

# Dielectric Measurements of Aniline and Alcohol Mixtures at 283, 293, 303, and 313 K Using the Time Domain Technique

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Complex dielectric spectra  $\epsilon^*(\omega) = \epsilon' - j\epsilon''$ , in the frequency range 10 MHz to 10 GHz, have been determined using the time domain reflectometry (TDR) technique at various temperatures and different concentrations of alcohols, viz., ethanol, propan-1-ol, butan-1-ol, hexan-1-ol, and heptan-1-ol + aniline binary mixtures. The static dielectric constant ( $\epsilon_0$ ) and relaxation time ( $\tau$ ) have been obtained. The values of  $\epsilon_0$  and  $\tau$  decrease with an increasing percent of aniline in the alcohol.

## 1. Introduction

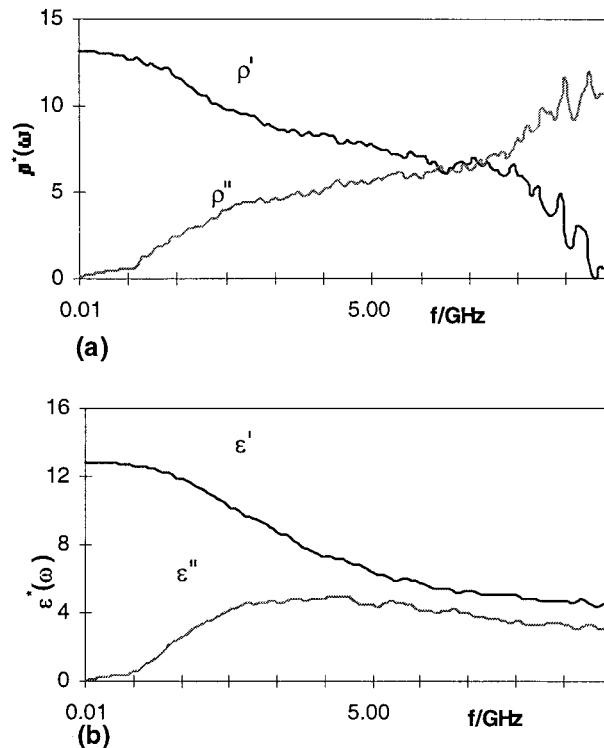
Suryavanshi and Mehrotra (1991) have studied dielectric relaxation of aniline in benzene solution at 10 GHz in the temperature range of 10–50 °C and obtained relaxation time. Garabudu and Swain (1994) have studied aniline with alcohols with a greater number of carbon atoms at 30 °C and at 455 kHz. Fattepur et al. (1994) have studied pure aniline and its mixture with methanol with different concentrations and estimated a strong interaction between the two components in the aniline rich region.

The aim of this paper is to report about the dielectric study of aniline in *n*-alcohols in the frequency range 10 MHz to 10 GHz at different temperatures.

## 2. Experimental Section

Aniline (Glaxo, India Ltd., Bombay), ethanol, hexan-1-ol, heptan-1-ol (spectroscopy grade, Fluka Chemicals Fabrik CH-9470 Buchs), propan-1-ol, Butan-1-ol (E Merck (India) Ltd., Bombay) are obtained commercially and purified by the vacuum distillation method. The solutions were prepared at different volume percentage of aniline ( $\phi_1$ ) in alcohols in steps of 10%, within 0.02% error limit.

The complex permittivity spectrum was studied using TDR (Puranik et al., 1991). A Tektronix 7854 sampling oscilloscope with a 7S12 TDR unit was used. A fast rising step voltage pulse of 200 mV with 25 ps rise time and repetitive frequency of 1 kHz generated by a tunnel diode was propagated through a coaxial line system. The sample was placed at the end of a coaxial cell of 3.5 mm outer diameter and 1.35 mm effective pin length. The change in the pulse after reflection from the sample placed in the cell was monitored. A time window of 5 ns was used. The reflected pulses without sample,  $R_1(t)$ , and with sample,  $R_x(t)$ , were digitized into 1024 points and transferred to



**Figure 1.** (a)  $\rho^*(\omega)$  spectra of aniline + ethanol mixture for  $x_1 = 0.7011$  and  $T = 283$  K. (b)  $\epsilon^*(\omega)$  spectra of aniline + ethanol mixture for  $x_1 = 0.7011$  and  $T = 283$  K.

the computer through GPIB (general purpose interface bus).

The temperature controller system with water bath and a thermostat has been used to maintain the constant temperature within the accuracy limit of  $\pm 1$  °C. The sample cell is surrounded by a heat insulating container through which the water of constant temperature using a temperature controller system is circulated. The temperature at the cell is checked using the electronic thermometer.

## 3. Data Analysis

The time dependent data were processed to obtain complex reflection coefficient spectra  $\rho^*(\omega)$  over the fre-

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**Table 1**

(a) Comparison of Data for the Liquids Used with Literature Values at 293 K				
liquid	$\epsilon_0$		$\rho/\text{g}\cdot\text{cm}^{-3}$	
	this work	lit.	this work	lit.
ethanol	25.41	26.1 <sup>a</sup>	0.789 35	0.789 37 <sup>b</sup>
propan-1-ol	20.02	21.5 <sup>a</sup>	0.803 57	0.803 75 <sup>b</sup>
butan-1-ol	15.97	17.85 <sup>a</sup>	0.810 04	0.809 7 <sup>b</sup>
hexan-1-ol	13.65 (at 298 K)	13.3 <sup>c</sup>	0.814 11	0.819 8 <sup>b</sup>
heptan-1-ol	11.54	11.48 <sup>a</sup>	0.822 11	0.822 3 <sup>b</sup>
aniline	6.55	6.89 <sup>c</sup>	1.020 58	1.021 9 <sup>b</sup>

(b) Observed Density at Different Temperatures in  $\rho/\text{g}\cdot\text{cm}^{-3}$ 

liquid	$\rho/\text{g}\cdot\text{cm}^{-3}$					
	T/K = 283		T/K = 303		T/K = 313	
	calc	lit. <sup>b</sup>	calc	lit. <sup>b</sup>	calc	lit. <sup>b</sup>
ethanol	0.79782	0.7979	0.78074	0.7808	0.77201	0.7721
propan-1-ol	0.81112	0.8116	0.79561	0.7957	0.79809	0.7875
butan-1-ol	0.81798	0.8170	0.80192	0.8022	0.79309	0.7946
hexan-1-ol	0.82138	0.8267	0.81108	0.8127	0.80486	0.8054
heptan-1-ol	0.83372	0.8294	0.81604	0.8150	0.79894	0.8077
aniline	1.02813	1.0304	1.01333	1.0133	1.00601	1.0047

<sup>a</sup> Digest of literature on dielectrics, Vol. 40; National Academy of Sciences: Washington, D.C., 1976. <sup>b</sup> TRC Thermodynamic tables—Non-Hydrocarbons; Thermodynamic Research Center, Texas A&M University System, College Station, TX, extant 1998, d-5000, d-5090, d-5100, d-5100, d-9370. <sup>c</sup> Weast, R. C. *Handbook of Chemistry and Physics*, 64th ed.; CRC Press: Boca Raton, FL, 1983–84.

**Table 2. Temperature Dependent Dielectric Relaxation Parameters for the Aniline + Ethanol Mixture**

$x_1$	T/K = 283	T/K = 293	T/K = 303	T/K = 313	$\epsilon_0$
					0.0000
0.0000	26.43	25.41	24.15	22.6	24.15
					22.6
0.1480	24.7(2)	23.2(7)	22.2(1)	20.8(6)	22.2(1)
					20.8(6)
0.2810	22.2(4)	21.0(6)	20.1(7)	18.9(5)	20.1(7)
					18.9(5)
0.4012	20.0(6)	19.2(4)	17.8(8)	16.7(5)	17.8(8)
					16.7(5)
0.5104	17.6(6)	16.7(5)	15.4(4)	14.2(4)	16.7(5)
					14.2(4)
0.6099	14.8	14.1	13.2	11.9	14.1
					11.9
0.7011	12.7(5)	12.0(2)	11.1(2)	10.2(3)	12.0(2)
					10.2(3)
0.7849	10.5(3)	9.9(2)	9.0(2)	8.5(2)	9.9(2)
					8.5(2)
0.8621	9.0(2)	8.4(2)	7.7(3)	7.1(2)	8.4(2)
					7.1(2)
0.9336	7.9(2)	7.4(1)	6.7(2)	6.4(2)	7.4(1)
					6.4(2)
1.0000	7.0	6.5	6.0	5.7	6.0
$\epsilon_{\infty}$					
0.0000	3.26	3.83	3.06	3.42	3.83
					3.06
0.1480	3.7(0)	4.0(2)	3.1(0)	3.4(2)	4.0(2)
					3.1(0)
0.2810	4.0(1)	3.9(2)	3.5(1)	3.5(2)	3.9(2)
					3.5(1)
0.4012	4.3(2)	3.8(1)	3.0(2)	3.6(2)	3.8(1)
					3.0(2)
0.5104	4.5(2)	3.7(2)	3.2(2)	3.5(2)	3.7(2)
					3.5(2)
0.6099	5.0	3.6	2.9	3.4	3.6
					2.9
0.7011	3.9(2)	3.0(1)	3.1(2)	3.1(3)	3.0(1)
					3.1(3)
0.7849	4.0(1)	2.9(1)	3.0(1)	2.8(2)	2.9(1)
					2.8(2)
0.8621	3.3(2)	2.3(1)	3.0(2)	2.8(1)	2.3(1)
					2.8(1)
0.9336	3.4(1)	2.8(1)	2.5(1)	3.3(1)	2.8(1)
					2.5(1)
1.0000	2.6	2.8	2.5	2.7	2.5
$\tau$ (ps)					
0.0000	211.2	164.7	127.7	108.3	164.7
					127.7
0.1480	253(3)	116(9)	87.9(3)	69.7(4)	116(9)
					87.9(3)
0.2810	125(6)	93.3(7)	66.4(5)	55.2(4)	93.3(7)
					66.4(5)
0.4012	111(9)	82.9(4)	53.9(5)	49.9(4)	82.9(4)
					53.9(5)
0.5104	100(7)	69.2(5)	51.3(4)	44.9(4)	69.2(5)
					51.3(4)
0.6099	84.8	58.94	47.49	40.24	58.94
					47.49
0.7011	63.7(7)	52.4(2)	43.4(3)	33.3(3)	52.4(2)
					43.4(3)
0.7849	46.3(4)	40.4(3)	37.2(4)	26.7(2)	40.4(3)
					37.2(4)
0.8621	34.9(4)	30.5(2)	28.9(5)	21.0(2)	30.5(2)
					28.9(5)
0.9336	27.3(3)	24.3(3)	22.8(4)	16.8(4)	24.3(3)
					22.8(4)
1.0000	22.9	19.4	16.0	13.8	19.4
$g^{\text{eff}}$					
0.0000	3.25	3.23	3.17	3.06	3.23
					3.17
0.1480	3.07	2.98	2.94	2.84	2.98
					2.94
0.2810	2.79	2.73	2.69	2.61	2.73
					2.69
0.4012	2.55	2.52	2.41	2.33	2.52
					2.41
0.5104	2.27	2.22	2.11	1.98	2.22
					2.11
0.6099	1.93	1.90	1.83	1.68	1.90
					1.83
0.7011	1.67	1.62	1.54	1.45	1.62
					1.54
0.7849	1.40	1.35	1.24	1.19	1.35
					1.24
0.8621	1.20	1.13	1.05	0.97	1.13
					1.05
0.9336	1.06	1.00	0.91	0.87	1.00
					0.91

<sup>a</sup>  $x_1$  is the mole fraction of aniline in the mixture. The numbers in parentheses indicate the error in the value, e.g., 24.7(2) means 24.7 ± 0.2.

**Table 3. Temperature Dependent Dielectric Relaxation Parameters for the Aniline + Propan-1-ol Mixture**

$x_1$	T/K = 283	T/K = 293	T/K = 303	T/K = 313
0.0000	21.89	20.0	18.86	17.34
0.1194	19.3(3)	18.2(6)	16.6(1)	15.7(3)
0.2337	16.7(3)	16.0(4)	15.1(7)	14.0(4)
0.3433	14.5(3)	13.8(3)	13.4(8)	12.1(3)
0.4485	12.9(2)	12.3(2)	11.7(4)	10.6(1)
0.5495	11.7	11.2	10.2	9.6
0.6466	10.4(2)	10.0(2)	9.3(2)	8.6(1)
0.7400	9.5(1)	9.032	8.6(2)	7.6(2)
0.8299	8.5(2)	8.101	7.8(3)	7.0(2)
0.9165	7.6(2)	7.181	7.0(2)	6.4(2)
1.0000	7.0	6.55	6.0	5.7
$\epsilon_{\infty}$				
0.0000	3.19	2.89	2.90	3.05
0.1194	3.4(1)	3.5(2)	3.2	

**Table 4. Temperature Dependent Dielectric Relaxation Parameters for the Aniline + Butan-1-ol Mixture**

$x_1$	$T/K = 283$	$T/K = 293$	$T/K = 303$	$T/K = 313$
$\epsilon_0$				
0.0000	17.2	15.97	14.59	12.72
0.0998	14.0(4)	13.1(4)	11.9(2)	11.1(2)
0.1996	11.8(3)	10.9(3)	10.5(3)	9.4(3)
0.2994	10.3(4)	9.7(3)	9.1(3)	8.2(2)
0.3993	9.3(3)	8.8(2)	8.3(2)	7.7(1)
0.4993	8.4	8.3	7.8	7.4
0.5993	8.1(2)	7.8(2)	7.4(2)	6.9(2)
0.6994	7.6(2)	7.3(2)	6.8(2)	6.5(1)
0.7996	7.5(3)	7.0(3)	6.5(2)	6.2(1)
0.8998	7.3(3)	6.8(4)	6.3(1)	5.8(1)
1.0000	7.0	6.5	6.0	5.7
$\epsilon_\infty$				
0.0000	1.9			
0.0998	2.3(1)	2.4(1)	2.3(1)	2.4(1)
0.1996	2.4(1)	2.6(1)	2.6(1)	2.9(1)
0.2994	2.8(1)	2.5(1)	2.6(1)	2.6(1)
0.3993	2.9(1)	2.8(1)	2.7(1)	2.7(1)
0.4993	2.8	2.9	2.8	2.8
0.5993	2.9(1)	2.8(1)	2.7(1)	2.8(1)
0.6994	3.0(1)	1.9(2)	2.9(1)	2.8(1)
0.7996	3.0(1)	1.2(2)	2.9(1)	3.0(1)
0.8998	3.0(1)	0.05(3)	2.9(1)	3.2(1)
1.0000	2.6	2.8	2.5	2.7
$\tau$ (ps)				
0.0000	833.0	519.8	342.3	200.3
0.0998	381.9(29)	257.5(19)	188.3(9)	131.4(7)
0.1996	214.1(15)	149.9(11)	129.9(9)	85.6(7)
0.2994	140.3(12)	114.5(8)	86.9(7)	63.6(3)
0.3993	99.4(8)	76.5(4)	61.1(4)	45.8(3)
0.4993	71.74	56.3	45.6	33.4
0.5993	55.1(3)	45.1(3)	37.8(3)	31.4(3)
0.6994	45.1(3)	37.5(3)	36.6(3)	29.2(3)
0.7996	39.1(5)	32.4(3)	29.3(4)	23.9(3)
0.8998	33.8(5)	27.9(4)	23.5(3)	20.3(3)
1.0000	22.9	19.4	16.0	13.8
$g^{\text{eff}}$				
0.0000	2.99	2.85	2.68	2.38
0.0998	2.36	2.28	2.13	2.02
0.1996	1.94	1.84	1.81	1.66
0.2994	1.65	1.58	1.52	1.39
0.3993	1.44	1.40	1.35	1.26
0.4993	1.27	1.28	1.23	1.18
0.5993	1.19	1.19	1.13	1.08
0.6994	1.09	1.04	1.01	0.95
0.7996	1.06	0.99	0.93	0.89
0.8998	1.01	0.95	0.87	0.79
1.0000	0.95	0.88	0.81	0.76

#### 4. Results and Discussion

The values of the dielectric constant and density, for the pure liquids used, along with literature values are given in Table 1a, and the observed density values are given in Table 1b.

The static dielectric constant, the dielectric constant at higher frequency, and the relaxation time by fitting experimental data in the Havriliak–Negami equation are listed in Tables 2–6. The values of  $\alpha$  and  $\beta$  are taken to be 0 and 1, respectively, for the systems therefore not listed in Tables 2–6.

The static dielectric constant and relaxation time value decrease as the percent of aniline in the alcohol increases. These values also decrease with an increase in temperature, as expected.

The Kirkwood correlation factor  $g$  (Frolich, 1949) is also a parameter for getting information regarding orientation of electric dipoles in polar liquids. The  $g$  for pure liquid

**Table 5. Temperature Dependent Dielectric Relaxation Parameters for Aniline + Hexan-1-ol Mixture**

$x_1$	$T/K = 283$	$T/K = 293$	$T/K = 303$	$T/K = 313$
$\epsilon_0$				
0.0000	14.4	13.65	12.11	11.3
0.0998	11.5(8)	10.8(4)	10.1(6)	9.1(3)
0.1996	9.8(6)	9.2(5)	8.8(4)	8.1(3)
0.2994	8.5(5)	8.1(4)	7.7(3)	7.2(3)
0.3993	7.8(2)	7.6(2)	7.3(2)	7.0(2)
0.4993	7.5	7.3	7.0	6.5
0.5993	7.4(2)	7.0(1)	6.6(0)	6.4(1)
0.6994	7.2(2)	6.8(2)	6.6(1)	6.1(1)
0.7996	7.3(1)	7.0(1)	6.7(2)	6.4(2)
0.8998	7.1(2)	6.8(2)	6.4(0)	6.1(1)
1.0000	7.0	6.5	6.0	5.7
$\epsilon_\infty$				
0.0000	2.34	2.32	2.6	2.24
0.0998	2.6(1)	2.7(1)	3.0(2)	2.9(1)
0.1996	2.7(1)	2.8(1)	3.1(2)	3.1(1)
0.2994	2.8(1)	2.9(1)	3.0(1)	3.0(1)
0.3993	2.9(0)	3.1(0)	3.0(0)	3.1(1)
0.4993	2.9	2.9	2.9	2.8
0.5993	3.0(0)	3.0(0)	2.9(0)	2.9(1)
0.6994	2.9(1)	3.0(1)	3.0(0)	2.9(0)
0.7996	3.4(1)	3.4(1)	2.9(1)	2.4(2)
0.8998	3.0(2)	2.3(2)	2.8(0)	2.4(1)
1.0000	2.6	2.8	2.5	2.7
$\tau$ (ps)				
0.0000	1209.0	1070.0	704.0	408.0
0.0998	545.0(11)	415.0(5)	313.0(30)	192.0(20)
0.1996	324.0(6)	239.0(4)	195.0(30)	125.0(10)
0.2994	195.0(3)	138.0(2)	115.0(10)	89.7(10)
0.3993	120.0(1)	95.9(8)	80.3(8)	63.0(7)
0.4993	82.90	70.3	56.9	40.2
0.5993	65.9(6)	52.9(3)	46.2(2)	39.9(3)
0.6994	51.7(6)	49.8(3)	36.5(4)	29.8(3)
0.7996	45.4(4)	25.7(3)	24.7(4)	17.7(4)
0.8998	29.7(3)	22.0(3)	20.7(2)	13.9(3)
1.0000	22.9	19.4	16.0	13.8
$g^{\text{eff}}$				
0.0000	3.21	3.13	2.84	2.71
0.0998	2.42	2.37	2.24	2.07
0.1996	1.93	1.88	1.77	1.70
0.2994	1.58	1.54	1.49	1.42
0.3993	1.37	1.36	1.35	1.28
0.4993	1.24	1.26	1.21	1.15
0.5993	1.18	1.15	1.09	1.07
0.6994	1.10	1.06	1.04	0.96
0.7996	1.05	1.01	0.99	0.92
0.8998	1.00	0.97	0.92	0.87
1.0000	0.95	0.88	0.81	0.76

may be obtained by the expression

$$\frac{4\pi N \mu^2 \rho}{9kTM} g = \frac{(\epsilon_0 - \epsilon_\infty)(2\epsilon_0 + \epsilon_\infty)}{\epsilon_0(\epsilon_\infty + 2)^2} \quad (3)$$

where  $\mu$  is the dipole moment in the gas phase,  $\rho$  is the density at temperature  $T$ ,  $M$  is the molecular weight,  $k$  is the Boltzmann constant, and  $N$  is the Avogadro's number.

For the mixture of two polar liquids 1 and 2, eq 3 is modified (Kumbharkhane et al., 1993) with the following assumption:

Assume that, for the mixture,  $g^{\text{eff}}$  is effective correlation factor in the mixture. The Kirkwood equation for the mixture may be expressed as

$$\frac{4\pi N}{9kT} \left[ \frac{\mu_1^2 \rho_1}{M_1} \phi_1 + \frac{\mu_2^2 \rho_2}{M_2} \phi_2 \right] g^{\text{eff}} = \frac{(\epsilon_{0m} - \epsilon_{\infty m})(2\epsilon_{0m} + \epsilon_{\infty m})}{\epsilon_{0m}(\epsilon_{0m} + 2)^2} \quad (4)$$

The Kirkwood correlation factors,  $g^{\text{eff}}$ , which gives angular correlation between the molecules of the system, are

**Table 6. Temperature Dependent Dielectric Relaxation Parameters for the Aniline + Heptan-1-ol Mixture**

$x_1$	T/K = 283	T/K = 293	T/K = 303	T/K = 313
$\epsilon_0$				
0.0000	12.64	11.5	10.75	9.69
0.0669	9.9(6)	8.9(4)	8.5(4)	7.8(2)
0.1390	8.4(5)	8.1(5)	7.7(4)	7.2(3)
0.2167	7.6(3)	7.4(4)	7.1(3)	6.7(2)
0.3009	7.1(2)	6.9(3)	6.7(2)	6.4(1)
0.3923	6.9	6.7	6.5	6.1
0.4920	6.9(3)	6.5(1)	6.3(1)	5.9(2)
0.6010	6.9(2)	6.4(2)	6.2(2)	5.8(1)
0.7208	6.9(2)	6.4(2)	6.1(2)	5.7(1)
0.8532	7.0(2)	6.3(2)	6.1(1)	5.7(1)
1.0000	7.0	6.5	6.0	5.7
$\epsilon_\infty$				
0.0000	2.4	2.2	2.4	2.7
0.0669	2.7(2)	2.6(1)	2.8(1)	2.9(1)
0.1390	2.6(1)	2.8(2)	2.9(1)	3.0(1)
0.2167	2.7(1)	3.0(1)	3.0(1)	3.1(1)
0.3009	2.9(0)	3.0(1)	3.1(1)	3.3(0)
0.3923	2.9	3.1	3.1	3.3
0.4920	3.1(1)	3.2(1)	3.1(0)	3.3(0)
0.6010	3.2(1)	3.3(1)	3.0(1)	3.3(0)
0.7208	3.4(1)	3.4(1)	3.2(1)	3.1(1)
0.8532	3.0(1)	3.4(1)	2.9(1)	3.0(1)
1.0000	2.6	2.8	2.5	2.7
$\tau$ (ps)				
0.0000	1640.0	1280.0	914.0	575.0
0.0669	629.0(13)	454.8(74)	359.0(50)	248.1(26)
0.1390	344.2(17)	295.8(65)	217.2(39)	158.8(21)
0.2167	194.5(28)	171.8(32)	129.5(17)	90.7(9)
0.3009	135.2(11)	115.6(50)	98.3(10)	73.3(7)
0.3923	104.10	86.7	74.5	52.2
0.4920	88.0(11)	75.7(7)	58.2(4)	44.9(7)
0.6010	53.9(6)	49.8(8)	42.3(6)	37.4(5)
0.7208	49.2(7)	43.7(9)	33.0(5)	24.9(3)
0.8532	32.1(4)	30.5(6)	25.1(2)	18.9(3)
1.0000	22.9	19.4	16.0	13.8
$g^{\text{eff}}$				
0.0000	3.06	2.86	2.73	2.50
0.0669	2.11	2.03	1.98	1.84
0.1390	1.74	1.72	1.68	1.59
0.2167	1.46	1.47	1.43	1.39
0.3009	1.28	1.27	1.27	1.23
0.3923	1.17	1.17	1.15	1.09
0.4920	1.11	1.06	1.05	0.98
0.6010	1.06	1.00	0.98	0.91
0.7208	1.02	0.94	0.92	0.86
0.8532	0.99	0.89	0.86	0.81
1.0000	0.95	0.88	0.81	0.76

tabulated in Tables 2–6. The values of  $g^{\text{eff}}$  decrease, for all the systems, as the percent of aniline in the aniline + alcohol mixture increases at all temperatures. The values

of  $g^{\text{eff}}$  for aniline are less than 1, indicating antiparallel orientation of the electric dipole.

## 5. Conclusion

Dielectric relaxation parameters and the Kirkwood correlation factor have been reported for aniline + alcohol mixtures for various concentrations and temperatures. The dielectric constant and relaxation time decrease with an increase in the percent of aniline in the alcohol and also with temperature.

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