

DIELECTRIC ABSORPTION STUDIES IN SOME α -SUBSTITUTED BENZYL CYANIDES IN DILUTE SOLUTION

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Dielectric absorption studies on four α -substituted benzyl cyanides, namely α -butyl benzyl cyanide, α -heptyl benzyl cyanide & α -cyano stilbene have been made. The observed α values being sufficiently high, indicate the existence of more than a single relaxation process occurring in the system. When further resolved, the dielectric dispersion yielded relaxation time $\tau(1)$ and $\tau(2)$ which are sufficiently different from one another, showing the presence of the molecular and some other process. In some cases the enthalpies of activation and the other energy parameters have also been evaluated. It has been concluded that the dielectric relaxation takes place due to both molecular and group processes except for the case of α -benzyl benzyl cyanide, where the enthalpy of activation for the group process has been found to be very low.

Introduction

Dielectric absorption studies are very helpful in probing the molecular flexibility or rigidity which is of vital importance for the investigations on carcinogenic activity. FISCHER and his collaborators [1] studied benzyl chloride for its dielectric behaviour and concluded that the nonrigidity is an important feature of the molecule. PURCEL et al. [2] studied the dielectric relaxation of benzyl chloride in the dilute solution of benzene and analysed the data in terms of the two relaxation times; Cole-Cole's molecular relaxation time τ_1 (=23.7 ps) and the group relaxation time τ_2 (=2.3 ps). KLAGES and KNOBLOCH [3] investigated for dielectric behaviour, some substituted benzyl halides including benzyl chloride for which they obtained τ_1 =14.6 ps and τ_2 =1.6 ps with the contribution due to the group relaxation (C_2 =0.3). FOREST and SMYTH [4] investigated benzyl cyanide in dilute solution and below 40° C obtained two relaxation times. HASSAN et al. [5] studied microwave absorption in some benzyl derivatives *i.e.* $C_6H_5CH_2X$ ($X = Cl, Br, CN$ and NH_2) in pure liquid state and analysed the data in terms of the two relaxation processes. For benzyl cyanide the dielectric data of FOREST and SMYTH [4] in the dilute solution of benzene and HASSAN et al. [5] in the pure liquid state above 40° C give very surprising results by showing the absence of group rotation.

Due to the interesting results obtained on benzyl cyanide the authors in the present work have chosen four higher homologues of α -substituted benzyl cyanides

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namely α -butyl benzyl cyanide, α -heptyl benzyl cyanide, α -benzyl benzyl cyanide and α -cyano stilbene for the dielectric and thermodynamic investigations in the dilute solutions of benzene. The permittivity data have been analysed by GOPALA KRISHNA [6], HIGASI [7], KOGA and NAKAMURA [8] methods. The results show that at x-band nonrigidity is an important feature of the molecules under investigation over the range of temperature (288–320° K) investigated.

Experimental: The dielectric constant (ϵ') and the dielectric loss (ϵ'') of the dilute solutions of a polar solute in the nonpolar solvent have been measured over a range of temperature at x-band described elsewhere [9] by a technique due to ROBERTS and VON HIPPEL [10] and later modified by DAKIN and WORKS [11]. The static dielectric constant ϵ_0 of solutions have been measured at 300 KHz by a dipole meter based on the principle of heterodynebeat method. The refractive indices (Na light) of all the solutions have been measured by an Abbe's Refractometer. The measurements for ϵ' & ϵ'' are accurate up to $\pm 2\%$ and $\pm 5\%$ respectively.

Materials: The chemicals have been obtained from the Central Drug Research Institute, Lucknow, India and the solvent (A.R. grade) has been obtained from E. Merck (Germany). The chemicals have been distilled twice before use.

Method: The permittivity data have been analysed by the GOPALA KRISHNA method [6] for the evaluation of the dielectric relaxation time (τ_{GK}) using the equations

$$X = P + Y/\omega\tau_{GK} \quad (1)$$

where $P = \left(\frac{\epsilon_\infty - 1}{\epsilon_\infty + 2} \right)$ has been assumed to be constant for the dilute solutions;

$$W = \frac{2\pi C}{\lambda} \quad \text{and}$$

$$\left. \begin{aligned} X &= \frac{\epsilon'^2 + \epsilon''^2 + \epsilon' - 2}{(\epsilon' + 2)^2 + \epsilon''^2} \\ Y &= \frac{3\epsilon''}{(\epsilon' + 2)^2 + \epsilon''^2} \end{aligned} \right\} \quad (2)$$

The slope $\left(\frac{dy}{dx} \right)$ of equation (1) gives the relaxation time

$$\text{as } \tau_{GK} = \frac{\lambda}{2\pi C} \frac{dy}{dx}; \quad (3)$$

The most probable relaxation time τ_{OH} and the distribution parameter (α) have also been determined by HIGASI method [7] using the equations

$$\tau_{OH} = \frac{1}{\omega} \left[\frac{A^2 + B^2}{C^2} \right]^{\frac{1}{2(1-\alpha)}} \quad (4)$$

$$1 - \alpha = \frac{2}{\pi} \tau_{an}^{-1} \left(\frac{A}{B} \right)$$

where

$$\left. \begin{aligned} A &= a''(a_0 - a_\infty) \\ B &= (a' - a_\infty)(a_0 - a') - a''^2 \\ C &= (a' - a_\infty)^2 + a''^2 \end{aligned} \right\} \quad (5)$$

The quantities a_0 , a' , a'' and a_∞ are the slopes of the straight line plots of the respective dielectric quantity ϵ_0 , ϵ' , ϵ'' and $\epsilon_\infty = n_D^2$ against the concentrations in the dilute solution range and are defined as

$$\epsilon_0 = \epsilon_{01} + a_0 W_2$$

$$\epsilon' = \epsilon'_{1} + a' W_2$$

$$\epsilon'' = a'' W_2$$

$$\epsilon_\infty = \epsilon_{1\infty} + a_\infty W_2$$

in which subscript 1 refers to the pure solvent and O to the static frequency, ∞ to the high frequency or optical frequency measurements, W_2 is the weight fraction of the solute.

The absorption processes have further been resolved by the recent HIGASI, KOGA and NAKAMURA method [8] in terms of two independent Debye type relaxation times $\tau_{(1)}$ and $\tau_{(2)}$ defined by the equation

$$\tau_{(1)} = \frac{a''}{\omega(a' - a_\infty)} \quad (7)$$

$$\tau_{(2)} = \frac{1}{\omega} \left[\frac{a_0 - a'}{a''} \right]. \quad (8)$$

The slopes a_0 , a' , a_∞ and a'' are defined by the set of equations (6).

It has been found [12-14] that the relaxation time $\tau_{(2)}$ leads to that for the Cole-Cole's molecular relaxation process τ_1 and $\tau_{(1)}$ has been found to be an implicit function of group relaxation time τ_2 , τ_1 and weight factor C_2 for the group rotation process; any difference beyond experimental error in the value of $\tau_{(1)}$ and $\tau_{(2)}$ for a molecule is indicative of more than one relaxation process present in the system.

The activation energy parameters for the dipole relaxation process have been estimated using Eyring's theory of rate process [15]. The enthalpy of activation corresponding to the relaxation time τ_{GK} , τ_{OH} , $\tau_{(1)}$, $\tau_{(2)}$ and $\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$ have been estimated from the slopes of plots of $\log \tau_{GK}T$, $\log \tau_{OH}T$, $\log \tau_{(1)}$, T , $\log \tau_{(2)}$, T and $\log \tau_0T$ against $-1/T$ respectively.

Results and Discussion: The slopes of the dielectric quantities *vs* concentration plots *i.e.* a_0 , a' , a'' and a_∞ at different temperatures of the molecule under investigation are reported in Table I. The relaxation time $\tau_{(1)}$, $\tau_{(2)}$, $\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$, τ_{GK} , τ_{OH} and the distribution parameter of the molecules at different temperatures are presented in Table II. The enthalpies of activation $\Delta H\tau_{(1)}$, $\Delta H\tau_{(2)}$, $\Delta H\tau_0$, $\Delta H\tau_{GK}$ and $\Delta H\tau_{OH}$ corresponding to the relaxation time $\tau_{(1)}$, $\tau_{(2)}$, τ_0 , τ_{GK} and τ_{OH} have been tabulated in Table III. In Table IV, the activation parameters for the dipole reorientations associated with the relaxation times τ_{GK} and τ_{OH} have been compared:

α -Butyl Benzyl Cyanide (A)

The examination of Table II. shows that the molecule has an appreciable value of the distribution parameter ranging from (0.09–0.22) in the temperature range of (288–320° K) and the most probable relaxation time τ_{OH} evaluated using Higasi method for the molecule has been found to be 23.5 p.s. at 288° K. The occurrence of large distribution parameter in the molecular system indicates the non-rigidity of the molecule in the microwave region under investigation. The dielectric dispersion, was therefore, further resolved in terms of two independent Debye type relaxation times $\tau_{(1)}$ and $\tau_{(2)}$. It is found that the molecular relaxation time $\tau_{(2)}$ is 30.0 p.s. and $\tau_{(1)}$ associated with the relaxation process other than the molecular

Table I

The slopes a_0, a', a'', a_∞ and dipole moment of molecules in dilute solution of Benzene

Compound	Temp. K	a_0	a'	a''	a_∞
α -Butyl benzyl cyanide (A)	288	14.0	5.00	5.50	0.58
	296	12.5	4.75	4.76	0.40
	304	11.1	4.50	3.90	0.39
	312	8.5	4.00	3.12	0.37
	320	6.5	3.50	2.63	0.30
α -Heptyl benzyl cyanide (B)	296	18.0	5.00	5.71	0.28
	304	14.0	4.70	4.70	0.25
	312	12.05	4.00	4.44	0.15
	320	10.00	3.33	4.00	0.75
α -Benzyl benzyl cyanide (C)	288	16.00	3.13	3.80	0.75
	296	12.00	2.77	3.00	0.62
	304	10.50	2.50	2.66	0.54
	312	9.3	2.20	2.50	0.32
α -Cyano stilbene (D)	296	12.50	3.60	3.10	0.48
	304	11.80	3.30	2.90	0.35
	312	8.50	2.80	2.40	0.25
	320	6.15	2.18	1.90	0.08

is 18.4 p.s. at 288° K. An appreciable difference between $\tau_{(1)}$ and $\tau_{(2)}$ supports the presence of two independent relaxation processes occurring in the molecule. The longer relaxation time (30 p.s.) would correspond to the molecular rotation of butyl benzyl cyanide whereas the second process would be expected to be due to

the internal rotation of $\begin{array}{c} \text{C}_4\text{H}_9 \\ | \\ \text{---C---C}\equiv\text{N} \\ | \\ \text{H} \end{array}$ group. Similar conclusions have been made

by MISRA and SAXENA [16] on some substituted benzaldehydes.

A comparison of τ_{OH} (Higasi) and $\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$ (Higasi, Koga and Nakamura) shows a close agreement, as both represent the most probable (average) relaxation time parameters. A close agreement between τ_{OH} and $\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$ observed by MISRA et al. [17] on some substituted amides in dilute solution supports

Table II

Relaxation times of the molecules using Higasi, Higasi, Koga and Nakamura method and Gopala Krishna Method

Temp. K	Higasi Method		Higasi, Koga & Nakamura Method			Gopala Krishna Method
	τ_{OH} ps	α	$\tau_{(1)}$ ps	$\tau_{(2)}$ ps	$\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$ ps	τ_{GK} ps
α -Butyl Benzyl cyanide (A)						
288	23.5	0.09	18.9	30.0	23.8	26.6
296	22.4	0.12	16.7	27.1	21.3	24.4
304	22.2	0.20	14.5	25.8	19.3	22.5
312	17.8	0.17	13.1	22.6	17.2	20.7
320	15.4	0.20	12.5	19.0	15.4	17.5
α -Heptyl benzyl cyanide (B)						
296	33.2	0.18	18.4	34.4	25.2	30.2
304	27.3	0.20	16.1	30.1	22.0	25.5
312	25.4	0.14	17.6	27.6	22.0	22.1
320	24.4	0.12	14.3	26.5	19.4	20.7
α -Benzyl Benzyl cyanide (C)						
288	56.7	0.15	24.3	51.6	35.4	26.6
296	53.4	0.17	21.3	45.7	31.2	20.4
304	51.6	0.21	20.6	45.2	30.5	19.0
312	46.3	0.15	20.3	45.0	30.2	17.7
α -Cyano stilbene (D)						
296	44.2	0.29	15.0	43.7	25.7	22.1
304	41.1	0.29	14.9	42.0	25.0	19.0
312	33.0	0.27	14.3	36.2	22.7	16.6
320	30.0	0.25	13.8	31.9	21.0	14.7

Table III

Activation energies calculated for the relaxation times $\tau(1)$, $\tau(2)$, τ_0 , τ_{OH} and τ_{GK} (in kJ mol^{-1})

Compound	$\Delta H \tau(1)$	$\Delta H \tau(2)$	$\Delta H \tau_0$	$\Delta H \tau_{OH}$	$\Delta H \tau_{GK}$
α -Butyl Benzyl cyanide (A)	5.3	8.0	6.4	6.4	5.6
α -Heptyl Benzyl cyanide (B)	—	—	—	8.0	8.0
α -Benzyl Benzyl cyanide (C)	—	—	—	5.1	4.8
α -Cyano Stilbene (D)	1.7	11.9	6.4	10.6	9.6

Table IV

Relaxation times, free energy of activation, energy, enthalpy, and entropy of activation by Higasi and Gopala Krishna method

Compound	Temp. K	τ_{OH} ps	τ_{GK} ps	Using Higasi parameters			Using G. K. parameters		
				ΔF_e kJ mol ⁻¹	ΔH_e kJ mol ⁻¹	ΔS_e J/deg ⁻¹ mol ⁻¹	ΔF_e kJ mol ⁻¹	ΔH_e kJ mol ⁻¹	ΔS_e J/deg ⁻¹ mol ⁻¹
α -Butyl Benzyl cyanide (A)	288	23.5	26.6	11.7		-18.4	12.1		-22.5
	296	22.4	24.4	12.1		-19.2	12.1		-21.7
	304	22.2	22.5	12.1	6.4	-18.8	12.1	5.6	-20.1
	312	17.8	20.7	12.1		-18.4	12.5		-22.2
	320	15.4	17.5	12.1		-17.9	12.5		-21.7
α -Heptyl Benzyl cyanide (B)	288	—	30.2	13.0		—	12.5		-15.8
	296	33.2	25.5	13.0		-16.7	12.5		-15.0
	304	27.3	22.1	13.4	8.0	-16.3	12.5	8.0	-14.6
	312	25.4	21.0	13.4		-17.1	12.5		-14.6
	320	24.4	20.8	13.8		-16.7	13.0		-15.85
α -Benzyl Benzyl cyanide (C)	288	56.7	26.6	13.8		-30.1	12.1		-25.4
	304	53.4	20.4	14.2		-30.9	12.1		-24.2
	312	51.6	19.0	14.6	5.1	-31.8	12.5	4.8	-24.6
	329	45.3	17.7	14.6		-30.9	12.5		-24.2
α -Cyano Stilbene (D)	296	44.2	22.1	13.8		-10.4	11.7		-7.1
	304	44.1	19.0	13.8		-10.4	12.1		-7.9
	312	33.0	16.6	13.8	10.6	-10.0	12.1	9.6	-8.1
	320	30.0	14.8	14.2		-11.3	12.1		-8.2

the present observations. Also τ_{GK} (26.6 p.s.) has been found to be slightly longer than τ_0 (23.8 p.s.) or τ_{OH} (23.5) and comparatively smaller than $\tau(2)$ the relaxation time considered to be for molecular rotation. This is due to the fact that τ_{GK} gives an average of the different relaxation mechanisms involved in the system and thus has a smaller value.

The enthalpies of activations (ΔH) associated with the different modes of rotations are given in Table III $\Delta H \tau_{(1)}$ and $\Delta H \tau_{(2)}$ for **A** have been found to be 5.3 and 8.0 kJ mol⁻¹ respectively. These two values are respectively due to the group and the molecular relaxation processes. The enthalpies of activation $\Delta H \tau_{OH}$ and $\Delta H \tau_0$ 6.4 kJ mol⁻¹ corresponding to Higasi and the average relaxation due to Higasi *et al.* have been found to be in very close agreement; where as that due to Gopala Krishna relation, $\Delta H \tau_{GK}$ is slightly different from the former two values.

An examination of Table IV. shows that the free energies of activation ΔF_e in the range of temperature (288°–320° K) evaluated, using τ_{GK} and τ_{OH} parameters are comparable. The entropy values have been found to be negative suggesting the presence of cooperative orientations [18] resulting from the steric forces.

α -Heptyl Benzyl Cyanide (B)

For this molecule also appreciable value of the distribution parameter α (0.12–0.20) has been observed. The value of the most probable relaxation time τ_{OH} has been found to be 33.2 p.s. at 296° K. The appreciably high value of the distribution parameter suggests the presence of more than one relaxation process. Absorption corresponding to these processes have thus further been resolved in terms of two independent Debye type dispersions defined by $\tau_{(1)}$ and $\tau_{(2)}$ using Higasi, Koga & Nakamura [8]. The two observed relaxation times are sufficiently different from one another suggesting further the probable flexibility of the molecule in the microwave region under investigation. As can be seen from Table II., the resolved relaxation times $\tau_{(1)}$ and $\tau_{(2)}$ at 296° K are found to be 18.4 and 34 p.s. respectively. The first process, having smaller relaxation time, would be assigned to the internal rotation of the group; whereas the second process, having longer relaxation time, would be expected to be due to the molecular rotation. τ_{GK} (30.2 p.s.) has been found to be smaller than that for molecular process $\tau_{(2)}$ (34.4 p.s.). Also the other two average processes giving rise to the most probable relaxation times at 296° K *i.e.* τ_{OH} (33.2 p.s.) and τ_0 (25.2 p.s.) are comparatively smaller than that for the molecular process. However these values are not in very good agreement to one another. The difference can not be ruled out within the experimental errors and also because of broad dispersions associated with the above two processes.

A comparison of $\tau_{(1)}$ and $\tau_{(2)}$ of B with that of the corresponding values of A shows that $\tau_{(2)}$ (=34.4 p.s.) for this molecule is longer than $\tau_{(2)}$ (=30.0 p.s.) for the previous one, which is smaller in molecular size, showing that the process occurring is due to the molecular rotation. The second process, which could be associated to the internal rotation of the group has no significant variation from the corresponding process in A.

In this case also, an attempt to evaluate different enthalpies of activation was made. However, due to irregular variations, the evaluation of $\Delta H\tau_{(1)}$ and $\Delta H\tau_{(2)}$ could not be done properly. The enthalpies, using Gopala Krishna and Higasi relaxation time values were however calculated and the two parameters $\Delta H\tau_{GK}$ (8.00 kJ mol⁻¹) and $\Delta H\tau_{OH}$ (8.00 kJ mol⁻¹), are in very good agreement.

Entropy values for the molecules are found to be negative suggesting the presence of the cooperative process [18].

 α -Benzyl Benzyl Cyanide (C)

Having obtained the distribution parameter α in the range of (0.15–0.21) and the most probable relaxation time τ_{OH} as 56.7 p.s. at 288° K, the absorption process has been further resolved by Higasi, Koga and Nakamura method [8] in terms of the two independent relaxation times and the parameters observed were $\tau_{(1)}$ (=25.6 p.s.) and $\tau_{(2)}$ (=51.6 p.s.) respectively at 288° K. The relaxation time for the overlapped process has been calculated as $\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$ and been found to be 35.4 p.s. at 288° K.

The dielectric relaxation time associated with the molecular process $\tau_{(2)}$ was 51.6 p.s. whereas that associated with the internal rotation was $\tau_{(1)} = 25.6$ p.s. A comparison of the relaxation time values obtained by Higasi's τ_{OH} 56.7 p.s. and

Gopala Krishna's τ_{GK} 26.6 p.s. method show that τ_{OH} is predominantly due to the molecular relaxation process whereas τ_{GK} corresponds to the other process probably arising from the internal rotation of the group. Also $\tau_{OH} > 2\tau_{GK}$, which is similar to the behaviour observed by MATHUR et al. [19] in the case of some substituted anilines.

On comparing the relaxation time of this molecule with the relaxation time of α -Heptyl benzyl cyanide it is found that τ_{OH} , $\tau_{(1)}$, $\tau_{(2)}$ and $\tau_0 = \sqrt{\tau_{(1)}\tau_{(2)}}$ for the former molecule are longer than for the latter molecules whereas in both the molecules the substitution at α -position has the same number of carbon atoms but with different spatial configuration. It is interesting to point out here that in α -Heptyl benzyl cyanide the substituted heptyl group has seven carbon atoms arranged in a linear chain whereas in α -benzyl benzyl cyanide the substituted benzyl group at α -position has also seven carbon atoms but arranged in a cyclic form. The difference in the relaxation time in these molecules may be attributed to the difference in the geometry of the substituted group.

The enthalpy parameters in response to the relaxation processes $\tau_{(1)}$ and $\tau_{(2)}$ could not be ascertained due to some irregular variation in these parameters with respect to the temperature. The other two enthalpy parameters corresponding to τ_{OH} and τ_{GK} are not very accurate. However a comparison shows that $\Delta H\tau_{OH} > \Delta H\tau_{GK}$.

For this molecule also the entropy of activation has been found to be negative. The free energy of activation evaluated using τ_{GK} and τ_{OH} shown in Table IV, and are different being greater for τ_{OH} than for τ_{GK} .

α -Cyano Stilbene (D)

This molecule gives the distribution parameter α — in the range of (0.29–0.25) and this value decreases with the increase of temperature. τ_{OH} for the molecule has been found to be 44.2 p.s. at 296° K. Due to the high value of α the dielectric dispersion has been resolved in terms of two independent relaxation times $\tau_{(1)}$ and $\tau_{(2)}$. The average relaxation time τ_0 obtained as the geometrical mean of $\tau_{(1)}$ and $\tau_{(2)}$ has been found to be 25.7 p.s. at 296° K.

On comparing the relaxation times evaluated using Gopala Krishna method and Higasi method it is found that $\tau_{OH} > \tau_{GK}$. Similar observations have been made by KRISHNAJI et al. [20]. Lower value of τ_{GK} as compared to τ_{OH} is probably due to the fact that in the evaluation of τ_{GK} it has been assumed that the molecule follows single Debye type dispersion and the Cole-Cole's distribution parameter α is zero or nearly zero.

The enthalpy of activation corresponding to the various modes of rotation have been evaluated and the value 11.9 kJ mol⁻¹ for $\Delta H\tau_{(2)}$ corresponds to those for the molecular rotation $\Delta H\tau_{OH}$ (10.6 kJ mol⁻¹) and $\Delta H\tau_{GK}$ (9.6 kJ mol⁻¹) giving the most probable enthalpy values observed for the average dispersion and are comparable to one another. $\Delta H\tau_{(1)}$ (1.7 kJ mol⁻¹) corresponding to the dispersion occurring from the process other than the molecular one, is too low and can not be predicted to be due to the internal motion of the group. However, it could be said to have arisen from some segmental motion of the longer group.

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ИЗУЧЕНИЕ ДИЭЛЕКТРИЧЕСКИХ ПОТЕРЬ В НЕКОТОРЫХ α -ЗАМЕЩЕННЫХ БЕНЗИЛЦИАНИДОВ В РАЗБАВЛЕННЫХ РАСТВОРАХ

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Исучены диэлектрические потери для четырех α -замещенных бензилцианидов. Наблюдаемые значения « α » достаточно высоки, что указывает на наличие не одного релаксационного процесса в системе, носящих молекулярный и другой характер. Сделан вывод, что диэлектрическая релаксация относится как к молекулярным, так и групповым процессам.