

## EFFECT OF TEMPERATURE ON FLUORESCENCE QUENCHING BY STEADY STATE AND TRANSIENT METHODS IN SOME ORGANIC LIQUID SCINTILLATORS

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(Received 20 May 1997; accepted 26 September 1997)

**Abstract** – The effect of temperature on the fluorescence quenching of 2-(4-Methoxyphenyl)-5-(1-naphthyl)-1,3,4-oxadiazole (MPNO1), 2-(4-Methoxyphenyl)-5-(2-naphthyl)-1,3,4-oxadiazole (MPNO2), by aniline, and 2-Phenylindole (2-PI) by CCl<sub>4</sub> in toluene by steady state method and in benzene by time-resolved method have been carried out in the temperature range 30 - 70 °C. The Stern-Volmer (S-V) plots, I<sub>0</sub>/I against quencher concentration [Q] at different temperatures show positive deviations. The fluorescence lifetimes determined at different temperatures show no systematic variations and the variations being within the experimental error, the average values of lifetimes  $\tau(t)$  are taken for further calculations. Rate constants such as Stern-Volmer quenching constants K<sub>sv</sub>, quenching rate parameters k<sub>q</sub> and k'<sub>q</sub>, static quenching constant V and kinetic distance r are determined using the modified Stern-Volmer equation and sphere of action static quenching model. In order to see whether the reactions are diffusion limited, equations  $k_q = e^{-E_q/RT}$  and  $k'_q = e^{-E'_q/RT}$  are used to determine the values of E<sub>q</sub> and E'<sub>q</sub>, the activation energies for collisional quenching and the values of E<sub>q</sub> are 14.53, 17.28 and 16.20 kJ mole<sup>-1</sup> for MPNO1, MPNO2 and 2-PI respectively and the values of E'<sub>q</sub> are 14.62 and 17.73 for MPNO1 and MPNO2 respectively. From the magnitudes of various quantities it has been concluded that the reactions are diffusion limited and the observed positive deviations in the S-V plot are due to static and dynamic quenching.

### INTRODUCTION

Considerable amount of work has been done on the study of various intermediate processes that are responsible for the excitation energy quenching of the excited solute molecules by the quencher molecules.<sup>1-6</sup> The excitation energy of the solute molecules can either be transferred to the quencher molecules by various intermediate mechanisms or dissipated through the non-radiative processes.<sup>7-11</sup> This energy can be measured in terms of the quenching rate parameter.<sup>12,13</sup> These processes are excimerization, energy migration, self quenching, exciplex formation, energy transfer, impurity quenching, Brownian diffusion, long range interaction *etc.*<sup>14-16</sup> The phenomenon of quenching appears to be complex because of the various intermolecular mechanisms, formation of charge transfer complexes both at ground and excited states, static and dynamic quenching *etc.* Apart from this, the polarity of the solvent medium<sup>10-15</sup> and the range of quencher concentration and the temperature are also expected to play a part in this mechanism. It appears that the effect of temperature has not received adequate attention. Though some work has been reported earlier<sup>17</sup> its scope has been quite limited and is not sufficient to understand the effect thoroughly. The change in temperature brings about change in the

probabilities of the radiative and non-radiative transitions.

It is important to know the temperature dependence to understand the mechanism underlying these processes. Different experimental techniques such as steady state, time-resolved methods *etc.* are devised to study these mechanisms.

We report in the present paper the results of our investigations on these lines for three organic molecules, namely, (i) 2-(4-methoxyphenyl)-5-(1-naphthyl)-1,3,4-oxadiazole (MPNO1) (ii) 2-(4-methoxyphenyl)-5-(2-naphthyl)-1,3,4-oxadiazole (MPNO2) (iii) 2-Phenylindole (2-PI) in toluene by steady state method with aniline or CCl<sub>4</sub> as quencher and in benzene by time-resolved method without quencher in the temperature range 30 - 70 °C.

### MATERIALS AND METHODS

In the present work, we have used toluene as solvent in the case of steady state method while for time-resolved method benzene is used as solvent. The choice of two different solvents for two different methods makes not much difference or error because it is assumed that lifetime  $\tau$  is approximately constant for a given solute in chemically similar solvents.<sup>12</sup> The solutions were prepared with a fixed solute concentration of 0.5 g/L and quencher concentrations were varied from 0.02 to

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Table 1. Fluorescence lifetime of the solutes in the absence of a quencher as a function temperature in benzene.

Solute	Temperature (°C)	Lifetimes (ns)		Mean $\tau_1/\tau$ (ns)	Mean $\tau_2$ (ns)
		$\tau_1/\tau$	$\tau_2$		
MPNO1	30	0.90	2.40	0.98	2.44
	40	0.97	2.44		
	50	1.08	2.42		
	60	0.99	2.44		
	70	0.99	2.54		
MPNO2	30	2.68	2.54	2.54	3.47
	40	2.44	3.23		
	50	2.97	3.18		
	60	2.38	3.75		
	70	2.27	4.38		
2-PI	30	3.28		13.35	
	40	3.27			
	50	3.25			
	60	3.39			
	70	3.57			

0.1 M in each case. The solutions were taken in a 10 mm path rectangular, stoppered quartz cell and placed in the sample portion of the temperature regulated cell holder for fluorescence intensity. Using the same experimental setup and procedure for steady state measurements as explained in our earlier papers<sup>14-16</sup> the fluorescence intensities  $I_0$  without quencher and  $I$  with different concentrations of quencher  $[Q]$  were measured at different temperatures in the range 30 - 70 °C for all the solutes. The temperature of the cell holder was controlled between 30 and 70 °C with the help of a thermostatic with an accuracy of  $\pm 0.5$  °C.

Fluorescence decay curves of all the solutes were recorded in the absence of quencher in benzene in the temperature range 30 - 70 °C with an Edinburgh Instruments (U.K.) model 199 time-domain spectrometer, operating in single-photon counting mode at BARC, Mumbai. The 199 system consist of a thyratron-

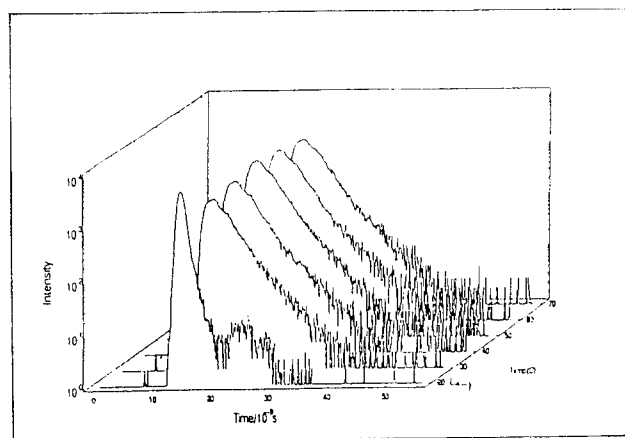


Figure 1. Fluorescence decay profiles of MPNO1 in benzene at different temperatures in the range 30 - 70 °C along with a lamp profile

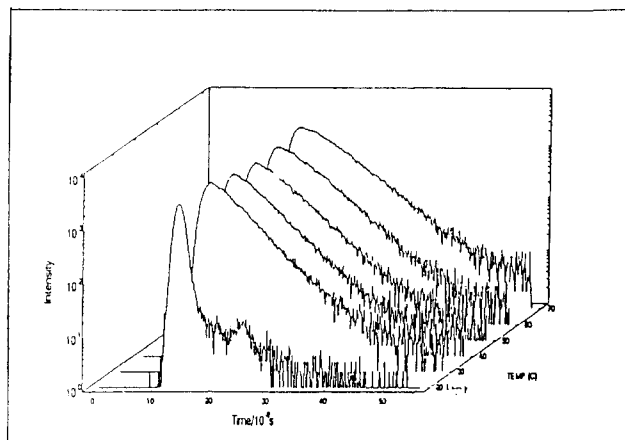


Figure 2. Fluorescence decay profiles of MPNO2 in benzene at different temperatures in the range 30 - 70 °C along with a lamp profile

triggered, metal-bodied, coaxial, gated hydrogen discharge lamp as light source, operating at a repetition rate of 30 kHz (full width at half-maximum (FWHM), approximately 1 ns), a Phillips XP 2020 Q stop photomultiplier and an EG&G Ortec data acquisition system interfaced with a 4K MCA card and IBM PC/AT 386 computer. Fluorescence decay curves were analyzed with a reconvolution program using an iterative non-linear least squares fitting method, assuming that the decay is a sum of first-order processes, i.e. the observed fluorescence decay function  $f(t)$  was obtained as a convolution of the true fluorescence decay curve  $g(t)$  and the instrumental response function  $i(t)$

$$f(t) = \int_0^t i(t-t') g(t') dt'$$

The true decay function  $g(t)$  was obtained by the reconvolution program, using the form

$$g(t) = \sum_i B_i e^{-t/\tau_i}$$

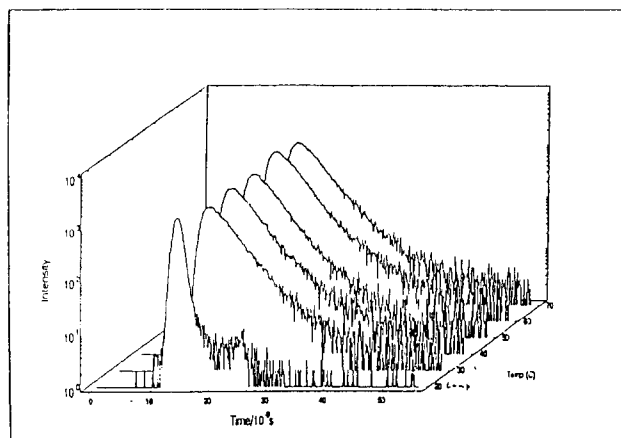


Figure 3. Fluorescence decay profiles of 2-PI in benzene at different temperatures in the range 30 - 70 °C along with a lamp profile

where  $B_i$  is the pre-exponential or proportional constant for the  $i$ th component and  $\tau_i$  is the fluorescence lifetime of the  $i$ th component. The computer fit was evaluated by the minimum reduced  $\chi^2$  value and by the weighted residuals between the data and the fit generated by the theoretical parameters. The temperature of the system was maintained constant using a microprocessor based temperature controller (Eurotherm, U.K. type-810), which maintains the sample temperature within  $\pm 1^\circ\text{C}$  of the required value, utilizing a combined effect of heating by a small heater attached to the sample holder and cooling by a cold finger attachment at the bottom of the sample holder dipped into liquid nitrogen. Temperature of the sample is sensed by a thermocouple sensor attached to the sample holder. The fluorescence lifetime values of all the solutes in the temperature range of  $30 - 70^\circ\text{C}$  are given in Table 1 and the decay curves along with the lamp profiles are shown in Figs. 1 to 3. These are all three dimensional plots as two dimensional plots are highly intermixing in nature.

## RESULTS AND DISCUSSION

When some species of molecules are added as impurities to the scintillator systems, diminish or quench the electronic excitation energy considerably and the quenching is governed by well known Stern-Volmer (S-V) equation given by

$$I_0/I = 1 + K_{sv} [Q] \quad (1)$$

where  $I_0$  and  $I$  are the steady state fluorescence intensities without and with quencher respectively,  $K_{sv}$  is the S-V quenching constant and is equal to  $k_q \tau(t)$  where  $k_q$  is the quenching rate parameter and  $\tau(t)$  is the fluorescence lifetime of the molecule at temperature  $t^\circ\text{C}$  and  $[Q]$  is the quencher concentration. In the case of pure dynamic quenching the S-V plot of  $I_0/I$  against  $[Q]$  is a straight line with intercept and the slope equal to unity and  $K_{sv}$  respectively. The S-V plots  $I_0/I$  against  $[Q]$  in accordance with equation 1 were obtained for all the solutes (Fig. 4) using the experimentally measured values of  $I_0$  and  $I$  at different temperatures. These plots are nonlinear with positive curvature for all the solutes. Further, it is observed that there is a regular but slightly nonlinear decrease in quenching with increasing temperature in the case of MPNO1 and MPNO2 whereas in the case of 2-PI nonlinearity increases initially and becomes independent of temperature at and above  $50^\circ\text{C}$ . This type of positive deviations in the S-V plots, also observed by others<sup>8,15,18</sup>, reveals the role of static quenching process and is explained on the basis of "sphere of action" model. According to this model, the deviation from linear S-V plots is due to the fact that only a certain fraction 'W' of the excited state is actually quenched. This static quenching was explained by introducing an additional factor 'W' in linear S-V equation and is modified to

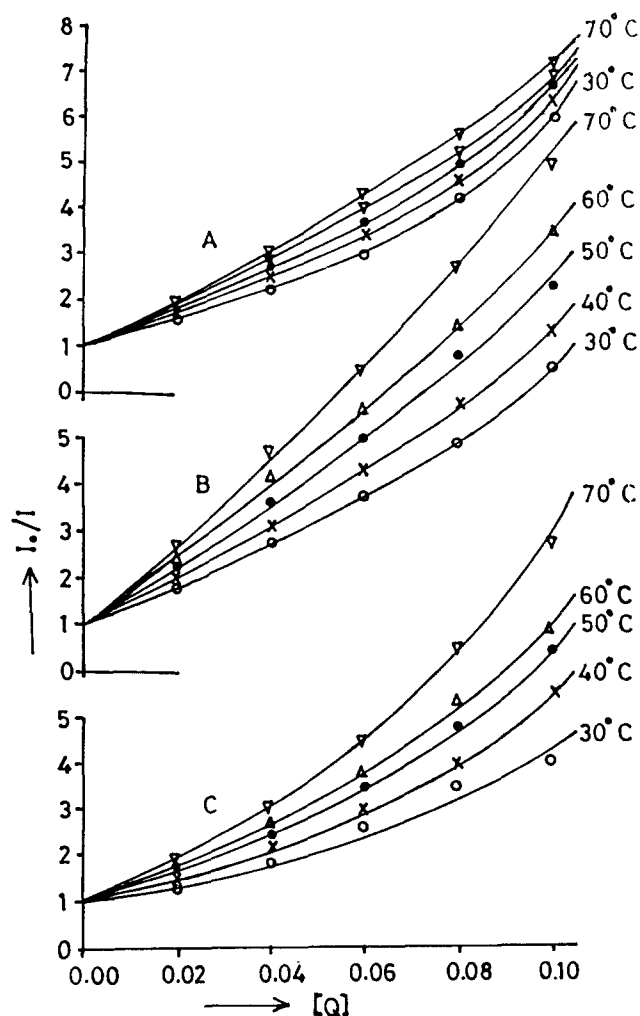


Figure 4. Stern-Volmer plots of  $I_0/I$  against  $[Q]$  at different temperatures in the range  $30 - 70^\circ\text{C}$ . A) MPNO1 + Aniline. B) MPNO2 + Aniline. C) 2-PI +  $\text{CCl}_4$ .

$$\frac{I_0}{I} = \frac{1 + K_{sv} [Q]}{W} \quad (2)$$

In such cases, some molecules in the excited state, the fraction of which is  $(1 - W)$ , are deactivated almost instantaneously after being formed because a quencher molecule happens to be randomly positioned in the proximity at the time the molecules are excited and interacts very strongly with them. Thus, the fraction 'W' decreases from unity in contrast to simple S-V equation where  $W=1$ . Thus the instantaneous or static quenching occurs if the quencher molecule is very near to, or in contact with, the fluorescent molecule at the exact moment that it happens to be excited. The factor 'W' in modified S-V equation is approximately equal to  $\exp(-V[Q])$ , where  $V$  is the static quenching constant and it represents an active volume element surrounding the excited solute molecule<sup>19</sup>. Static quenching takes place at the instances in a randomly distributed system when a quencher happens

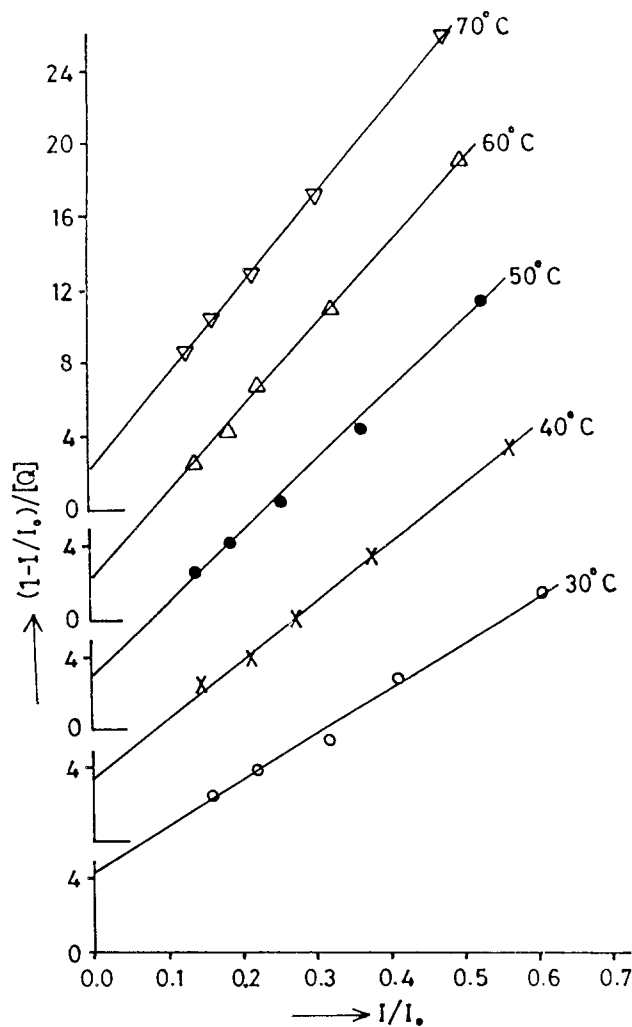


Figure 5. Stern-Volmer plots of  $[1 - (I/I_0)]/[Q]$  against  $I/I_0$  for MPNO1 + Aniline system at different temperatures in the range 30 - 70 °C.

to reside within a sphere of action with a volume  $V/N'$  and radius  $r$  (i.e. kinetic distance) given by  $V/N' = 4\pi r^3/3$  surrounding a solute molecule at the time of excitation. The probability of the quencher being within this volume at the time of excitation depends on the volume and on the quencher concentration. Hence it is meaningful to re-write Eq. (2) as follows.

$$[1 - (I/I_0)]/[Q] = K_{sv} (I/I_0) + (1 - W)/[Q] \quad (3)$$

The modified S-V plots  $[1 - (I/I_0)]/[Q]$  against  $I/I_0$  in accordance with Eq. (3) are shown in Figs. 5, 6 and 7 for MPNO1, MPNO2 and 2-PI respectively. From these, we see that the plots are linear as expected. If  $W=1$  as in the case of linear S-V equation, the non-zero intercepts of Figs. 5 to 7 should strictly go to zero. In the case of MPNO1 and MPNO2 the intercept decreases as a function of temperature whereas in the case of 2-PI, the

Table 2. The Stern-Volmer quenching constant  $K_{sv}$ , the quenching rate parameters  $k_q$  using  $\tau_1$  and  $K'_q$  using  $\tau_2$  as a function of temperature for different solute-quencher systems and the activation energy  $E_q$  using  $\tau_1$  or  $\tau_2$  and  $E'_q$  using  $\tau_2$  for collisional quenching process.

Solute	Quencher	Temperature (°C)	$K_{sv}$ ( $M^{-1}$ )	$k_q \times 10^9$ ( $M^{-1}s^{-1}$ )	$K'_q \times 10^9$ ( $M^{-1}s^{-1}$ )	$E_q$ ( $kJmol^{-1}$ )	$E'_q$ ( $kJmol^{-1}$ )
MPNO	Aniline	30	25.08	25.59		10.27	
		40	31.94	32.59	13.09		
		50	37.75	38.52	15.47	14.53	14.62
		60	45.35	46.35	18.58		
		70	49.65	50.66	20.34		
MPNO2	Aniline	30	37.09	14.60	10.68		
		40	46.55	18.32	13.47		
		50	59.27	*23.33	17.08	17.28	17.73
		60	70.94	27.92	20.44		
		70	84.94	33.44	24.47		
12-PI	$CCl_4$	30	17.54	5.23			
		40	23.28	6.94			
		50	25.16	7.5		16.20	
		60	30.81	9.19			
		70	39.42	11.76			

intercept increases first and then remains almost the same at and above 50 °C. In accordance with Eq. (3), the slope of the plots shown in Figs. 5 to 7 should give S-V quenching constant  $K_{sv}$ . Thus,  $K_{sv}$  was determined in all the cases and for each temperature by least square fit method using Eq. (3) and the values are given in Table 2. The quenching rate parameter  $k_q$  at different temperatures can be determined from the values of  $K_{sv}$  using the equation  $k_q = K_{sv}/\tau(t)$ , where  $\tau(t)$  is the fluorescence lifetime of the solute in the absence of the quencher at  $t$  °C. So, fluorescence lifetime of all the solutes in benzene without quencher and at different temperatures in the range 30 - 70 °C were measured and the values are tabulated in Table 1 for all the solutes. The fluorescence lifetimes of all the solutes in the absence of quencher are found to be different from the values obtained earlier at room temperature. The values of fluorescence lifetimes obtained earlier for MPNO1, MPNO2 and 2-PI are 1.45, 2.60 and 1.78 ns respectively and these were obtained by fitting the decay profiles monoexponentially. But, when we repeated the same experiment using other instrument (Edinburg), decay profiles in the case of MPNO1 and MPNO2 did not fit monoexponentially, on the other hand they fitted well biexponentially giving two values of lifetimes  $\tau_1$  and  $\tau_2$ . However in case of 2-PI decay profiles were fitted monoexponentially as before. In case of MPNO1 and 2-PI, the values obtained are little high compared to those obtained earlier. Further, we can see from Table 1 that the lifetimes of all the solutes in the absence of quencher are found to be independent of temperature. No systematic

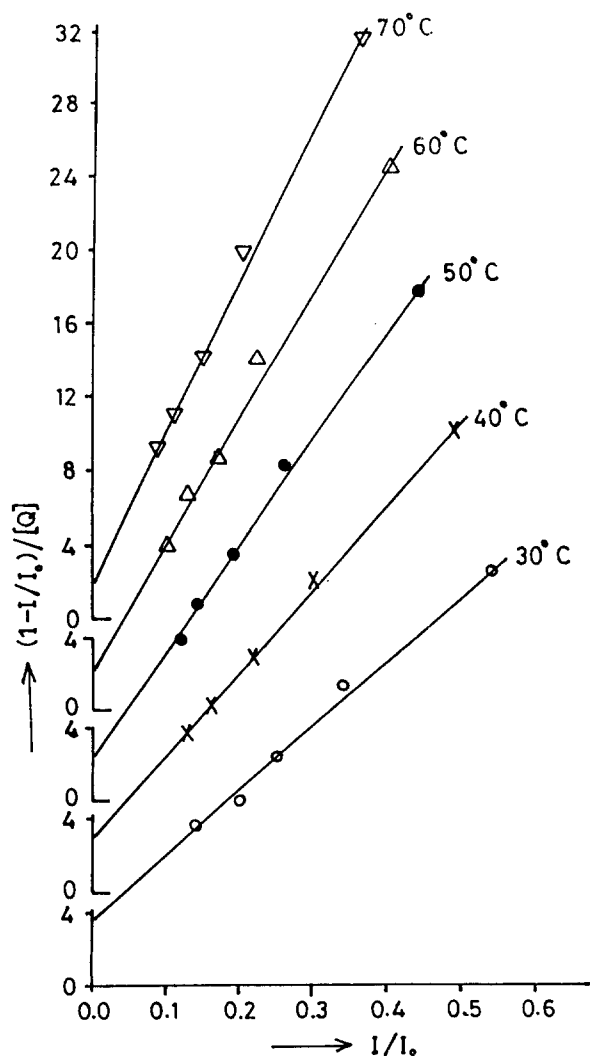


Figure 6. Stern-Volmer plots of  $[1 - (I/I_0)]/[Q]$  against  $I/I_0$  for MPNO2 + Aniline system at different temperatures in the range 30 - 70 °C.

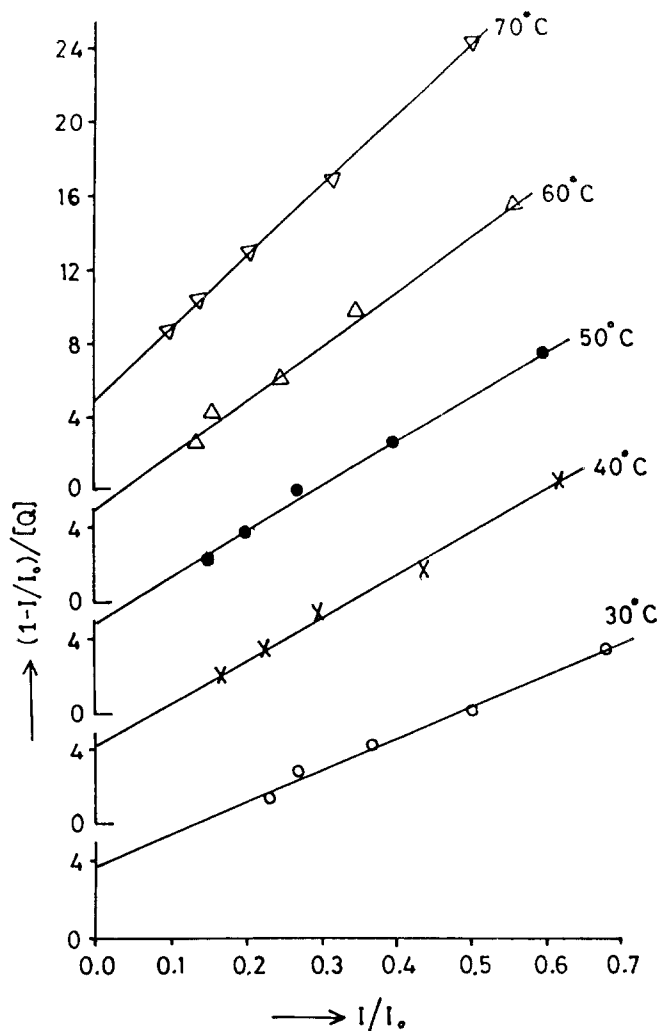


Figure 7. Stern-Volmer plots of  $[1 - (I/I_0)]/[Q]$  against  $I/I_0$  for 2-PI + CCl<sub>4</sub> system at different temperatures in the range 30 - 70 °C.

change in the values of lifetime as a function of temperature is observed. Variation in the values for lifetime of all the solutes are within experimental error because any changes that have taken place lie only in the first or second decimal place. Thus, we have taken the average value of lifetimes at different temperatures as the  $\tau(t)$  value for the solutes. In the case of MPNO1 average of  $\tau_1$  comes out to be 0.98 ns and that of  $\tau_2$  turns to be 2.44 ns. In the case of MPNO2 average of  $\tau_1$  is equal to 2.54 ns and that of  $\tau_2$  is equal to 3.47 ns. Quenching rate parameters  $k_q$  using  $\tau_1$  and  $k'_q$  using  $\tau_2$  are calculated in the case of MPNO1 and MPNO2 and in the case of 2-PI the average value of  $\tau(1)$  equal to 3.35 ns is used to calculate  $k_q$  in accordance with equation  $k_q = K_{sv}/\tau(t)$  and the values are given in Table 2.

According to Eq. (3), the intercepts of Figs. 5 to 7 are equal to  $(1 - W)/[Q]$  and the values of intercepts are given in Table 3. Using the values of intercepts the individual values of  $W$  for each quencher concentration

and for each temperature were calculated and the range of  $W$  values for each temperature is given in Table 3. From the values of  $W$ , we notice that these are less than unity and the values decrease with increase in quencher concentration. Further, from the set of values of  $W$  for each temperature, the static quenching constant  $V$  was determined by least square fit method using equation  $W = e^{-V[Q]}$  and in turn the kinetic distance i.e. the radius of "sphere of action" was determined using the calculated values of  $V$  according to equation  $V/N' = 4\pi r^3/3$ . The values of  $V$  and  $r$  for all the solutes and for the temperature range studied are given in Table 3. From Table 3, we see that the values of  $V$  and  $r$  decrease as a function of temperature in the case of MPNO1 and MPNO2 whereas in the case of 2-PI, the values of  $V$  and  $r$  increase with initial increase in temperature but become independent of temperature at and above 50 °C.

According to Andre *et al.*,<sup>21</sup> if the distance between the quencher molecule and excited molecule lies

Table 3. The values of intercept, range of W, static quenching constant V and kinetic distance r.

Solute	Quencher	Temperature (°C)	Intercept	Range of W	V (mol <sup>-1</sup> dm <sup>3</sup> )	r (Å)
MPNO1	Aniline	30	4.11	0.58-0.91	5.57	13.02
		40	3.22	0.67-0.93	4.08	11.74
		50	2.80	0.72-0.94	3.34	10.98
		60	2.16	0.78-0.95	2.48	9.95
		70	2.05	0.79-0.95	2.30	9.69
MPNO2	Aniline	30	3.21	0.67-0.93	4.09	11.74
		40	2.93	0.70-0.94	3.68	11.34
		50	2.27	0.77-0.95	2.62	10.12
		60	2.16	0.78-0.95	2.49	9.9
		70	1.81	0.81-0.96	2.08	9.39
2-P	CCl <sub>4</sub>	30	3.96	0.60-0.92	5.32	12.82
		40	4.32	0.56-0.91	6.01	13.35
		50	4.97	0.50-0.90	7.31	14.25
		60	4.97	0.50-0.90	7.31	14.25
		70	4.96	0.50-0.90	7.31	14.25

$$R_Y (\text{MPNO1}) = 4.02 \text{ \AA}$$

$$R_Y (2\text{-PI}) = 3.55 \text{ \AA}$$

$$R_Q (\text{CCl}_4) = 2.79 \text{ \AA}$$

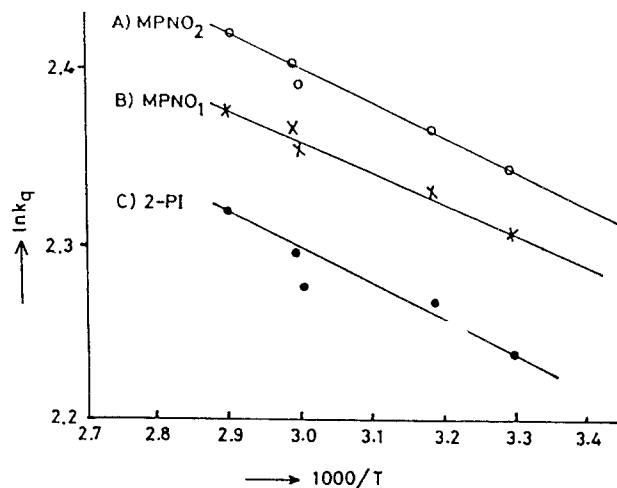
$$R_Y (\text{MPNO2}) = 4.02 \text{ \AA}$$

$$R_Q (\text{Aniline}) = 2.84 \text{ \AA}$$

between the encounter distance R (i.e. the sum of radii of the solute ( $R_Y$ ) and quencher ( $R_Q$ ) and the kinetic distance r, the static effect takes place especially in the case of steady state experiments, irrespective of ground state complex formation provided the reactions are limited by diffusion.

In order to examine whether static effect is playing a part we have determined the radii of the solute and quencher molecules as suggested by Edward<sup>22</sup> and are given at the bottom of Table 3. It is seen that the sum of the radii of the solute and quencher  $R = R_Y + R_Q$  for MPNO1 and MPNO2 systems is 6.86 Å and for 2-PI + CCl<sub>4</sub> system its value is 6.34 Å and the values of r are one and half to two times greater than the sum of radii of the solute and the quencher. Similar results were obtained by others<sup>8,15,23</sup>. So, static effect may play a part provided reactions are limited by diffusion.

In order to check whether reactions are diffusion limited, temperature dependence of rate constant is examined. The S-V quenching constant,  $K_{sv}$ , depends of course, on the temperature through the temperature dependence of the quenching rate parameter  $k_q$  and the lifetime  $\tau(t)$ . However, lifetimes  $\tau_1(t)$  and  $\tau_2(t)$  have not changed as a function of temperature in the present case. But as is evident from Table 2, the values of  $k_q$  and  $k'_q$  are found to increase with increase in temperature in all the cases. It has been assumed that  $k_q$  is proportional to  $\exp(-E_q/RT)$ , where  $E_q$  is the activation energy for collisional quenching process, R is the gas constant and T is the absolute temperature<sup>8</sup>. Thus, the value of  $E_q$  and  $E'_q$  can be determined using the experimentally determined values of  $k_q$  and  $k'_q$  at different temperatures T according

Figure 8. Plots of the rate constants for collisional quenching against  $1/T$  for all the solutes A) MPNO2. B) MPNO1. C) 2-PI.

to equation  $E_q = \exp(-E_q/RT)$  and the least square fit values of  $E_q$  and  $E'_q$  are given in Table 2. From Table 2 we see that the values of  $E_q$  and  $E'_q$  are almost the same. Hence in Fig. 8 we have given plots of  $\ln k_q$  against  $1000/T$  for one of the values of  $k_q$  for each solute. Similar values of  $E_q$  have also been observed by Moriya<sup>8</sup> and this suggests that the rate of reactions is limited only by the diffusion of the reactants through the solvent solution.

Further a positive deviation in the S-V plot is likely when both static and dynamic quenching occur simultaneously<sup>23</sup>. In view of these facts we may conclude that both static and dynamic quenching processes are responsible for the observed positive deviation in the S-V plots in accordance with Andre *et al.*,<sup>22</sup>.

## CONCLUSION

In view of the fact that the encounter distance R and the kinetic distance r are in accordance with the literature values and the values of  $E_q$  agree with the literature values, we may conclude that the positive deviations observed in S-V plots are due to both static and dynamic quenching.

*Acknowledgement*—The authors (TPG and JSK) are grateful to the authorities of Kamatak University, Dharwad and UGC, New Delhi for the award of teacher fellowship under FIP scheme.

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