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# Steady-state luminescence studies of some $\alpha$ and $\beta$ derivatives of naphthanilide at 293 K and 77 K

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## Abstract

The absorption and fluorescence excitation spectra of six naphthalene derivatives have been measured in various solvents at 293 K and 77 K. The absorption spectra do not change significantly from dry hydrocarbon to polar solvents at the two temperatures. The emission spectra measured at 293 K exhibit two fluorescence bands: the normal $F_1$(LE) fluorescence band and the $F_2$ band, which consists of the $F_2'$(PT) intramolecular proton-transfer and the $F_2''$(CT) twisted intramolecular charge-transfer fluorescence emissions. *N*,$\beta$-naphthyl-$\alpha$-naphthanilide (IV) is an exception to that pattern; in all the solvents used, only the $F_1$(LE) fluorescence has been observed. At 77 K, the total luminescence shows two resolved bands: the first is either the normal $F_1$(LE) (molecules III, IV, V, VI) or the excimer $F_{agr}$ (molecules I, II) fluorescence band, and the second is the phosphorescence band, $F_{Ph}$(LE). Our spectroscopic studies point that the $F_1$(LE) and $F_{Ph}$(LE) bands originate from perturbed naphthalene moieties. © 1998 Elsevier Science S.A. All rights reserved.

*Keywords:* Naphthanilides; Absorption and emission spectra at 293 K and 77 K

## 1. Introduction

Naphthanilides shown in Fig. 1 belong to the class of hetero- and homopolar molecules in which two chromophores are linked by a peptide bridge. In this group of molecules, the peptide linkage plays a significant role of a 'spacer' between two rigid molecular structures, donor (D) and acceptor (A). Bichromophoric molecules, in which intramolecular electronic energy transfer takes place, exhibit dual fluorescence: the normal $F_1$(LE) fluorescence which is characteristic of molecules with one chromophore, and another, structureless $F_2$ emission (at energies lower than $F_1$) originating from the excited state of a mutually perpendicular chromophore $D^+$–$A^-$ structure reached in the twisted intramolecular charge-transfer (TICT) process [1,2]. This class of molecules plays an important role in photobiology (in living organisms [3,4]), quantum optics (dye lasers [5], optoelectrical switches [6], light frequency converters [7]) and photochemistry (photopolymers [8,9], photoreactions [2,10]). For that reason, this class of compounds has evoked a considerable experimental and theoretical interest over the past few decades [11–18,32].

From the studies of Lipczynska-Kochany [19–21], it follows that the absorption spectra and the normal $F_1$(LE) fluorescence bands of the molecules like these given in Fig. 1 can be analysed on the basis of the naphthalene absorption and fluorescence spectra. As it was shown in Ref. [17], in dry hydrocarbon solvents (i.e., methylcyclohexane (MCH) and cyclohexane (CH)), the $F_2$ fluorescence band of $\beta$-naphthanilide (I) and $\alpha$-naphthanilide (II) consists of two overlapping fluorescence bands. These two are an $F_2'$(PT) intramolecular proton-transfer band and an $F_2''$(CT)–TICT band. The methyl-substituted derivatives (V, VI), (where the proton-transfer phenomenon cannot happen) possess only the $F_2''$(CT) fluorescence band. This identification was confirmed using chemical substitution and polarity perturbation effects, as well as by picosecond time-resolved transient absorption studies [17].

Up to now, little attention has been paid to low-temperature (77 K) luminescence studies of the compounds like these shown in Fig. 1. Kasha et al. [23], analysing the accessible data concerning phosphorescence studies of molecules exhibiting excited state intramolecular proton transfer, predicted the existence of one or two such phosphorescence bands. The number of phosphorescence bands depends on the magnitude of the two triplet state energies of a locally excited moiety, $T_1$(LE), and an excited state intramolecular proton-transfer

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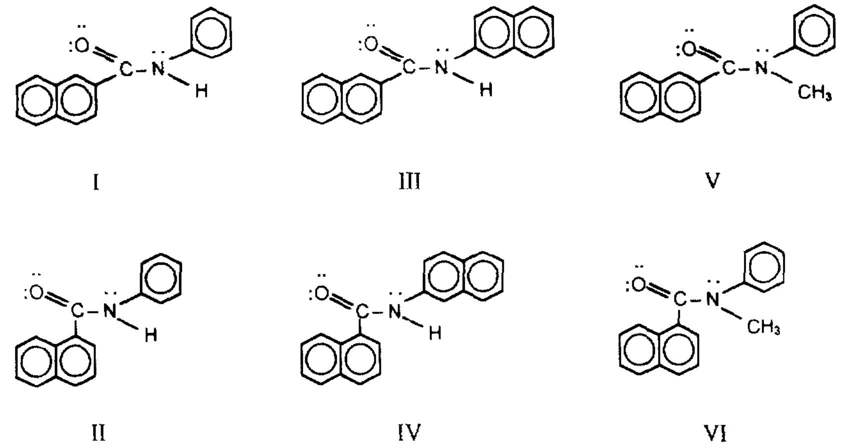

Fig. 1. The chemical structures of β-naphthanilide (I); α-naphthanilide (II); N,β-naphthyl-β-naphthanilide (III); N,β-naphthyl-α-naphthanilide (IV); N-methyl-β-naphthanilide (V); N-methyl-β-naphthanilide (VI).

tautomer $T_1'$(PT). Three cases may be taken into consideration, the first when $T_1(LE) < T_1'(PT)$, the second for $T_1(LE) \cong T_1'(PT)$, and the third one, when $T_1(LE) > T_1'(PT)$.

In the first case, it is assumed that the energy difference $T_1'$(PT)-$T_1$(LE) is too great for significant population of the $T_1'$(PT) state from the $T_1$(LE) state due to Boltzmann statistics of population of vibrational levels of the latter state, so an enhancement of the $T_1(LE) \rightarrow S_0$ phosphorescence can be only expected. Such an effect has been observed for some aminosalicylates [23]. If the $T_1$(LE) triplet state energy is almost equal to $T_1'$(PT), as it happens for 2-(2′-hydroxyphenyl)benzoxazole, the dual phosphorescence is observed [24]. In the third case, when $T_1(LE) > T_1'(PT)$, the energy splitting $S_1$(LE)-$T_1$(LE) is small and $S_1'$(PT)-$T_1'$(PT) is comparable or larger, then only one, i.e., $T_1'(PT) \rightarrow S_0'(PT)$ proton-transfer tautomer phosphorescence band is observed [25,26].

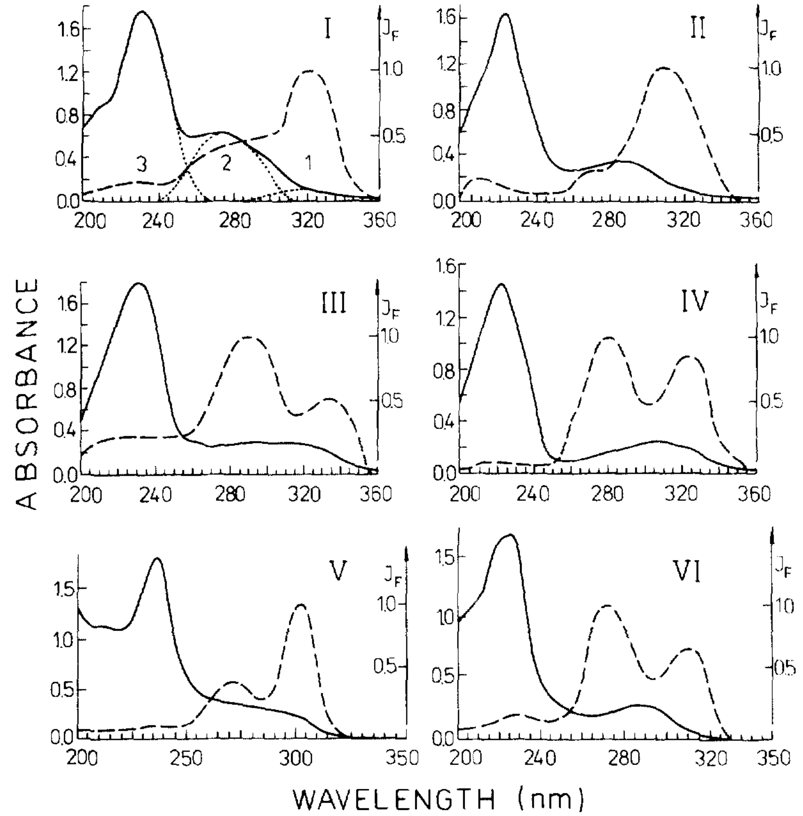

Fig. 2. The absorption (———) and fluorescence excitation (- - -) spectra of molecules I–VI in MCH at 293 K. The first graph shows exemplary decomposition of the absorption spectrum of molecule I into three bands.

This work reports on the results of complete spectroscopic studies (absorption and emission) of the six naphthanilide derivatives examined at 293 K and 77 K. Particular attention has been paid to the luminescence (fluorescence and phosphorescence) spectra at 77 K, especially to compounds III and IV, for which any spectroscopic data had been unknown.

## 2. Experimental details

The compounds under study were obtained and purified by Lipczynska-Kochany as described in Ref. [21]. The methylcyclohexane solvent was distilled from a sodium potassium amalgam before using, to ensure it is pure and free of water. Ethyl ether, isopentane, ethanol and toluene were distilled in the usual way. The spectroscopic studies at 77 K were performed in MCH, ethyl ether/isopentane/ ethanol 5:5:2 v/v (EPA), and ethyl ether/ethanol/toluene 2:1:1 v/v (EET) rigid glasses. The concentration of the molecules in the glass mixtures was about $10^{-4}$ M.

Absorption spectra measurements were carried out using a Shimadzu UV-200 and a HP8452A spectrophotometer. Suprasil absorption cells with 2 or 5 mm absorption pathlength were used. The luminescence spectra at 293 K and 77 K were recorded on a Baird-Atomic (Model SFR-100) or an IBM spectrofluorometer using a low-temperature standard sample compartment with a vacuum-sealed quartz cold finger dewar. The luminescence radiation was observed perpendicularly to the direction of the excitation beam. For the measurements carried out at 293 K, a sample was put into a standard rectangular Suprasil 10 mm cell, whereas in the case of a low temperature run it was put in a 2 mm diameter quartz tube placed in a dewar containing liquid nitrogen. For the phosphorescence spectra measurements, a mechanical chopper performing as a phosphoroscope was used. The luminescence spectra have been corrected for the spectral response of the detection system. The fluorescence and phosphorescence excitation spectra have been not corrected for the spectral characteristics of the high-pressure Ar/Hg lamp mounted in the spectrofluorometer.

## 3. Results and discussion

Fig. 2 shows the absorption spectra of $\alpha$- and $\beta$-naphthanilide (I, II), their *N*-methyl derivatives (V, VI), as well as *N*,$\beta$-naphthyl-$\alpha$- or $\beta$-naphthanilide (III, IV) in MCH at 293 K. The absorption spectra of the compounds in question possess a few distinct bands, which are similar to those of 2-naphthalenecarbohydroxamides (investigated by Lipczynska-Kochany [21]) and of naphthalene [27,28]. Therefore, they can be analysed on the basis of the naphthalene

Table 1

The wavelengths $\lambda_{max}$ (nm) corresponding to the maximum intensity of absorption, fluorescence ($F_1$, $F_2$), excimer fluorescence ($F_{agr}$), and phosphorescence (Ph) emission for molecules I–VI in various solvents

| Compound | Temperature (K) | Solvent | Absorption | | | Emission | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | $B_b$ | $L_b$ | $L_a$ | $F_1$ | $F_{agr}$ | $F_2$ | Ph |
| I | 293 | MCH | 230 | 282 | 322 | 338 | | 490 | |
| | | EET | – | 280 | 320 | 358 | | 532 | |
| | 77 | MCH | | | | | 403 | | 490 |
| | | EET | | | | | 430 | | 496 |
| | | EPA | 235 | 285 | | | 428 | | 497 |
| II | 293 | MCH | 223 | 295 | 310 | 338 | | 510 | |
| | | EET | – | 280 | 315 | 380 | | 560 | |
| | 77 | MCH | | | | | 424 | | 488 |
| | | EET | | | | | 415 | | 484 |
| | | EPA | 243 | 290 | | | 430 | | 484 |
| III | 293 | MCH | 228 | 283 | 340 | 370 | | 506 | |
| | | EET | – | 290 | 345 | 380 | | 506 | |
| | 77 | EET | | | | | 426 | | 497 |
| | | EPA | 230 | 290 | | | 430 | | 495 |
| IV | 293 | MCH | 230 | 292 | 325 | 396 | | – | |
| | | EET | – | 295 | 323 | 400 | | – | |
| | 77 | EET | | | | 400 | – | | 496 |
| | | EPA | 230 | 292 | | 402 | – | | 497 |
| V | 293 | MCH | 237 | 290 | 305 | 335 | | 516 | |
| | 77 | MCH | | | | 334 | | | 481 |
| | | EPA | 243 | 291 | | 334 | | | 481 |
| VI | 293 | MCH | 225 | 285 | 305 | 336 | | 535 | |
| | 77 | MCH | | | | 336 | | | 490 |
| | | EPA | | | | 340 | | | 485 |

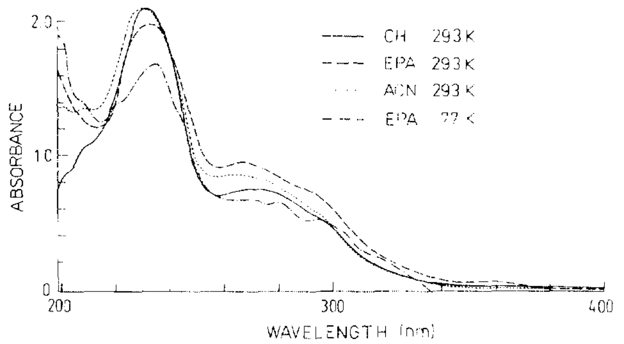

Fig. 3. The absorption spectra of β-naphthanilide (II) in various solvents and temperatures.

absorption spectrum. A substitution of groups containing π-electrons in the α or β position of naphthalene results in extended conjugation primarily along the longitudinal or along the perpendicular axis, respectively [29,33]. This causes the observed red shift of the respective bands.

In the absorption spectra of the molecules studied, three main bands appear (see Fig. 2). The corresponding wavelengths for molecule I are: $\lambda_{max\ 1} \approx 320$ nm ($S_1(^1L_b) \leftarrow S_0(^1A)$), $\lambda_{max\ 2} \approx 280$ nm ($S_2(^1L_a) \leftarrow S_0(^1A)$), and $\lambda_{max\ 3} \approx 230$ nm ($S_3(^1B_b) \leftarrow S_0(^1A)$). The exact values of $\lambda_{max}$ of the three main bands for all the molecules measured in the hydrocarbon and the polar solvents are collected in Table 1. The $\lambda_{max}$ values of the $S_1$ and the $S_2$ band are mainly based on the excitation spectra. From the analysis of the absorption and the fluorescence excitation spectra (pre) and the $\lambda_{max}$ data given in Table 1, column 4, it follows that the α substituted molecules shift the $^1L_b$ band to the red more than the $^1L_a$ band with regard to the positions of the corresponding naphthalene bands; the $\lambda_{max}$ values of the three bands do not change significantly with temperature and with the identity of the solvent (Fig. 3). The weak bands $^1L_b$ and $^1L_a$ (onset at about 250 nm) have their structure very blurred, which is more visible in the polar solvents. The existence of such the structure is distinctly pronounced in the fluorescence excitation spectra. Serious differences noticed between the shapes of the absorption and the excitation spectra indicate that in the $S_1$(LE) excited states, the molecules undergo structural rearrangement.

Figs. 4 and 5 show the luminescence spectra of the molecules under study in different solvents and temperatures. Spectroscopic data for all the performed emission measurements are given in Table 1. The fluorescence spectra of the five molecules under study (all except IV) exhibit two separate bands in the non-polar and the weak polar solvents at 293 K; these are the $F_1$ and the $F_2$ fluorescence bands (Fig. 4). The $F_1$ fluorescence band of the β substituted molecules (I, III and V), has vibrational peaks analogous to those of naphthalene [27]; in the case of the α substituted compounds (II, IV and VI), the peaks are less visible. The $F_1$ fluorescence band of α- and β-naphthanilide is red-shifted by about 2460 cm$^{-1}$, whereas for N,β-naphthyl-α- and β-naphthanilide by 5020 cm$^{-1}$ in comparison with the naphthalene fluorescence

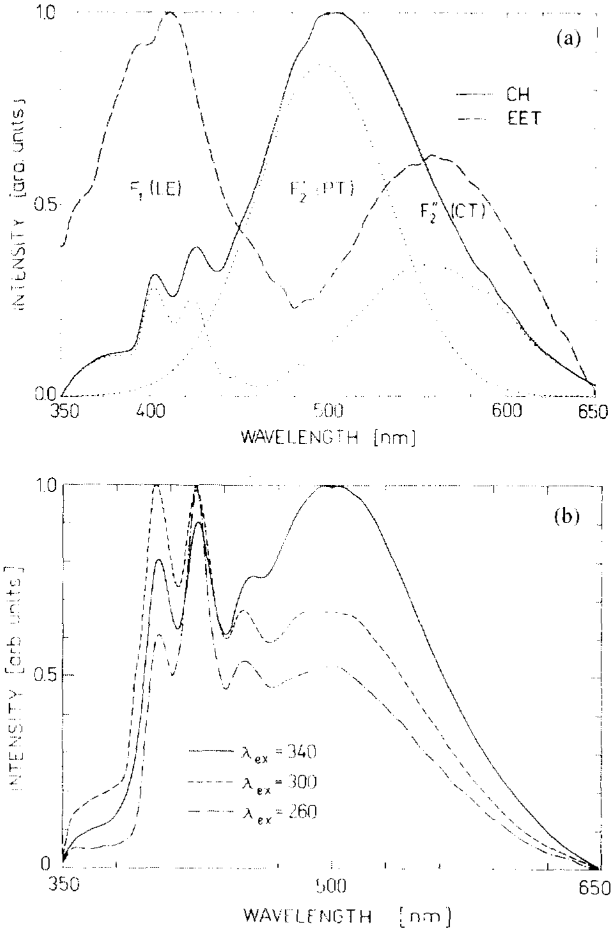

Fig. 4. The fluorescence spectra of N,β-naphthyl-β-naphthanilide (III) obtained at 293 K in various solvents (a), and if the sample was excited by various wavelengths (b). The fluorescence spectrum in CH has been decomposed (· · · ·) into three components: the normal $F_1$(LE) fluorescence spectrum, the $F_2'$(PT) proton-transfer, and the $F_2''$(CT) charge-transfer bands. The spectrum obtained in EET has the $F_1$(LE) and the $F_2''$(CT) bands only.

band. The $F_1$ structured band shows mirror symmetry with the long-wave absorption band. The vibrational peaks' positions do not depend on the excitation wavelength, whereas their intensity increases along with the increase of the excitation wavelength (Fig. 4b). The $F_1$ fluorescence maximum intensity position reveals strong environmental dependence (Fig. 4a); the polar solvents shift the $F_1$ band to shorter wavelengths, simultaneously increasing its intensity. In very strong polar and protic solvents, the $F_1$, as well as the $F_2$ fluorescence bands are quenched due to H-bonding and dielectric medium modulation effects.

It should be noticed that N,β-naphthyl-α-naphthanilide, molecule IV, yields only the $F_1$ fluorescence emission in all the solvents used, i.e., in CH, MCH, EPA, and EET. This band has the distinctive naphthalene vibrational structure and therefore can be ascribed to the locally excited state transition, $S_1(LE) \rightarrow S_0$. The relative intensities of the vibrational peaks depend on the solvent. Our results and the results of other authors [17,19] indicate that the $F_1$ fluorescence band derives from the perturbed weak absorption band $^1L_b \rightarrow {}^1A$ of the naphthalene moiety.

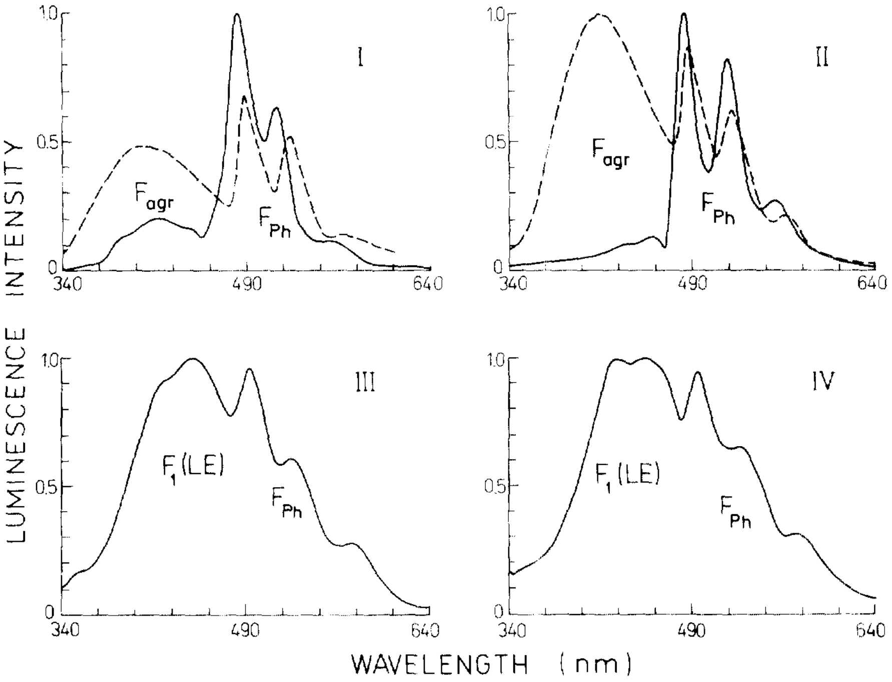

Fig. 5. The luminescence (fluorescence plus phosphorescence) spectra of molecules I–IV in EPA at 77 K.

The $F_2$ fluorescence bands of compounds I, II, III, V, and VI in MCH are shifted respectively 9178, 9978, 7264, 10 470, 11 070 $cm^{-1}$ to the red in comparison with the $F_1$ fluorescence. This red shift depends on the polarity of the solvents: it is larger when the solvent is more polar (see Table 1). As an example, Fig. 4a shows the $F_2$ fluorescence band of molecule III in cyclohexane decomposed (the dotted line) into the $F_2'(PT)$ ($\lambda_{max}$ at 497 nm) and the $F_2''(CT)$ ($\lambda_{max}$ at 558 nm) bands. It is seen that the peak positions of the $F_1(LE)$ and the $F_2''(CT)$ components of the spectrum agree well with that obtained for this molecule in the EET solution, where only $F_1(LE)$ and $F_2''(CT)$ appear. The decomposition procedure adapted to the total fluorescence spectra of molecules I and II gives a similar result. The value of $\lambda_{max}$ for $F_2'(PT)$ equals to 479 nm and 485 nm, whereas the maximum wavelength for $F_2''(CT)$ equals to 509 nm and 524 nm for molecules I and II, respectively. The $\lambda_{max}$ values determined for the $F_2''(CT)$ band are in good agreement with that obtained in direct measurements for molecules V and VI, for which only the $F_1(LE)$ and $F_2''(CT)$ fluorescence bands are observed. Taking into account these and the results of our earlier studies [17,18] on half-width changes of the $F_2$ fluorescence band with the dielectric constant of the solvents and on the transient absorption spectra, we are convinced that in hydrocarbon solvents the $F_2$ fluorescence bands of molecules I, II and III exhibit two emission modes, i.e., $F_2'(PT)$ (the proton-transfer tautomer emission $S_1'(PT) \rightarrow S_0'(PT)$) and $F_2''(CT)$ (the intramolecular charge-transfer fluorescence $S_1''(CT) \rightarrow S_0$). Molecules V and VI as well as the other molecules (I, II and III) in the polar solvents show the $F_2''(CT)$ emission only. For the molecules above, the $\lambda_{max}$ value of the $F_2''(CT)$ fluorescence band is greater than that of the $F_2'(PT)$ band. As we have mentioned before, molecule IV in all the solvents used shows the normal $F_1(LE)$ fluorescence band only.

The luminescence spectra of the molecules examined at 77 K are shown in Figs. 6 and 7. The total luminescence spectra consist of two parts: the first is either the broad structureless excimer band (molecules I and II) or the normal $F_1(LE)$ fluorescence emission (molecules III-VI), and the second is the phosphorescence spectrum. The intensity of the structureless band of $\alpha$- and $\beta$-naphthanilide, I, II, as well as *N*,$\beta$-naphthyl-$\beta$-naphthanilide, III, (onset at 310 nm, $\lambda_{max} = 409$ nm in MCH) depends on the solvent. It is the largest in MCH. If polarity of the solvent increases the band intensity decreases (Fig. 5). This emission band is attributed to the excimer fluorescence [22]. The excimers are formed in a sandwich structure, as it has been proved in Ref. [21], by polar molecules in non-polar solvent at low temperature. The excimer fluorescence band, $F_{agr}$, has its maximum at 428 nm, which is in good agreement with the results obtained for compounds of the naphthalene–$(CH_2)_n$–naphthalene type [22,30]. The excimer fluorescence band is shifted about 6500 $cm^{-1}$ in comparison with the normal $F_1(LE)$ fluorescence band of naphthalene. The structured short-wave parts of the broad-band emission of compounds III–VI belong to the normal $F_1(LE)$ fluorescence. This band has the vibrational structure similar to that observed at 293 K and is shifted about 1500 $cm^{-1}$ to longer wavelengths for molecules III, IV and 700 $cm^{-1}$ to shorter wavelengths for V and VI, all relatively to their position at 293 K. Their intensities show strong solvent polarity dependence (Fig. 4a).

The phosphorescence spectra of the molecules in question show the vibrational structure characteristic of naphthalene

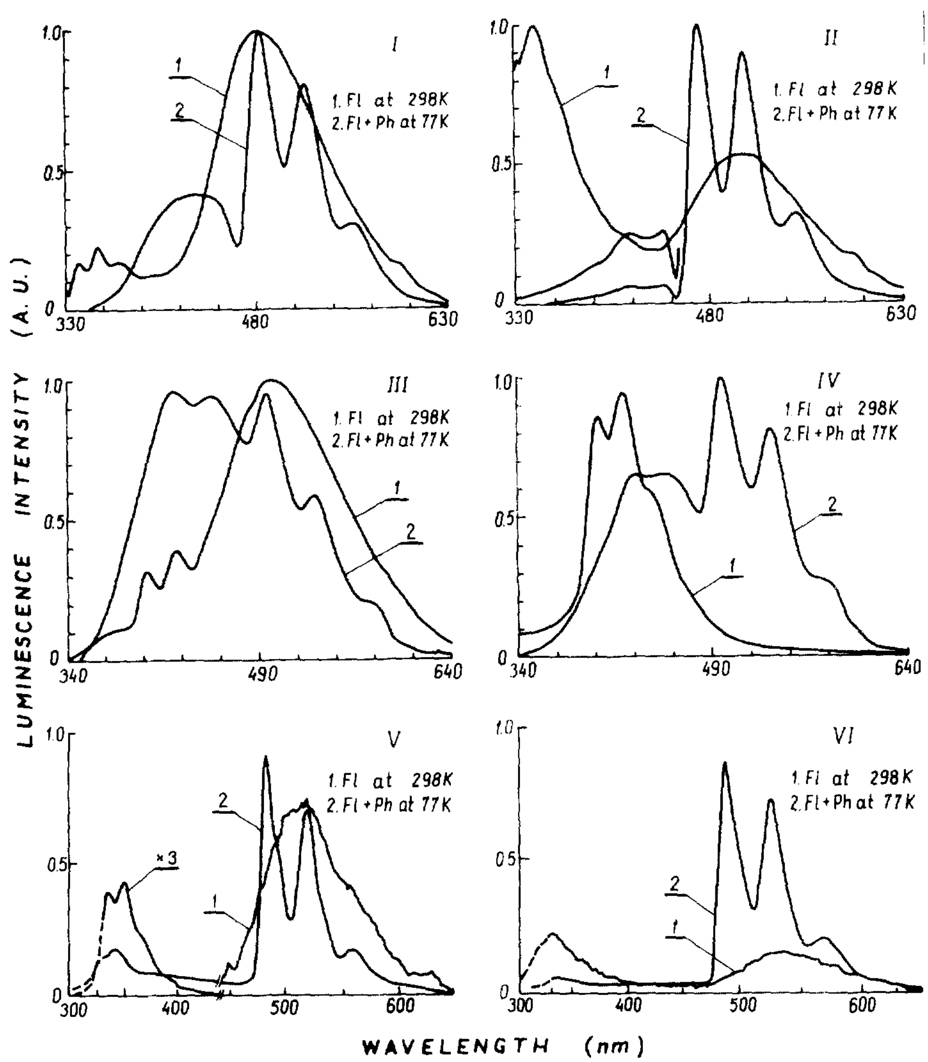

Fig. 6. The luminescence (fluorescence at 293 K (1), and total luminescence, i.e., fluorescence plus phosphorescence, at 77 K (2)) spectra of molecules I–IV obtained in EET and V, VI in MCH.

Table 2
Energy values of the excited states of the molecules under study

| Molecule | | | | | | | |
|---|---|---|---|---|---|---|---|
| State | Naphthalene | I | II | III | IV | V | VI |
| $S_0$ ($^1A_g$) | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| $T_1$ ($^3B_{2u}$) | 21 180 [a] | 20 408 | 20 492 | 20 120 | 20 160 | 20 790 | 20 410 |
| $S_1$* ($^1B_{3u}$) | 32 151 [b] | 31 056 | 31 258 | 30 850 | 30 770 | 32 790 | 32 560 |
| $S_2$* ($^1B_{2u}$) | 36 365 [b] | 35 460 | 36 364 | 35 483 | 35 090 | 36 360 | 36 630 |
| $S_3$ ($^1B_{3u}$) | 45 250 [b] | 43 480 | 44 440 | 43 103 | 45 055 | 42 190 | 44 440 |

*The energy values of the $S_1$ and $S_2$ states are taken from the fluorescence excitation spectrum.
[a] [A.P. Marchetti, D.R. Kearns, J. Am. Chem. Soc., 89 (1967) 768].
[b] [H. Suzuki Electronic Absorption Spectra and Geometry of Organic Molecules. Academic Press New York London, 1967, Chap. 10].

(Figs. 5–7). Three vibrational peaks are red-shifted (by about 650 $cm^{-1}$ for molecules II, V and VI, and about 1050 $cm^{-1}$ for molecules I, III, IV) in comparison with their positions in the case of naphthalene [30]. The vibrational structure of N,β-naphthyl-α- and β-naphthanilide (molecules III, IV) is strongly blurred. As it is shown in Fig. 7b, this is caused by overlapping of the wing of the broad and blurred normal fluorescence emission. The phosphorescence spectrum measured independently by using a rotating chopper display a very distinct vibrational structure; this is valid for

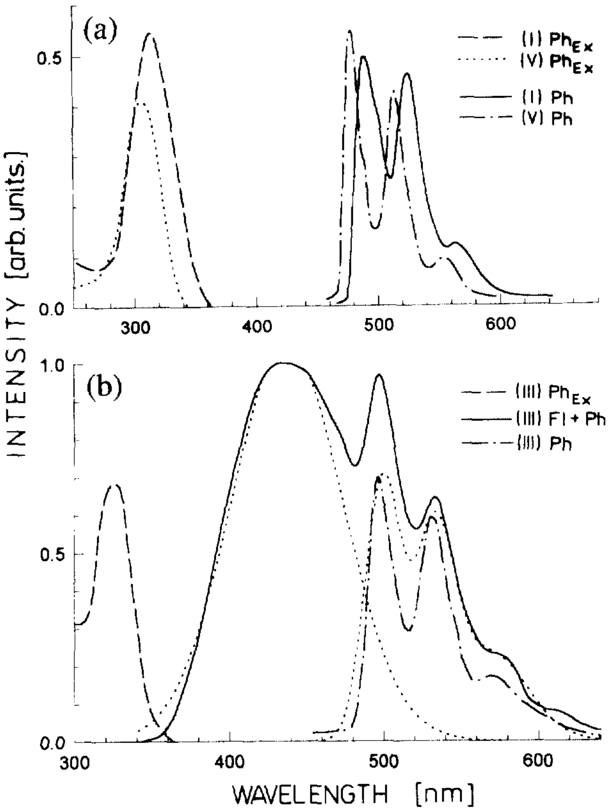

Fig. 7. The phosphorescence emission (Ph) and excitation ($Ph_{Ex}$) spectra of molecules I and V in EPA at 77 K (a); the total luminescence (Fl + Ph) spectrum of molecule III in EET at 77 K and its decomposition (· · ·) into the excimer fluorescence spectrum ($\lambda_{max} = 432$ nm) and the phosphorescence spectrum (b). The phosphorescence spectrum obtained from the decomposition is compared with the pure phosphorescence spectrum (– · –) obtained using a phosphoroscope.

the six molecules. As it is shown in Fig. 7b, the phosphorescence spectrum is similar to that obtained by the decomposition procedure of the total luminescence spectrum. The decay time measurements of the different emission modes of these groups of bichromophoric molecules are in progress.

The experimental data obtained in the studies performed, as well as the adequate data characterizing naphthalene, are summarised in Table 2. In conclusion of the discussion above, and from the analysis of the data assembled in Table 2, the following may be deduced.

(1) The absorption, the normal fluorescence, and the phosphorescence spectra originate from the perturbed naphthalene moiety of the compounds under study. Proton-transfer tautomers are responsible for the $F_2'(PT)$ fluorescence, while the $F_2''(CT)$ emission results from these molecules in which the charge-transfer phenomenon takes place.

(2) The $T_1$ level of the molecules in question is shifted to the lower energies. This finding is in agreement with the results of other authors [22,30,31].

(3) The energy values of the $S_1$ state of molecules I–IV are less than that of naphthalene by 850 $cm^{-1}$ for $\alpha$-, $\beta$-naphthanilide (I, II), and 1200 $cm^{-1}$ for $N,\beta$-naphthyl-$\alpha$- and $\beta$-naphthanilide (III, IV). For $N$-methyl-$\alpha$- and $\beta$-naphthanilide (V, VI), these values are greater than that of naphthalene (about 500 $cm^{-1}$).

(4) The energy values of the $S_2$ state of molecules I–VI are greater than that of naphthalene. The $(E(S_2)-E(S_2^{naph}))$ differences varies from 200 to 1600 $cm^{-1}$ and reach greater values for the $\alpha$-substituted derivatives.

(5) The energy values of the $S_3$ state of the molecules studied are less than that of naphthalene.

The measurements carried out by us do not allow the determination of the energy values of the first singlet states of the proton-transfer tautomers, $S_1'(PT)$, and the charge-transfer molecules, $S_1''(CT)$. The fluorescence bands ascribed to $S_1'(PT) \rightarrow S_0'(PT)$ and $S_1''(CT) \rightarrow S_0$ transitions have their $\lambda_{max}$ values respectively at 479 nm and 509 nm for molecule I, 485 nm and 524 nm for molecule II, and 497 nm and 558 nm for molecule III. In all the solvents used, whenever both $F_2'(PT)$ and $F_2''(CT)$ fluorescence appear, the $\lambda_{max}(PT) < \lambda_{max}(CT)$ inequality is fulfilled.

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