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Triplet–Triplet Annihilation in Dilute, Solid Solutions of Aromatic Polymers**, ***

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Summary. The triplet–triplet annihilation of mass fractionated *PVCA* ($M = 3.6 \cdot 10^3$–$1.4 \cdot 10^6$) in toluene glasses was studied by phosphorescence, for glasses of *PVCA* and *P2VN* (order of $M = 10^6$) in *MTHF* the intensity of phosphorescence and delayed fluorescence were studied as a function of the excitation intensity. A survey of T–T annihilation in solid solutions of aromatic polymers is given and requirements for a theory explaining low dimensional energy transport and exciton–exciton annihilation are pointed out.

Keywords. Polymers; Energy transfer.

Triplet–Triplet-Auslöschung in verdünnten festen Lösungen aromatischer Polymerer

Zusammenfassung. Die Triplet–Triplet-Auslöschung von nach Masse fraktionierten *PVCA* ($M = 3.6 \cdot 10^3$–$1.4 \cdot 10^6$)-Toluol-Gläsern wurde mittels Phosphoreszenzmessungen untersucht, für Gläser von *PVCA* und *P2VN* (M im Bereich 10^6) in *MTHF* wurde die Intensität der Phosphoreszenz und der verzögerten Fluoreszenz als Funktion der Anregungs-intensität untersucht. Es wird ein Überblick über T–T-Auslöschung in festen Lösungen aromatischer Polymerer gegeben und die Voraussetzungen für eine Theorie des Energietransportes und der Exciton–Exciton-Auslöschung diskutiert.

Introduction

Aromatic Polymers in dilute, solid solutions are spectroscopically unique systems. They offer a high density of chromophores, as in films, but in **pseudo one-dimensional** arrangement in the form of matrix-isolated, well separated macromolecules. The solvents used should form clear, rigid glasses and be transparent to the radiation used for excitation and to the luminescences emitted by the polymer dissolved. It is their function to immobilize and to keep apart the polymer chains. The solvent also influences the shape of the macromolecules in liquid solution and thus likely also after solidification.

A strictly one-dimensional ordering of the monomeric units would come close

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** Dedicated to Professor Otto Kratky, Graz, on the occasion of his 90th anniversary

*** Presented at the ACS-Symposium Polymer Photophysics: Fundamentals and Applications. ACS-Spring Meeting 1992, San Francisco, April 5–10, 1992 to the original model proposed by Staudinger in the early 30's [1, 2], which took not into account the free rotation around single bonds. Staudinger proposed extended chain rod-like macromolecules. The modern model of polymer coils, developed by Kuhn [3] and Mark [4] is based on the rotation around single bonds. It takes the form of "worm-like chains" (Fadenmoleküle [5, 21]), if the free rotation is hindered by bulky substituents. In polyvinylaromatics [e.g. poly(styrene) (*PS*), poly(vinyl naphthalene) (*PVN*), and poly(N-vinyl carbazole) (*PVCA*)] the side-groups have to wind up for sterical reasons, even if a perfect order (as in helices) is not possible for reasons of low tacticity and high mobility of the segments in liquid solutions.

A measure for the stiffness of macromolecules in solution is the "persistence length" (s) after Kratky and Porod [5]; it amounts to about $s = 2.33$ nm in solutions of *PVCA* in toluene [6]. This corresponds to about 8–9 monomer units in the extended chain or roughly three turns of the hypothetical 3/1-helix in a perfect isotactic order. The persistence length of the less stiff *PS* amounts to $s = 1.22$ nm (measured in cyclohexane [35]). The well known model by Kuhn [3] uses straight model-segments of length $a = 2s$, which perform a random flight in 3 dimensions and n steps ($n \cdot a$ = chain length). The end-to-end distance ($a \cdot \sqrt{n}$) is a measure of the coil dimension. Kuhn's statistical segment amounts to $a = 4.66$ nm or 17 monomer units in the case of the stiff macromolecule *PVCA* in a Θ-solvent [6].

The measurements concerning the chain stiffness (light scattering, viscosity etc.) were performed in *liquid* solutions [6] and it is not clear a priori what the coil dimensions are likely to be in rigid solution at low temperature. There is, however, good evidence that during cooling and glass-formation in suitable solvents, the relatively open coils persist. Spectroscopically, we do not observe any "true" (= sandwich) excimer fluorescence [7] in dilute, solid solutions of *PVCA* and several other polymers. This means that there is no detectable amount of excimer-forming sites (*EFS*) in these solutions. The absence of any true excimer fluorescence also proves the *absence of solid particles or collapsed coils* which may have formed during the cooling of the polymer solution and the glass-forming process.

The shallow second excimer in *PVCA* ("Trap II" [8]), which is formed without thermal activation and involves the overlap of only *one* aromatic ring [9] can be observed in solid solutions of radically prepared, predominantly syndiotactic *PVCA*.

What do we mean by "pseudo one-dimensional"? A truly one-dimensional (1D) object exists only in theory. In reality we can study only three-dimensional (3D) objects, e.g. molecular crystals with strong overlap in one direction, conjugated polymers, helical polymers, or polymer coils. The latter are evidently not 1D in space; this is of no importance, however, from the point of view of energy transfer and triplet–triplet annihilation. In this case, the decisive question is, how many non-excited immediate neighbours has an excited side-group ("hopping exciton")? If there are two, than we have one-dimensional energy transfer, provided the excitation energy can only be transferred to next neighbours. It has *not* been shown beyond doubt, however, whether this is the case or not in aromatic polymer coils, which are crowded with bulky sidegroups. The dimensionality may depend on the nature of electronic coupling between the side groups: short range coupling (as in the case of triplets!) may preferentially lead to 1D, long range coupling (Förster transfer with allowed transitions) may lead to 3D behaviour. This different behaviour was recently reported by Bässler [10, 11] for singlet transfer in Poly (isopropenyl naphthalene) (weak coupling, 1D) and *PVCA* (strong coupling, 3D). Burkhart [12, 36, 40], also argues in favour of 3D energy transfer, whereas Frank [13, 14] finds evidence for 1D electronic energy transport (EET) in Poly(2-vinyl napthalene) (*P2VN*).

Conventional EET studies involve the addition of fluorescent or phosphorescent guest molecules which are able to trap and to reemit, al least partly, the excitation energy [15–17]. Alternatively, the excitation energy may be quenched non-radiatively. The guest molecules may also be parts of the polymer chains, e.g. in suitable co-polymers.

A recent re-evaluation of the published results on sensitized luminescence and quenching experiments showed [7] that 1D-EET is the predominant energy transfer mechanism in most cases. The model used in this work was a very simple one, so that the results should be critically tested both with further experiments and with refined theories.

Here we report on triplet–triplet (T–T) annihilation as a further tool is studying EET for triplet states. In contrast to sensitized guest luminescence, no addition of foreign compounds, copolymerization or chemical modification of the polymer is needed. T–T annihilation can easily be studied by delayed fluorescence (DF), the *singlet channel* (1) of the merger of the triplet excitons [18–20]:

$$T_1 + T_1 \rightarrow S_n, \tag{1a}$$

$$S_n \rightarrow S_1 + \text{heat}, \tag{1b}$$

$$S_1 \rightarrow S_0 + h\nu_{DF}; \tag{1c}$$

T_1, S_1: 1st excited triplet or singlet state, S_n: higher excited singlet state, S_0: singlet ground state, $h\nu_{DF}$: photon of delated fluorescence.

A fraction of the S_1 created (1b) returns to T_1 by inter-system crossing.

The *triplet channel* of T–T annihilation (2) only leads to the loss of triplets without new emission:

$$T_1 + T_1 \rightarrow T_n, \tag{2a}$$

$$T_n \rightarrow T_1 + \text{heat}, \tag{2b}$$

$$T_1 \rightarrow S_0 + h\nu_p \text{ (or heat)}; \tag{2c}$$

T_n: higher excited triplet state, $h\nu_p$: photon of phosphorescence.

Delayed fluorescence caused by annihilation of triplet excitons is a much studied phenomenon in molecular crystal research [18, 20]. It may also occur if one of the two T_1 in (1) or (2) is actually trapped or belongs to another chemical species (guest molecule) in its (long lived) 1st excited triplet state. This process is called hetero fusion [22].

The effects of dimensionality and coherence (of the exciton) on T–T annihilation have been studied theoretically [23, 24]. Here we need to consider only incoherent exciton hopping (electronic energy transport, EET). Coherent excitons only occur in well ordered structures, as in single crystals, at low temperature [20]. Furthermore, dispersive EET is likely due to the energetical disorder of the excitation energies of side-groups [51], leading to time-dependent hopping rates ($k_h = f(t)$), whereas in the classical exciton hopping model k_h is taken to be constant.

A Short History of T–T Annihilation in Solid Solutions of Aromatic Polymers

Cozzens and Fox [25] observed for the first time delayed fluorescence in rigid solutions of Poly (1-vinylnaphthalene) (*P1VN*) and in copolymers of styrene and 1-vinylnaphthalene [26]. The low temperature delayed emission spectrum shows clearly phosphorescence (P) *and* delayed fluorescence (DF). The new emission (DF) was correctly explained as "due to triplet–triplet annihilation following intramolecular triplet energy transfer through the naphthalene chromophores" [25]. The mobility of the triplets was demonstrated indepentently by quenching experiments [7, 17, 25].

The triplets can be generated either by UV-absorption in the singlet system or by sensitization using benzophenone as triplet sensitizer. The classical papers by Fox and Cozzens [25, 26] were presented in part at the 155th National Meeting of the Am. Chem. Soc., April 1968, in San Francisco [28].

Later it was shown by Klöpffer and Fischer [8] and by Yokoyama et al. [19, 30] that the same effect can be observed in solid solutions of *PVCA*. It also was found by both groups that there is a clear molecular weight effect on the DF/P intensity ratio: high P at low, high DF at high molecular weight (M) of the *PVCA* sample studied. Cozzens and Fox [25] had already reported that there is some minimum chain length required in order to show DF in their experimental arrangement.

The experiments on *PVCA* were repeated using fractionated samples [31], and showed essentially the same results: The intensity of the DF increases with M (at constant excitation intensity) and levels off at high M. The intensity of P decreases with M and also approaches a constant value at high M [31]. Qualitatively, this behavior can easily be understood: at low M, many chains carry only one triplet at the same time, or none at all (only P possible); at high M, most chains carry 2 or more triplets, so that T–T annihilation becomes possible, as manifested by the singlet channel (1) which leads to DF. Quantitatively, this effect is still poorly understood.

We also found [30, 31] that the decay time of P is about $\tau_p \approx 8$ seconds, as in monomeric N-alkylcarbazoles, but $\tau_{DF} \approx 3$–30 milliseconds, much lower than expected for the ordinary T–T kinetics ($\tau_p = 2\tau_{DF}$ [18]). This result means that the phosphorescence observed originates from trapped triplets, *not* from the mobile ones ("triplet excitons")! The same observation was made in conventional quenching experiments [7]. A small red shift in phosphorescence, compared to monomeric model compounds, supports this interpretation and indicates a trap depth of a few 100 cm^{-1} [7].

In *P2VN* and Poly(2-naphthyl methacrylate), a qualitatively similar, although less pronounced M-dependance was observed by Pasch and Webber [32, 33]. T–T annihilation in Poly(naphthyl methacrylate) had already been observed in 1973 by Somersall and Guillet [34]. Similar effects have been observed in other aromatic polymers [37–39].

No T–T annihilation was ever observed in solid solutions of Poly(acenaphthylene); this seems to be due to immobile or very slow triplets, probably a consequence of the sterical arrangement of the chromophores which offers little overlap of the π-electrons of neighbouring groups [7] which is necessary for the electron exchange mechanism to be efficient.

Webber and coworkers showed in a series of papers [47–49] that the triplet trapping behaviour of *PVCA*, *P2VN* and Poly(acenaphthylene) is quite different. Trapping seems to be most efficient in *PVCA* [47]. In *P2VN* [49], on the other hand, there are long-lived triplet excitons escaping the shallow traps present [7]; this was shown in quenching experiments using the short lived triplet state of biacetyl as a probe. Our own experiments on pure, radically polymerized *P2VN* of high molar mass showed that the trap depth is about 200 cm^{-1}, compared to about 400–500 cm^{-1} in *PVCA*. This is lower than the 480 cm^{-1} trap depth formerly derived from Nishijima's spectra of *P2VN* [7]; 200 cm^{-1} corresponds to a barrier hight of about 4 kT at 77 K, corresponding to a Boltzmann-factor of $e^{-4} \approx 0.02$.

In Poly(acenaphthylene), the triplets are confined to a few segments [49] so that T–T annihilation could be detected, if at all, only at very high excitation intensities.

Results and Discussion

PVCA in Toluene Glasses

Toluene is a Θ-solvent of *PVCA* at 37–39 °C [6, 41] and forms a glass upon rapid cooling to temperatures below 113 K [42]. The glasses tend to crack, however, so that many experiments cannot be evaluated. It is to be expected that the polymer coils are as small as possible for a given M in this solvent and that any contact between the coils is very unlikely. The usual concentrations of about 10^{-4} mol basic unit/L are far below c^*, the "critical concentration" ($c^* \approx 1/[\eta]$ to $3/[\eta]$ where $[\eta]$ = intrinsic viscosity [43–46]) which determines the transition between the dilute and the semi-dilute region of polymer solutions. For *PVCA* with $M = 10^7$ in toluene, we obtain $c^* \approx 0.4$ to 1.2 g/dL ≈ 0.02 to 0.06 mol basic unit/L.

The experiments were designed in such a way that (in principle) all relevant emission-intensities could be obtained in absolute terms. Unfortunately, only the

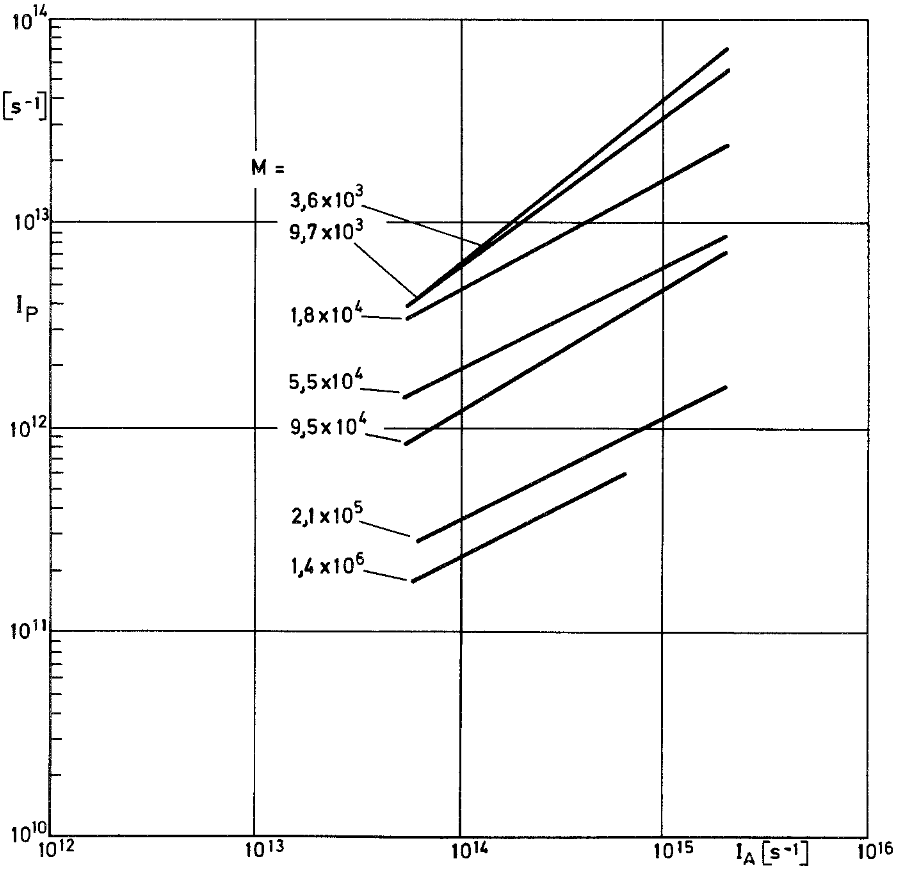

Fig. 1. *PVCA* in toluene glass at 77 K: phosphorescence intensity I_p (number of emitted photons) vs. number of absorbed photons I_A per second; molar mass indicated

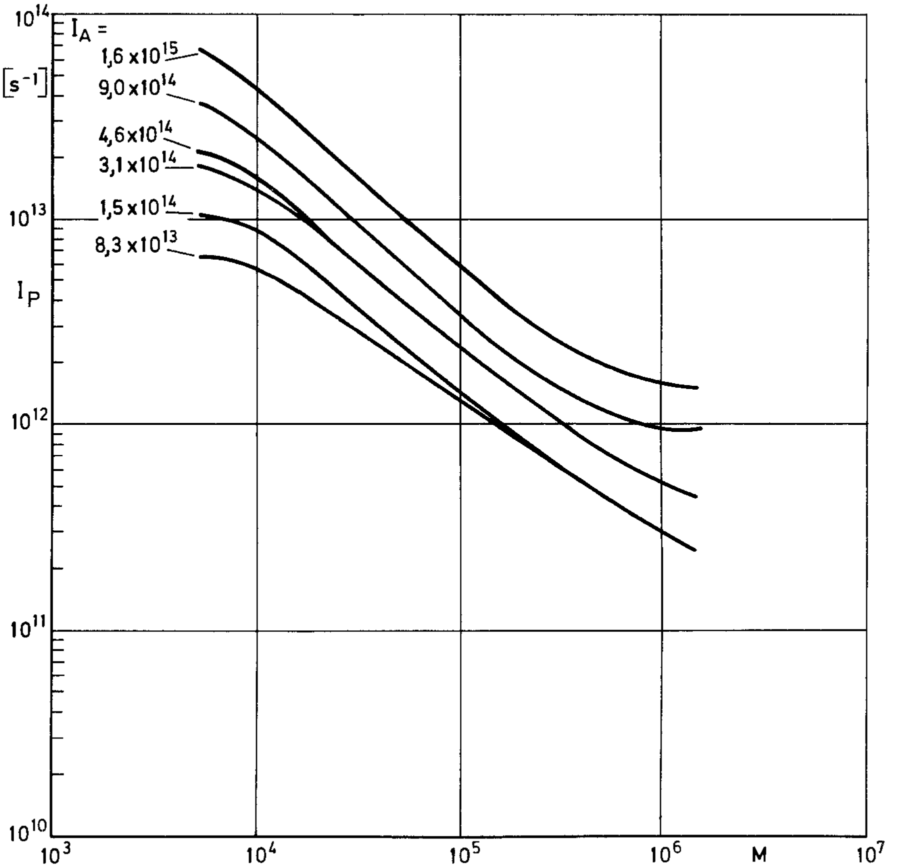

Fig. 2. *PVCA* in toluene glass at 77 K: phosphorescence intensity I_p (number of emitted photons) vs. molar mass (M) of *PVCA*; excitation intensity indicated

phosphorescence data could be evaluated, since the fast decaying part of the non-exponential DF was not or only partly transmitted by the mechanical phosphoroscop, whose time limit is about 1 ms. The illumination in these experiments (excitation wavelength: 313 nm) was pseudo-stationary with dark- and light periods in the millisecond range. The essential results are shown in Figs. 1 and 2.

The average slope of I_p vs. I_A is 0.6 ± 0.1 (Fig. 1). For a unimolecular process, ordinary reaction kinetics would predict a slope of 1, i.e. a linear dependence of the phosphorescence intensity in the intensity of the exciting radiation.

Using narrow M-fractions, I_p was found to depend inversely on the molar mass at constant excitation intensity I_A and between $M \approx 10^4$ to 10^6 (Fig. 2),

$$I_p \text{ (\textit{PVCA}, M-fractions) prop. } 1/M.$$

At higher excitation intensities ($I_A \approx 10^{15}$ photons/s) I_p tends to saturate near $M \approx 10^6$. Simple calculations using Poisson distribution of the triplets among the chains showed that saturation, if only due to multiple occupancy of the chains, should occur at much lower M at the excitation intensity used in these experiments.

PVCA and P2VN in MTHF-Glasses

New samples of high molar mass *PVCA* (cat. and rad.) and *P2VN* (rad.) were prepared and purified according to standard procedures. All important data of *PVCA* agree well with older work.

The fluorescence spectrum of *P2VN* (Fig. 3) shows mirror-image symmetry with the first absorption band, as expected in pure *P2VN*, and agrees with the one published by Holden and Guillet [50] and with the fluorescence spectrum of the monomeric model 2-Methylnaphthalene (Fig. 3); it does not agree with the spectra published by Pasch and Webber [32] and Siu and Burkhart [51].

The T–T annihilation of *PVCA* and *P2VN* was studied in dilute, solid solutions in *MTHF* without fractionation. Since all average molar masses are in the order of 10^6, no molar-mass dependence could be studied. The intensity of phosphorescence (I_p) and of delayed fluorescence (I_{DF}) was studied as a function of the excitation intensity I_A (number of photons absorbed/s).

Figure 4 is typical for all measurements with *PVCA* and *P2VN*: The slope of I_{DF} vs. I_A is about twice that of I_p vs. I_A, indicating bimolecular kinetics, although no simple linear (P)/square (DF) relationship was observed, as in molecular crystals [18, 20]. All relevant data are compiled in Table 1.

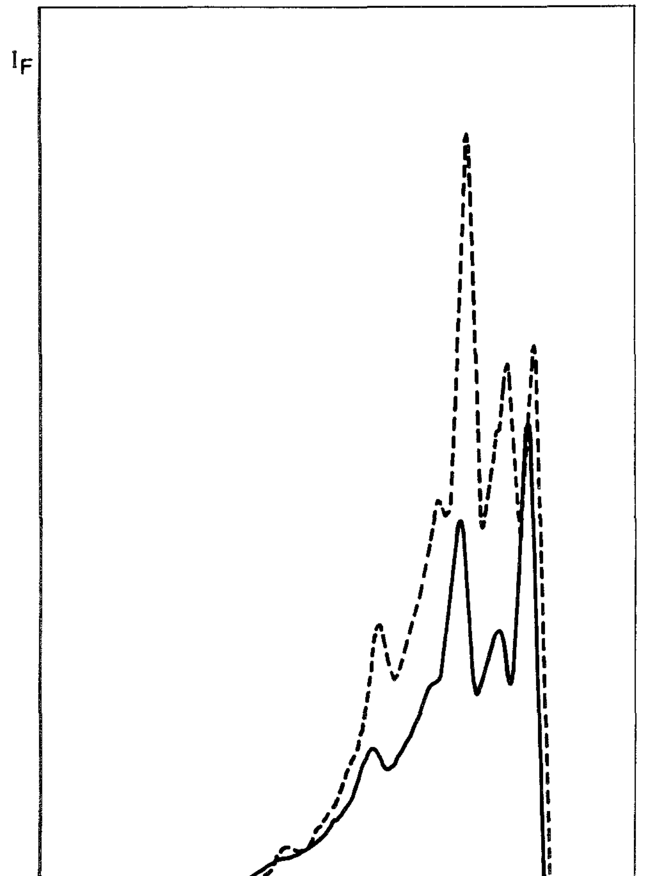

Fig. 3. Fluorescence spectrum of *P2VN* in *MTHF* (solid line); 2-methylnaphthalene (dashed); relative fluorescence intensity I_F vs. wavelength [nm]

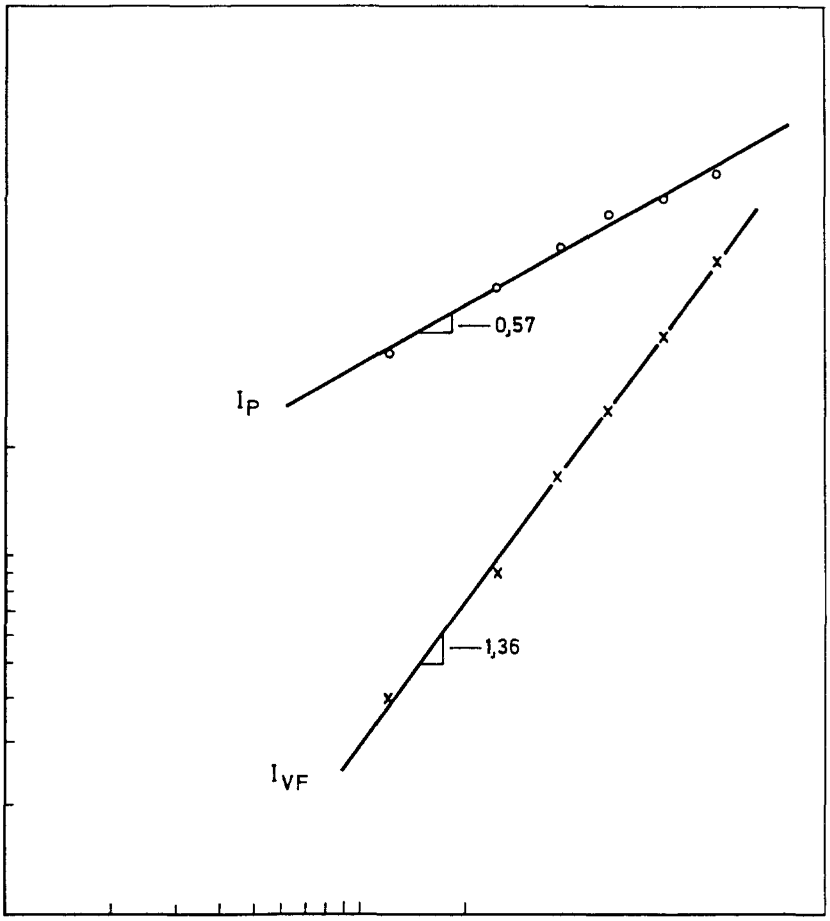

Fig. 4. *PVCA* cationic in *MTHF* at 77 K: phosphorescence intensity (I_p) and delayed fluorescence intensity (I_{VF}) vs. excitation intensity in relative units

Table 1. Bilogarithmic plot of luminescence intensities versus excitation intensity I_a in *MTHF* at 77 K

Polymer[a]	Slope I_p vs. I_A	Slope I_{DF} vs. I_A	Slope DF/Slope P
PVCA cat II	0.57	1.36	2.4
PVCA rad I	0.69	1.31	1.9
P2VN I	0.67	1.55	2.3
P2VN II	0.79	1.75	2.2
Average:	0.7 ± 0.1	1.5 ± 0.2	2.2 ± 0.2
PVCA (Tol.):	0.6 ± 0.1		

[a] I and II designates different batches of cationically or radically polymerised *PVCA* and radically prepared *P2VN*. All "new" polymer samples have average molar masses of about 10^6 or higher

The average of all I_p vs. I_A slopes amounts to 0.7 ± 0.1 which agrees well with the results obtained with *PVCA* in toluene (0.6 ± 0.1).

The average of all I_{DF} vs. I_A slopes is 1.5 ± 0.2. The correlation coefficients of all determinations of slopes are excellent ($r \geqslant 0.98$ in most cases).

It was found again that the decay of DF is much faster (< 100 ms) in all cases than that of phosphorescence (> 1 s). This indicates that the phosphorescence observed experimentally originates from relatively shallow traps. The trap depth can be estimated from the spectral shift of the polymer emission relative to that of a monomeric model [7, 17]. In agreement with earlier claims, the traps are more shallow in *P2VN* and the decay of DF is slower (about 50–70 ms) in this polymer compared to *PVCA*. The decay of *DF* in *PVCA* (about 10 ms) agrees well with an early measurement by Yokoyama et al. [29].

Conclusion

From a theoretical point of view, there has been an increasing interest in problems of low dimensional energy transport and exciton–exciton annihilation [13, 23, 24, 36, 51–58]. However, despite so many excellent papers on this subject, there is no theory directly applicable to the present problem. We therefore can only formulate some requirements which should be met by a future theory explaining the experimental observations:

(1) Pseudo 1D (or higher?) EET.
(2) Dispersive EET.
(3) Finite length/volume effects.
(4) Role of triplet traps and end groups.

A suitable theory should also explain explicitely the peculiar M-dependence of P and DF and the dependence of I_{DF} and I_p on the excitation intensity of I_A. Further experimental work should include the exact time-dependence of the processes studied, both at the singlet- and triplet level.

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