [22] are nearly equal within the accuracy which is shown table 6.

**Table 6. Comparative data on dipole moment by cavity and plunger techniques**

| % of Compound in Isopropanol | Cavity technique $\mu$ (Debye) (Present study) | Plunger technique] $\mu$ (Debye) [22] |
|------------------------------|------------------------------------------------|---------------------------------------|
| 0.01%                        | 5.81                                           | 5.39                                  |
| 0.02%                        | 5.60                                           | 5.19                                  |
| 0.03%                        | 5.32                                           | 5.10                                  |
| 0.04%                        | 4.89                                           | 5.08                                  |

In the microwave region also the dielectric constant increased with increased concentration of 9HB in binary system. But at optical frequency region the dielectric constant decreases when the concentration of solute increases in the binary system. It is due to the decrease in dipolar polarization contribution at high frequency. At high frequency the dipolar polarization disappears and only atomic polarization or electronic polarization will present. The dielectric constant decreased with increased frequency from static (low frequency) to optical (high) frequency. It means the contribution of dipolar polarization decreased with increased in frequency. At static frequency all polarizations are present.

#### IV. CONCLUSIONS

The dielectric constant decreases with the increased frequency. It is because the orientation polarization fails to reach its equilibrium value and contributes less and less to the polarization as the frequency rises. It decreases the dielectric constant. The low frequency static dielectric constant and microwave frequency dielectric constant increased with increased concentration of the solute binary system in non-polar solvent benzene at room temperature. The optical refractive index value is increased with decrease in the concentration of the solute binary system (benzoate and alcohols) in non-polar solvent benzene at room temperature. With an increase in concentration of the benzoate in solute system decreases the in the dipole moment in a non-polar solvent benzene and it is due to formation of the hydrogen bond between the solute system.

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