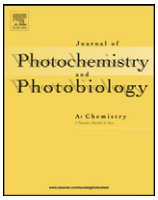
Journal of Photochemistry and Photobiology A: Chemistry 225 (2011) 52–57

Contents lists available at SciVerse ScienceDirect

Journal of Photochemistry and Photobiology A: Chemistry

journal homepage: www.elsevier.com/locate/jphotochem



Luminescence properties of diamino-dicyano substituted benzene and 1,4-pyrazine

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ARTICLE INFO

Article history:
Received 30 June 2011
Received in revised form 25 August 2011
Accepted 24 September 2011
Available online 1 October 2011

Keywords:
Photophysics
Fluorescence
Organic molecules

ABSTRACT

The photophysics of several positional isomers of dicyano-diamino benzenes and 1,4-pyrazines in liquid and frozen solutions has been investigated in several organic solvents. Depending on the relative position of donor and acceptor groups the fluorescence lifetimes and quantum yields change over an order of magnitude and more than a double, respectively. This effect can be rationalized in terms of the relative positioning of amino and nitrile groups and the interplay between singlet and triplet sublevels as a direct consequence of the former.

1. Introduction

The recent discovery of highly fluorescing *p*-phenylenediamines (PPDs) [1–3] poses interesting questions about the origin of the phenomenon as previous to such findings this class of molecules was not thought to possess any remarkable fluorescence properties [4–6]. In the two cases reported in the literature, the addition of nitrile groups to the benzene ring increased the fluorescence quantum yield from a modest 0.1 for *p*-phenylenediamine to 0.6 for 2,6-dicyano-*N*,*N*′-tetramethyl-*p*-phenylenediamine (DCTMPPD) and to 0.9 for tetracyano-*p*-phenylenediamine (TCPPD), both of them emitting with lifetimes as long as 20 ns. In none of these molecules the dipole moments of the excited states were found to be large, so intramolecular charge transfer states were ruled out. Additionally, in the latter case a reversible electrochemical reduction process could be followed showing a logical evolution from the typical PPDs electrochemical behavior to that of cyanobenzenes. In none of these cases a clear explanation was found for the large lifetimes and quantum yields of fluorescence observed, despite the remarkable interest that TCCPD has as possible solvent H-bonding acceptance ability probe and the fact that this is one of the very few known purely organic molecules emitting in the 600 nm wavelength range with a fluorescence lifetime longer than 20 ns.

A similar behavior was observed in the past for the mono and dicyano substituted anilines [7–9]. Moreover, it was found that the position of the cyano group strongly affects the fluorescence quantum yield and lifetime of the molecule, leading in the extensively studied case of *p*-DMABN to dual fluorescence due to the appearance of an emissive charge transfer state close in energy to the local excited emissive singlet [10]. Zachariasse and co-workers have reported on 3,4- and 3,5-dicyanodimethylaminobenzene (34DCDMA and 35DCDMA, respectively) and *o*- and *m*-cyanodimethylaminobenzene (*o*-DMABN and *m*-DMABN, respectively), in which despite the lack of dual emission, the differences in excited state lifetimes are remarkable and stay not fully explained. For example, *o*-DMABN has a lifetime of the order of 5 ns, while *m*-DMABN of 23 ns [11,12].

These positional effects in the benzene ring on photophysics have been studied for a long time [13–16], though for the case of more than two substituents rationalization is rather difficult. When comparing both groups of molecules, anilines and phenylenediamines (PDs), the second ones show generally larger fluorescence quantum yields and longer excited state lifetimes. On the other hand, dicyanobenzenes (DCBs) show much larger emission quantum yields and longer lifetimes than phenylenediamines, though emitting more in the blue than the latter. For instance, *p*-dicyanobenzene has an emission quantum yield in organic solvents at room temperature close to unity while *p*-phenylenediamine of less than 0.1, and the former a lifetime around 10 ns while the latter only about 3 ns [5,6,17–20].

Another interesting aspect of these molecules is the possibility to use them as building blocks for molecular wires and molecular magnets [21]. Aniline based polymers have been widely used, and the possibility to change their properties by the addition of substituents to the ring may render useful properties. Obviously, upon polymerization over the amino group the properties of the

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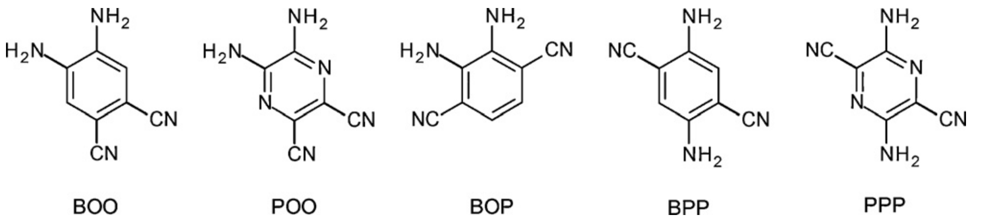
doi:10.1016/j.jphotochem.2011.09.025



Scheme 1. Compounds investigated in this work and the short names employed in the text.

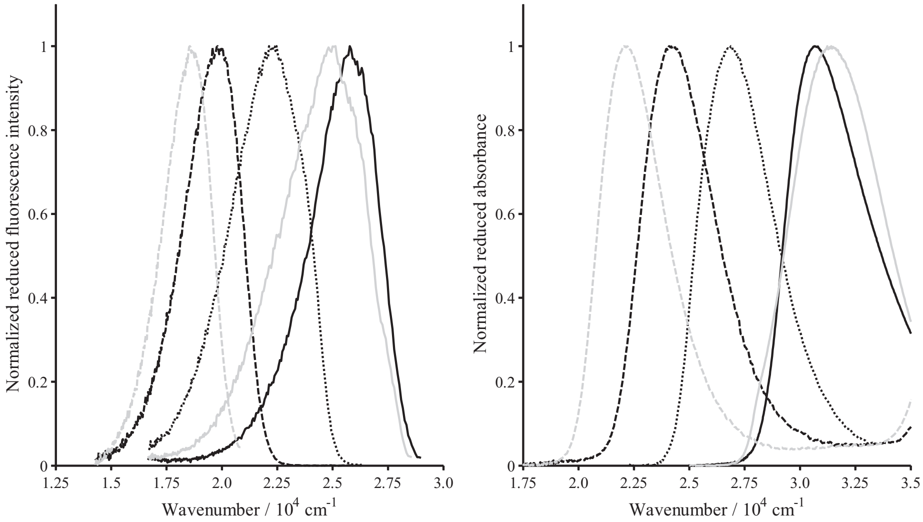


Fig. 1. Room temperature absorption (right) and fluorescence (left) spectra in ACN solutions. BOO: solid black; POO: solid gray; BOP: dotted black; BPP: dashed black; and PPP: dashed gray.

material are dramatically changed if those are involved in the electronic transitions. Recently, cyano anilines have been proposed to serve as probes in biological media as some show effective water induced fluorescence quenching [9]. Some interesting derivatives with other potential applications have also been recently synthetized [22]. For example, 2,5-diamino-3,6-dicyanopyrazine has been proposed as either a good material for electroluminescent devices or a precursor for them due to its high luminescence yield also in the solid state and its reactivity [23–25]. Therefore, the clarification of the photophysics of the monomers and of the specific role of each group is essential prior to any attempt of their use for other purposes.

Despite the quite large amount of data collected about the photophysics of these molecules, several questions remain open: (i)

Table 1
Experimental photophysical properties in several solvents at room temperature. A stands for absorption, F for fluorescence, and P for phosphorescence; ε is the absorption extinction coefficient at the absorption maximum; φ_F and τ_F are the fluorescence quantum yield and lifetime respectively; k_r is the radiative rate constant; k_{nr} the sum of all non-radiative deactivation processes of the excited singlet; k_r^{SB} is the radiative rate constant calculated according to the Strickler–Berg equation; M_A and M_F are respectively the transition dipole moments of absorption and emission as calculated from the corresponding spectra. *Not measured due to limited solubility.

		$\lambda_{\max}$ (A)/nm	ε/M^{-1} cm^{-1}	$\lambda_{\max}$ (F)/nm	φ_F	τ_F/ns	k_r/ns^{-1}	k_{nr}/ns^{-1}	k_r^{SB}/ns^{-1}	M_A/D	M_F/D	$\lambda_{\max}$ (P)/nm
BOO	ACN	325	11100	380	0.26	1.7	0.15	0.44	0.15	3.45	3.51	
	DMF	338	12500	387	0.24	1.6	0.15	0.48	0.14	3.44	3.59	
	THF	327	12400	373	0.29	2.0	0.15	0.35	0.17	3.62	3.35	530
	DEE	321	10000	364	0.35	2.5	0.14	0.26	0.16	3.36	3.14	
POO	ACN	317	16900	388	0.34	2.2	0.15	0.30	0.213	4.34	3.73	
	DMF	327	18900	386	0.32	2.2	0.15	0.31	0.207	4.34	3.67	
	THF	318	20100	386	0.29	2.0	0.15	0.36	0.242	4.69	3.63	560
	DEE	312	16600	388	0.37	2.3	0.16	0.27	0.244	4.65	3.78	
BOP	ACN	371	6000	437	0.71	14.5	0.049	0.020	0.052	2.54	2.46	
	DMF	383	6700	449	0.78	13.4	0.058	0.017	0.055	2.70	2.77	
	THF	375	6300	437	0.75	13.0	0.058	0.019	0.056	2.63	2.66	550
	DEE	370	6100	433	0.72	12.6	0.057	0.022	0.056	2.55	2.58	
BPP	ACN	411	4700	496	0.62	19.5	0.032	0.020	0.033	2.41	2.38	
	DMF	428	5100	517	0.67	21.0	0.032	0.016	0.030	2.46	2.53	
	THF	420	4900	498	0.70	21.0	0.033	0.014	0.034	2.49	2.46	
	DEE	414	*	488	0.76	21.5	0.035	0.011	*	*	2.46	
PPP	ACN	449	5900	528	0.59	23.0	0.026	0.018	0.034	2.66	2.33	
	DMF	472	6100	557	0.39	24.5	0.016	0.025	0.031	2.79	1.99	
	THF	465	6300	539	0.40	23.0	0.017	0.026	0.034	2.75	1.98	550
	DEE	456	7260	525	0.45	24.0	0.019	0.023	0.039	2.88	1.98	

which is the precise origin of the large spectral shift to lower energies respect to PDs without cyano substituents, (ii) why such a large increase in the quantum yields and lifetimes, and (iii) is this solely explainable in terms of the variation of the transition dipole moments with their energies or do other factors have to be invoked?

The strategy used in this work is to change the relative position of the −CN and $-NH_2$ groups in the benzene ring and compare the results to those obtained with a π-electron deficient ring: 1,4-pyrazine. The absorption, fluorescence and phosphorescence of five investigated compounds (see Scheme 1) were measured in few solvents to check for their solvatochromism though the prime intention is to compare them to each other. Some very elemental semi-empirical quantum mechanical calculations have also been performed in order to guide us through the discussion of the experimental results.

Table 2

Calculated photophysical properties in vacuum. μ_{S0}, μ_{S1}, and μ_{S2} are the state dipole moments in the ground, first, and second excited singlet states respectively; $\lambda_{S0\rightarrow S1}$ and $\lambda_{S0\rightarrow S2}$ are the transition wavelength for the two lowest singlet transition (together with the oscillator strength values f) and $\lambda_{S0\rightarrow T1}$ for the corresponding singlet–triplet transition. $\Delta q(S0\rightarrow S1)$ are the charge density changes upon the noted transition in several groups of atoms of each molecule.

	μ_{S0}/D	$\lambda_{S0\rightarrow S1}$/nm ($f$)	Orbitals in transition	μ_{S1}/D	$\lambda_{S0\rightarrow S2}$/nm ($f$)	Orbitals in transition	μ_{S2}/D	$\lambda_{S0\rightarrow T1}$/nm	Orbitals in transition	$\Delta q(S0\rightarrow S1)$ CN	 NH_2	 C^{Ar-CN}	 C^{Ar-NH_2}	 C^{Ar-H}/N^{Ar}
BOO	9.9	309 (0.01)	H → L + 1 H − 1 → L	10.7	282 (0.33)	H → L	14.8	600	H → L H − 1 → L + 1	−0.029	0.076	0.009	0.011	−0.067
POO	10.3	334 (0.39)	H → L	8.9	318 (0.01)	H − 1 → L	8.4	589	H → L + 1	0.038	0.064	0.136	−0.013	−0.226
BOP	1.6	325 (0.10)	H → L H − 1 → L + 1	3.7	266 (0.19)	H → L + 1 H − 1 → L	2.3	590	H → L + 1 H − 1 → L	−0.050	0.102	−0.088	0.036	0.025
BPP	0.0	339 (0.19)	H → L	0.0	266 (0.16)	H → L + 1 H − 1 → L	0.0	611	H → L H → L + 1	−0.108	0.129	.115	0.133	−0.038
PPP	0.0	374 (0.46)	H → L	0.0	334 (0.01)	H − 1 → L	0.0	714	H → L	−0.048	0.113	−0.040	0.150	−0.175

2. Experimental and computational details

2.1. Materials

The investigated compounds have been purchased from CHEMSTEP (France) – 1,2-diamino-4,5-dicyanobenzene (BOO), 1,4-diamino-2,5-dicyanobenzene (BPP), 2,5-diamino-3,6-dicyanopyrazine (PPP), CHEMIPAN (Poland) – 1,2-diamino-3,6-dicyanobenzene (BOP), and TCI (Japan) – 2,3-diamino-5,6-dicyanopyrazine (POO), companies, respectively. Spectroscopic grade solvents – acetonitrile (ACN), *N*,*N*-dimethylformamide (DMF), tetrahydrofurane (THF), and diethyl-ether (DEE) – were purchased from Sigma–Aldrich.

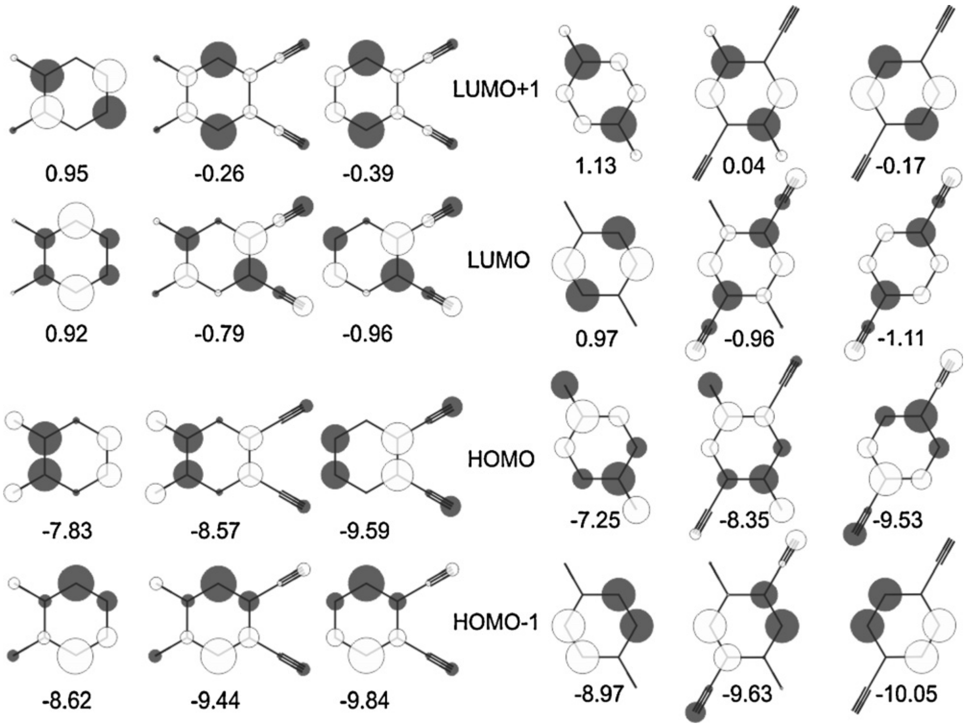
2.2. Experimental procedures

Steady-state UV–vis absorption measurements were done with a Shimadzu UV3100 absorption spectrometer. Stationary emission, luminescence and phosphorescence spectra were recorded with an Edinburgh FS 900 fluorescence spectrometer. The fluorescence decays were recorded with an Edinburgh FS 900 CDT instrument operating in TCSPC mode with measurements limit about 200 ps.

All room temperature measurements were performed in 1 cm × 1 cm fused silica cells. The solutions for the lifetime and fluorescence quantum yield measurements were prepared such that the absorbance at wavelengths equal or longer than the excitation one were smaller than 0.1. In both cases solutions were purged with solvent saturated Ar for at least half an hour. For the fluorescence quantum yields measurements a 0.05 M H_2SO_4 solution of quinine bisulfate was used as standard ($\phi_{em} = 0.51$ [26]). The lifetimes were all measured exciting at 337 nm. The fluorescence excitation spectra did coincide well with the first absorption band. The measurements at 77 K were performed in NMR like tubes by placing them in liquid nitrogen filled Dewar flask with a bottom transparent finger.

2.3. Computational details

All semi-empirical calculations were performed with the help of the program HyperChem^{TM} release 8.0.3. The ground state geometries were optimized by an AM1 calculation, whereas the transitions and excited state dipole moments and charge changes were obtained by a ZINDO/S single point calculation with 10 + 10 CI using the previously optimized geometry.



Scheme 2. Spin density distributions of the p_z orbitals of BOO (left) and BPP (right) in between the parent DCB (right) and PD (left) kernels. The surface of the circles is proportional to the density and black correspond to a positive sign. The numbers stand for the absolute energy of the orbitals in eV.

3. Results and discussion

Absorption and fluorescence emission spectra recorded at room temperature in ACN are shown in Fig. 1, in the reduced representation also called transition dipole moment [27], and the corresponding data for all solvents in Table 1. Two interesting simple facts can be inferred from it: there is a large change in the energy of the spectra on going from the BOO and POO compounds to the BPP and PPP ones, with BOP in between these two groups. The second is that there is little solvatochromism for any of the five investigated molecules. Additionally, one should note that the mirror symmetry between absorption and fluorescence is quite well fulfilled. For all three benzene derivatives absorption and emission transition dipole moments pair well, and the experimental radiative rate constant and the calculated Strickler–Berg value also coincide [28]. This is not the case for the 1,4-pyrazine derivatives where transition dipole moments for emission are slightly smaller than for absorption. This is not surprising knowing that in its *n*–π* excited state, which probably is mixed in the lowest transition with the π–π*, the 1,4-pyrazine kernel has been reported to significantly bend in the excited state after relaxation [29]. Despite the not large change in emission and absorption wavelengths, it can be noted a rather small bathochromic trend with the polarity of the solvent, except for ACN. This may be caused by specific solvent effect, more specifically H-bonding.

The above findings can be rationalized with the help of semi-empirical calculations combining AM1 and ZINDO/S methods for the geometry optimization in the ground state and estimation of

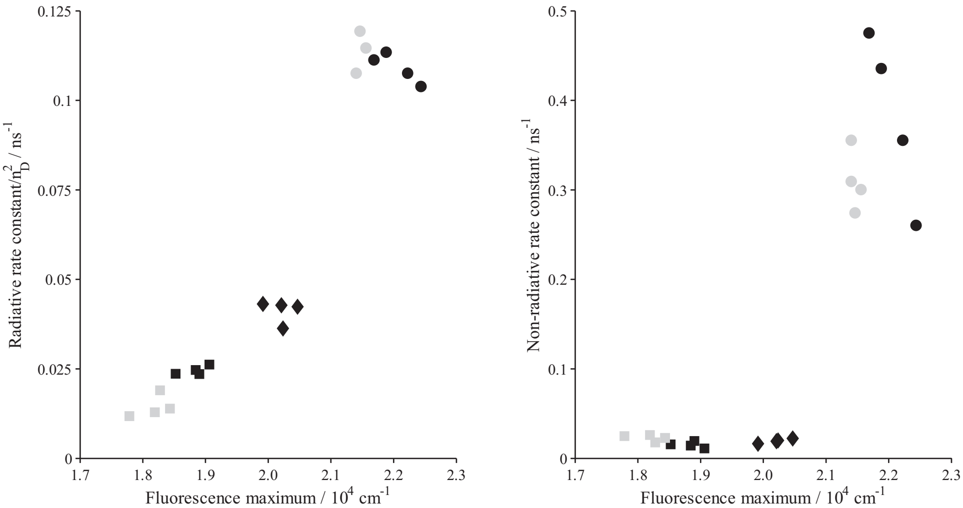


Fig. 2. Radiative (left) and non-radiative rate constants (right) at room temperature. BOO: black circles; POO: gray circles; BOP: black diamonds; BPP: black squares; and PPP: gray squares.

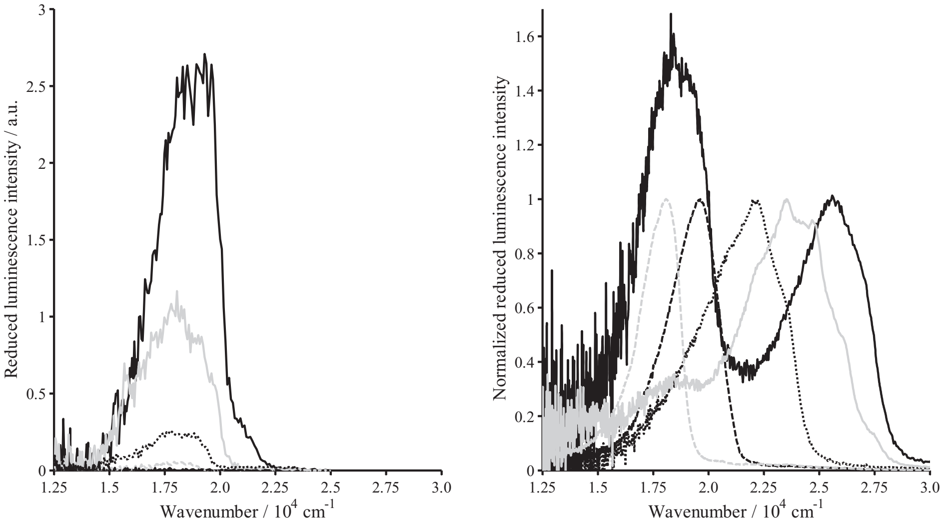


Fig. 3. 77 K phosphorescence (left) and total luminescence (right) spectra in THF glasses. BOO: solid black; POO: solid gray; BOP: dotted black; BPP: dashed black; and PPP: dashed gray.

the excited states energies and dipole moments, correspondingly. From these calculations (see Table 2) a hint of the underlying reason for the spectra position can be obtained: in the BPP and PPP compounds, the charge in the amino groups is shifted to the nitrile groups upon excitation to the lowest excited singlet state, while to a lesser extent in the BOP case and even less in the BOO and POO molecules. This charge redistribution is probably responsible for the lowering of the singlet excited state. It is interesting to note that the p_Z atomic orbitals coefficients in the ring for the HOMO and HOMO-1 are almost identical to those in the corresponding PD (donor), while for the LUMO and LUMO + 1 to those in the corresponding DCB (acceptor) in accordance to the difference in charge or spin distribution in the atoms upon excitation (Scheme 2). Though from Table 2 the contribution to the less energetic singlet–singlet transition in the gas phase of these four orbitals differs in the BOO and BPP cases (a mixture of HOMO to LUMO + 1 and HOMO – 1 to LUMO for the former and pure HOMO – LUMO for the latter) note that the next singlet transition in BOO has a larger oscillator strength and dipole moment and it is not energetically much higher than the lowest; this means that in solution the ordering of S1 and S2 can easily be reversed in this case. For BOP the second contribution is very small. Thus, for all 5 species except for BOP, in solution the first singlet–singlet transition is pure HOMO – LUMO. Additionally, it must be noted that in *o*-DCB and *o*-PD the symmetry of the HOMO and HOMO – 1 levels are almost identical. The energies of the DCBs and PDs calculated and depicted in Scheme 2 also give an idea for the red shift of the BOO and BPP compounds respect to them: the energies of the frontier orbitals roughly match for the HOMO the corresponding energy of the PD and for the LUMO of the DCB, in line with the argument of a kind of transfer or redistribution for a donor like wavefunction to an acceptor like one upon electronic excitation. The fact that there is no large solvatochromism in such case is simply due to the symmetrical nature of the mentioned charge redistribution and the consequent lack of distinct changes in permanent dipole moment values. From these calculations the ground state dipole moments of the BOO and POO molecules obtained are about 10 D, for BOP about 1.6 D and 0 D for the BPP and PPP cases, and none of them change appreciably in the excited state according to the calculations, except for BOP (3.7 D). The case of BOP points to the ortho position of the nitriles respect to the amino groups as the major factor governing the electronic distribution in both singlet states, though the relative position of the two amino groups respect to each other must play a role on the energy of its transitions.

The most remarkable difference is observed, however, in the measured quantum yields and lifetimes. As it can be seen in Table 1, these values are much larger for the BPP, PPP, and BOP molecules than for the BOO and POO ones. In order to rationalize this we have calculated the radiative and non-radiative rate constants, a representation of which as a function of the energy of the emissive transition (taken approximately to be proportional to the maximum of fluorescence), is given in Fig. 2. The radiative rate constants should follow a cubic increasing function of the transition energy [28], what is approximately the case. In fact, BOP excited state lifetime is shorter than for BPP and PPP just because its radiative rate constant is larger, as expected for its emission transition energy. The non-radiative rate constants on the other hand should decrease almost exponentially when increasing the transition energy [30] and this is obviously not the case for the BOO and POO compounds which clearly deviate from the tendency shown by the other three molecules. The reason for this departure from the expected behavior can be double: either an increase of the internal conversion rate constant or a more pronounced contribution of the intersystem-crossing to the triplet subsystem. To clarify the raised questions we have therefore performed phosphorescence measurements at liquid nitrogen temperature in THF glasses (see Fig. 3).

Two sets of data were collected: total luminescence and phosphorescence only. The motivation of the first one was to have an idea of the relative phosphorescence intensity respect to the fluorescence. From the “pure” phosphorescence measurements it is evident that the triplet levels of the BOO and POO molecules are much more efficiently populated than for the other cases (in view of the similar phosphorescence transition energies no large differences are expected in their radiative rate constants if the nature of the orbitals is the same). Thence, the higher intensities may be tentatively ascribed to larger intersystem-crossing efficiencies. Note that in the case of BOO the total luminescence intensity must be close to unity if the fluorescence quantum yield does not vary too much on freezing the solution, while this is not the case for POO most likely due to a competing IC driven by the already mentioned ring distortion in the excited singlet. It is also well possible that the triplet–singlet IC is larger in the pyrazines than in benzenes due to a larger n–π^* character of their triplet states. In any case, it is clear from the low temperature experiments that the increase of lifetime and quantum yield with a decrease of the fluorescence emission transition energy – a quite uncommon situation – is related to an increase of the intersystem-crossing.

Three explanations can be given to this increase of the ISC. The first one is a larger vibronic states density of the triplet lowest level for the molecules emitting in the blue. But one should take into account that the Franck–Condon factors are usually smaller for the higher vibronic levels. Moreover, one should expect for BOP a larger non-radiative rate constant than observed, which is practically identical to BPP and PPP. A second explanation could be related to the symmetry of the states involved in the crossing, as for example a larger difference in wavefunction symmetry in the OO cases better matching the El-Sayed's rule [31]. If this would be the case, a significant difference could be expected between pyrazines and benzenes, which is not the case here. A third explanation, and supported by our calculations (Table 2) is that at the S1 energy levels of the most phosphorescent compounds the density of electronic states of the triplet subsystem is larger what results in a better matching between the triplet and the lowest singlet levels. This would increase the probability for an inter-system crossing event via spin–orbit coupling. This agrees quite well with the fact that all phosphorescence transitions detectable take place (in contrary to the luminescence from the excited singlet states) in nearly the same spectral region. In other words: while the singlet subsystem of states is strongly affected by the charge shift from amino to nitrile groups, the triplet one seems to be relatively insensitive and therefore its states packing at the energies of the lowest excited singlet varies depending on its position. This fact is somehow at odds with the intuition that the singlet–triplet splitting is conserved for molecules of similar electronic properties if the wavefunction nature does not change from the singlet to the triplet subsystem. However, in these cases on going from OO to PP molecules this may not be the case, though a more definite statement cannot be made at the level of our calculations.

4. Conclusions

The photophysics of dicyano-diamino derivatives of benzene and 1,4-pyrazine follow similar trends. The position of the excited singlet state is argued to be a direct consequence of the electronic communication between nitrile and amino groups, more important when placed in ortho position respect to each other, what leads to a larger stabilization of such states. This may explain the red shift of the absorption spectra within this series of compounds and respect to phenylenediamines and di-cyanobenzenes. As the triplet subsystem level may not be affected in the same extent by this electronic interplay, their electronic state density in the vicinity of the excited singlet is larger for those molecules absorbing light at higher energies, providing through inter-system crossing an effective deactivation mechanism and reducing the compound fluorescence lifetime and quantum yield. Therefore, in the case of blue emitting species the non-radiative processes dominates the deactivation of the excited states and the quantum yield and lifetime are smaller than in those cases emitting at lesser energies. The fact that di-cyanobenzenes have much larger quantum yields is most likely due to the absolute lack in their case of intersystem crossing, wherever the participation in the excited state orbitals of the *n*-electrons in the phenylenediamines increase it. It is noteworthy mentioning that *o*-PD and *p*-PD have non-radiative rate constants similar to BOO, though the later has a much larger radiative rate constant (about 0.01 ns^{-1} in the case of PDs). None of the compounds here studied show remarkable solvatochromism, though specific solvation through hydrogen bonding should not be discarded.

Together with Zachariasse et al. conclusions about the charge transfer state energy level [11,12], our results may explain the long lifetimes and quantum yields of fluorescence observed in some instances on combining amino and nitrile groups directly attached to the aromatic ring: in absence of charge transfer states, the relative position of the amino and nitrile groups determine the transitions energies because of their electronic communication, and hence the accessibility to the triplet levels enhancing the intersystem crossing the higher the excited singlet state.

We expect that the current findings could help future research in the design of molecular wires and polymers with specific fluorescing and charge transporting properties.

Acknowledgements

The authors are grateful to the financial supports from the European Community (MC-RTN grant CT-2006-035533-SolarNtype) and Polish Ministry of Science and Higher Education (grant 776/6 PRUE/2008/7). Patrice Jacques (Mulhouse, France) comments and suggestions have been very helpful in the preparation of this work. GA appreciates the advices given by Michał Kijak regarding the computer quantum-mechanical calculations.

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