7.1 Introduction

When two or more liquids are mixed, the conformational change of the molecules is broadly studied by dielectric method. This change may be due to the interaction between the components of the molecules. The formation of a hydrogen bond between two molecules A-H and B always suggests the existence of some polarity in the proton donor, the component A. It is necessary in effect that the electron cloud around the proton should be weakened in order that A-H can present the acidic characters needed for the formation of the H-bond. On the other hand the proton acceptor or the component B of the molecule must show reinforced parts in the external electron clouds. On account of this the molecules A-H and B, which are able to form H-bond, show a certain dipolar character from the beginning, which will show the existence of dipole moments $\mu_a$ and $\mu_b$ in the separated molecules.

When the H-bond is formed, the dipole moment $\mu_{ab}$ of the complex never corresponds with the vector sum of the moments of the components $\mu_a$ and $\mu_b$. In effect the formation of the bond brings about variations in the relative positions of some nuclei and also deformation of the electron clouds. These displacements of the charges cause a difference between the moments of the complex and the vector sum of the components. The dipole increment $\Delta \mu$ is defined by this difference.

$$\Delta \mu = \mu_{ab} - \mu_a - \mu_b$$

The dipole increment is a very important characteristic of the H-bond. It describes the significance of the relative displacement of the nuclei and the electrons when the H-bond is formed. The dipolar increments were first studied by Smyth et al. [1]. In hydrogen bonding of proton donor-acceptor complexes, a redistribution of electron density takes place and the dipole moment of the H-bonded system is not additive. The increase in the distance of the O-H bond enhances the dipole moment $\mu$. The determination of this dipolar increment ($\Delta \mu$) will indicate the type of complex. Several workers have studied the dipolar increments in liquid mixtures [2-3].

There are many reasons for the dipolar increment. Pioneering work in this area was done by Sobczyk and his co-workers [4] and Huyskens et al. [5]. In many
$O - H \cdots N$ and $O - H \cdots O$ complexes, the dipolar increment is due to either polarization effect [6] or charge-transfer effect [7] or partial proton transfer effect [8] or complete proton transfer effect [9]. Reaction field effect of the environment is also a major cause of the change in dipole moment of the complexes in a non-polar medium [10].

7.2 Theoretical background

7.2.1 Determination of dipole moment of complex

The theory of Frohlich [11] is adequate to obtain the overall dipole moment $\mu$ of a system of solute-solvent mixture. For a solution containing polar substances and an apolar solvent, this quantity can be calculated from the relative dielectric constant $\varepsilon_o$ of the solution, refractive index $n_o$ for the sodium line and the density of the solution and the solvent. This is as follows;

$$
M^2 = \frac{9kTV}{4\pi} \left[ \frac{(\varepsilon_o - n_o^2)(2\varepsilon_o + n_o^2)}{\varepsilon_o(n_o^2 + 2)^2} \right] - X_s \frac{(\varepsilon_s - n_s^2)(2\varepsilon_s + n_s^2)}{\varepsilon_s(n_s^2 + 2)^2} \tag{7.1}
$$

where $V$ is the molar volume of the solution, $k$ is the Boltzmann constant and $X_s$ is the mole fraction of the apolar solvent. The second term is the correction term for the internal refractive index as measured for the sodium line.

Let us consider a ternary mixture of the polar components A and B in an apolar solvent S. A and B may involve in H-bonding. In view of the mobility of the liquid phase, the relative orientation of A and B continuously changes. The dipole moment of the solution at a given time, assuming that the time interval is short enough to consider the orientations as fixed, may be written as

$$
M^2 = \sum_{i=0}^{\infty} \sum_{j=0}^{\infty} N_{ij} \mu_{ij}^2 \tag{7.2}
$$

where $N_{ij}$ is the number of ij ensembles.

Siegel et al. [12] showed that Eq. (7.2) could be written as
Here, $< \frac{\mu_{ij}^2}{j} >$ is the mean share of the B molecule in the square of the total dipole moment of the entities. For ethers as proton acceptors B $< \frac{\mu_{ij}^2}{j} >$ is practically constant. The quantity $\left( \frac{\mu_{ij}^2}{i} - < \frac{\mu_{ij}^2}{j} > \right)$ represents the mean share of an A molecule in the square of the dipole moment of the entities. The formal numbers of moles of the polar components is related to $N_{ij}$’s as

$$n_A = \sum_{i=0}^{\infty} \frac{iN_{ij}}{N_A}$$  \hspace{1cm} (7.4)$$

$$n_B = \sum_{j=0}^{\infty} \frac{jN_{ij}}{N_A}$$  \hspace{1cm} (7.5)$$

where $N_A$ is Avogadro’s number. Hence Eq. (7.3) can be written as

$$\frac{M^2}{VN_A} = < \mu_A^2 - \mu_B^2 > C_A + < \mu_B^2 > C_B$$  \hspace{1cm} (7.6)$$

where $C_A$ and $C_B$ are the formal concentrations (in mol dm$^{-3}$) of the proton donor and proton acceptor respectively.

Comparing Eq. (7.6) with Eq. (7.1),

$$< \mu_{ab}^2 - \mu_B^2 > \frac{C_A}{C_B} + < \mu_B^2 >$$

$$= \frac{9kT}{4\pi N_A} \left( \frac{\frac{1}{\epsilon_D} \frac{1}{\epsilon_B} (2n_D + n_B^2)}{\epsilon_D (n_B^2 + 2)^2} \right) \frac{n_D (\epsilon_S - n_D^2 (2\epsilon_S + n_D^2))}{\epsilon_S (n_D^2 + 2)^2}$$

$$= \Omega_B$$  \hspace{1cm} (7.7)$$

Hence from the experimentally observed values of $\epsilon_D, n_D, \epsilon_S$ and $n_D$, it is possible to obtain $< \mu_B^2 >$ and $< \mu_{ab}^2 >$ by finding the slope and intercept of the
straight line between $\Omega_B$ and $\left(\frac{C_A}{C_B}\right)$. If the formal concentration of the proton donor is far greater than the proton acceptor such that

$$C_A - C_B >> K^{-1},$$

then according to Huyskens et al. [13], Eq. (7.7) could be written as

$$\left[\mu_{ab}^2 - i\mu_b^2\right] \frac{C_B}{C_A} + \mu_a^2 = \Omega_A \quad (7.8)$$

Here, $i$ represents the order of the complex. If the plot of $\Omega_A$ with $\left(\frac{C_B}{C_A}\right)$ is a straight line in the entire concentration range studied, it can be assumed that a 1:1 complex is formed and hence $i$ can be taken as 1 in Eq. (7.8). The dipole moment of the donor can be obtained from the intercept of the plot of $\Omega_A$ vs $\left(\frac{C_B}{C_A}\right)$ and $\mu_{ab}^2$ can be determined from the slope.

In general, it can be taken that if higher order complexes like $AB_2, A_2B, etc.$, are present, then the plot is no longer a straight line and curved upwards. This serves as a criterion for choosing the proper concentration $C_A$ and $C_B$ of the proton donor and proton acceptor for 1:1 complexation.

7.2.2 Computation of the dipolar increments of complexes

When the proton donor with dipole moment $\mu_a$ form a H-bond with a proton acceptor of dipole moment $\mu_b$, the direction of $\mu_a$ and $\mu_b$ with respect to A- H…. B axis can be defined as $\theta_a$ and $\theta_b$ respectively. If $\theta_a$ and $\theta_b$ differ from zero, one can define the azimuthal angle which describes the rotation position of $\mu_b$ around the hydrogen bond with respect to the plane formed by this bond and $\mu_a$ (Fig. 7.1). The formation of the H-bond is accompanied by the dipole moment of the nuclei involved and the displacement of the electron. As a consequence, the dipole vector of the complex is different from that of the vector sum of the components. If a single H-bond is formed between the partners, it can be presumed that $\Delta \mu$, the dipolar increment due to hydrogen bond has the direction of the hydrogen bond and can be given by [7]
\[ \Delta \mu = \left[ \mu_{ab}^2 - \mu_a^2 \sin^2 \theta_a - \mu_b^2 \sin^2 \theta_b - 2 \mu_a \mu_b \sin \theta_a \sin \theta_b < \cos \varphi > \right]^{\frac{1}{2}} \]

\[ -\mu_a \cos \theta_a - \mu_b \cos \theta_b \]

(7.9)

Fig. 7.1 Dipole moments $\mu_{ab}$, $\mu_a$ and $\mu_b$ of the solution and the dipolar increment $\Delta \mu$.

In the liquid phase complexes, the possibilities of mutual orientations of partners are far numerous than in the solid phase and hence fixation of the correct value of $\varphi$ for a system is rather difficult. However, the non-linear least square regression studies of the dipolar increments and $-\Delta H$ with various assumed values of $\theta_b$ and $< \cos \varphi >$ for a given value of $\theta_a$ by finding the slope for the mean line show that $< \cos \varphi >$ is -0.5 for O – H …O bonds. The mean values of $< \cos \varphi >$ were found to be zero for O – H … N systems [14].

7.2.3 Bond angles of the complexes

It is taken that H-bond formed between a proton donor and an acceptor is linear and is along the lone pair direction of the proton acceptor. It is highly probable that the molecular geometries undergo changes on complexation. From the study of crystal structures of about 200 systems of O-H…O, Kroon et al. [15] showed that there is flexibility in the direction of the lone pair. There are evidences in the literature that
the generalized hybridization theory [16, 17] is not strictly valid [18] especially when conformational strains are dominant due to steric influences.

The calculation of the energy dependence of H-bond on the distance between the molecule and on the angle between the functional group by Pople et al. [19, 20] and Schuster [21] by CNDO / 2 method throws considerable light. It has been shown that in structures with the same orientation of the functional groups forming the H-bond the arrangement with the smallest dipole moment has the lowest energy along the lone pair direction and is more stable. In liquid phase complexes the possibilities of mutual orientation of the partners are more numerous than in the solid phase due to the very fast exchange that occurs between the partners of the complexes in a liquid. For a given family of complexes the most probable structure of the complex is the one for which we obtain the most coherent values of the calculated dipole moment when compared with other characteristics of the complexes like $pK_a$ of the donor, or the complexation enthalpy. The most systematic work in this direction was done by Huyskens et al. [14, 22, 23]. They established that for all bonds of a given type (O-H…O, O-H…N) in a given solvent, the dipole increment confines to within a few tenths of a Debye.

$$\Delta\mu = \frac{A(-\Delta H_b) + [B + C(-\Delta H_b)] \exp A_1 + B_1(-\Delta H_b)}{1 + \exp A_1 + B_1(-\Delta H_b)}$$ (7.10)

This equation was tested for more than a hundred O-H…O complexes in cyclohexane [22] and for some sixty O-H…N complexes in CCl$_4$ and in benzene [14]. If $\Delta\mu$ is expressed in Debye and $\Delta H_b$ in KJ mol$^{-1}$, the numerical values of the constants are the following.

1. **For O – H… N bonds**
   \[A = 0.0074, \quad B = 4.41, \quad C = 0.045, \quad A_1 = -7.70, \quad B_1 = 0.172\]

2. **For O – H… O bonds**
   \[A = 0.028, \quad B = 3.20, \quad C = 0.075, \quad A_1 = -6.50, \quad B_1 = 0.085\]
7.3 Scope of the present work

The study of the complexing ability of esters with carboxylic acids in an apolar solvent medium such as benzene assumes significance with reference to the behaviour of acids as proton donors and the dipole moment changes that accompany in such compexation.

This study enables to understand the intermolecular interactions involving H-bonding of the type $OH \cdots O = C$ which is of utmost importance in biology and chemical physics [24]. Thenappan et al. [25] have shown that the dipole moment studies provides information on the type of interaction between a proton donor and a proton acceptor in an apolar medium and describes the nature of the resulting compexation.

Jeffrey et al. [26] and Grant et al. [27] have pointed out that molecular hydrogen bond dynamics helps to understand the conformational changes of molecules due intermolecular interactions. The following ternary systems were subjected to dipolar incremental studies involving Huyskens method [28] to determine dipole moment based on Onsager’s theory [29].

The dipolar incremental studies are carried out for the following systems:

System 1: Pentanoic acid and Ethyl Benzoate in Benzene
System 2: Propionic acid and Ethyl Benzoate in Benzene
System 3: Hexanoic acid and Ethyl Benzoate in Benzene
System 4: Hexanoic acid and Benzyl Benzoate in Benzene
System 5: Propionic acid and Benzyl Benzoate in Benzene
System 6: Pentanoic acid and Benzyl Benzoate in Benzene

The investigation was carried out at a temperature of 303 K.
7.4 Experimental procedure

7.4.1 Purification of Chemicals

BDH AnalR variety of benzene, carboxylic acids and esters were fractionally distilled and the distillate collected and used.

7.4.2 Sample Preparation

Esters were dissolved in Benzene at a concentration of 1 mol/l or 2 mol/l. Then appropriate amount of carboxylic acids were added for the preparation of the ternary solution at mole fractions 0.1 to 0.6. All the dipole moment measurements, the density and viscosity measurements were made at 303 ± 0.1 K as mentioned in earlier chapters.

7.5 Results and discussion

The calculated parameters are given in tables 7.1-7.6. For the systems 2, 3 and 5 (2: Propionic acid and Ethyl Benzoate, 3: Hexanoic acid and Ethyl Benzoate, 5: Propionic acid and Benzyl Benzoate) $\Omega_B$ is plotted against $\frac{c_A}{c_B}$. For the systems 1, 4 and 6 (1: Pentanoic acid and Ethyl Benzoate, 4: Hexanoic acid and Benzyl Benzoate, 6: Pentanoic acid and Benzyl Benzoate) $\Omega_A$ is plotted against $\frac{c_B}{c_A}$. For all the systems approximately straight lines have been obtained (figs. 7.2 - 7.7) and this indicates the formation of 1:1 complex. Similar views have been expressed by many workers [30-32].

Huyskens et al. [13] have carried out similar work on the binary mixtures of triethyl amine with butyric and propionic acids. They obtained straight lines in the case of the systems in which acids are dissolved at high dilution in triethyl amine indicating the formation of 1:1 complex. They also reported the existence of higher order complex and absence of 1:1 complex if amine is dissolved at high dilution in pure acids.

Oszust and Ratajczak [33] had reported the existence of higher order complexes such as $A_2B_3$ in carboxylic acid-primary and secondary amines exhibiting low or negative dipolar increments. For all the investigated systems, our results show negative or low value of dipolar increments and thereby our results are in agreement.
with those reported by Ratajczak et al. for mixture containing acid as one of the components.

From the slope of the line \( \mu_{ab} \) is determined and y-intercept gives the square of the dipole moment of the proton acceptor. Assuming that the dipole moment of the complex is along the axis of the H-bond, we take \( \theta_B = 60^\circ \). \( \theta_A \) is assumed to be 66°. Using the values of y-intercept, slope of the lines, \( \theta_A, \theta_B, \) and \( \mu_a, \Delta \mu \) has been calculated and is given in table 7.7. The \( \Delta \mu \) values are found to be small. It was observed that [34] if charge transfer interaction carrying the charge for the proton donor to proton acceptor takes place, the redistribution will reach the farther end of the molecules resulting in a very large change \( \Delta \mu \) in dipole moment of the complex, usually greater than 10 D. For the systems studied, the small \( \Delta \mu \) values rule out the contribution arising due to ionic structures and may be interpreted as the complexation is due to the polarization effect [29, 30]. The negative \( \Delta \mu \) value is due to the solvent induced medium effect (SIME). Results on similar lines have been reported by Sobhanadri [35], Thenappan et al. [36] and Parthipan et al. [31] for nitrile-ketone, amine-alcohol and anisole-alcohol mixtures respectively. Pan and Effio [37] had pointed out that due to solvent induced medium effect the solvent breaks the average length of the solute and is responsible for the negative and low value of dipolar increment.

For all the systems studied, plot of \( \Omega \) vs ratio of the formal concentration is a straight line for the given range of concentrations. This goes to say that in all the systems 1:1 complex is more prominent. Views on similar lines have been given by Thenappan et al. [38] Negative value of \( \Delta \mu \) indicates the existence of SIME. Our results are in line with those reported by Parthipan et al. [39] and Thenappan et al. [40]. Small value of \( \Delta \mu \) confirms that redistribution of charges by polarization alone is responsible for the complexation.

7.6 Conclusion

Dipolar incremental studies have been carried out on the binary mixtures of esters and caprylic acids at 303 K. For all the systems, the plot of \( \Omega \) against the ratio of the formal concentration is found to be a straight line indicating the formation of 1:1 complex. Low dipolar increment has been obtained for all the studied mixtures.
Table 7.1
Variation of static permittivity ($\varepsilon_m$), permittivity at optical frequency ($\varepsilon_\infty$), density ($\rho$), ratio of formal concentrations $C_B$ with $C_A$ and $\Omega_A$ at 303 K for Ethyl benzoate and Pentanoic acid in benzene

$C_B = 0.9802$ mol/l

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<th>$\varepsilon_m$</th>
<th>$\varepsilon_\infty$</th>
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<th>$C_B/C_A$</th>
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Fig. 7.2 Variation of $\Omega_A$ with $C_B/C_A$ for Ethyl Benzoate and Pentanoic Acid in Benzene

$y = 0.0773x + 2.5921$
Table 7.2

Variation of static permittivity ($\varepsilon_m$), permittivity at optical frequency ($\varepsilon_\infty$), density ($\rho$), ratio of formal concentrations $C_A$ with $C_B$ and $\Omega_B$ at 303 K for Ethyl benzoate and Propionic acid in benzene

$C_B = 2.0239 \text{ mol/l}$

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Fig. 7.3 Variation of $\Omega_B$ with $C_B/C_A$ for Ethyl Benzoate and Propionic Acid in Benzene
Table 7.3

Variation of static permittivity ($\varepsilon_m$), permittivity at optical frequency ($\varepsilon_\infty$), density ($\rho$), ratio of formal concentrations $C_A$ with $C_B$ and $\Omega_B$ at 303 K for Ethyl benzoate and Hexanoic acid in benzene

$C_B = 2.0240$ mol/l

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Fig. 7.4 Variation of $\Omega_B$ with $C_B/C_A$ for Ethyl Benzoate and Hexanoic Acid in Benzene
Table 7.4

Variation of static permittivity ($\varepsilon_m$), permittivity at optical frequency ($\varepsilon_\infty$), density ($\rho$), ratio of formal concentrations $C_B$ with $C_A$ and $\Omega_A$ at 303 K for Benzyl benzoate and Hexanoic acid in benzene

$C_B = 2.0166$ mol/l

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Fig. 7.5 Variation of $\Omega_A$ with $C_B/C_A$ for Benzyl benzoate and Hexanoic Acid in Benzene
Table 7. 5
Variation of static permittivity ($\varepsilon_m$), permittivity at optical frequency ($\varepsilon_\infty$), density ($\rho$), ratio of formal concentrations $C_A$ with $C_B$ and $\Omega_B$ at 303 K for Benzyl benzoate and Propionic acid in benzene

$C_B = 2.0174$ mol/l

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<td>0.1718</td>
<td>3.6707</td>
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<td>0.6366</td>
<td>3.3801</td>
<td>2.3055</td>
<td>0.9758</td>
<td>0.3155</td>
<td>3.8376</td>
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<tr>
<td>0.5589</td>
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<td>2.2989</td>
<td>0.9790</td>
<td>0.2770</td>
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</tr>
</tbody>
</table>
Fig. 7.6 Variation of $\Omega_B$ with $C_B/C_A$ for Benzyl benzoate and Propionic acid in Benzene
Table 7.6

Variation of static permittivity ($\varepsilon_m$), permittivity at optical frequency ($\varepsilon_\infty$), density ($\rho$), ratio of formal concentrations $C_B$ with $C_A$ and $\Omega_A$ at 303 K for Benzyl benzoate and Pentanoic acid in benzene

$C_B = 2.0155$ mol/l

<table>
<thead>
<tr>
<th>$C_A$ (mol/l)</th>
<th>$\varepsilon_m$</th>
<th>$\varepsilon_\infty$</th>
<th>$\rho$ (g/cc)</th>
<th>$C_B/C_A$</th>
<th>$\Omega_A$ $D^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0914</td>
<td>3.3571</td>
<td>2.3311</td>
<td>0.9645</td>
<td>22.0638</td>
<td>3.2397</td>
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<tr>
<td>0.1984</td>
<td>3.3341</td>
<td>2.3177</td>
<td>0.9651</td>
<td>10.1591</td>
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<tr>
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<td>3.2881</td>
<td>2.3110</td>
<td>0.9656</td>
<td>8.7044</td>
<td>2.8476</td>
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<tr>
<td>0.3648</td>
<td>3.2421</td>
<td>2.3086</td>
<td>0.9658</td>
<td>5.5256</td>
<td>2.7543</td>
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<tr>
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<td>4.2897</td>
<td>2.7180</td>
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<tr>
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<td>2.3037</td>
<td>0.9671</td>
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</table>
Fig. 7.7 Variation of $\Omega_A$ with $C_B/C_A$ for Benzyl benzoate and Pentanoic Acid in Benzene
Table 7.7

Variation of Dipole Moments (\( \mu \)) and Dipolar Increments (\( \Delta \mu \))

\[ \theta_a = 66^\circ, \quad \theta_b = 60^\circ \]

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>System</th>
<th>( \mu_a )</th>
<th>( \mu_b )</th>
<th>( \mu_{ab} )</th>
<th>( \Delta \mu )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ethyl Benzoate and Pentanoic acid in Benzene</td>
<td>1.61</td>
<td>1.9700</td>
<td>1.9895</td>
<td>-0.4593</td>
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<tr>
<td>2</td>
<td>Ethyl Benzoate and Propionic acid in Benzene</td>
<td>0.63</td>
<td>1.6609</td>
<td>2.6949</td>
<td>1.2988</td>
</tr>
<tr>
<td>3</td>
<td>Ethyl Benzoate and Hexanoic acid in Benzene</td>
<td>1.13</td>
<td>1.8037</td>
<td>2.4846</td>
<td>0.7074</td>
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<tr>
<td>4</td>
<td>Benzyl Benzoate and Hexanoic acid in Benzene</td>
<td>1.13</td>
<td>1.7232</td>
<td>1.7331</td>
<td>-0.2024</td>
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<tr>
<td>5</td>
<td>Benzyl Benzoate and Propionic acid in Benzene</td>
<td>0.63</td>
<td>1.7582</td>
<td>2.4352</td>
<td>0.9036</td>
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<td>6</td>
<td>Benzyl Benzoate and Pentanoic acid in Benzene</td>
<td>1.61</td>
<td>1.7634</td>
<td>1.7438</td>
<td>-0.5933</td>
</tr>
</tbody>
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REFERENCES


