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Two-photon absorption properties of Zn(II) complexes: Unexpected large TPA cross section of dipolar [ZnY$_2$(4,4′-bis(*para*-di-*n*-butylaminostyryl)-2,2′-bipyridine)] (Y = Cl, CF$_3$CO$_2$)

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ABSTRACT

The two-photon absorption (TPA) properties of 4,4′-bis(*para*-di-*n*-butylaminostyryl)-2,2′-bipyridine (NBu$_2$bipy) and related dipolar ([ZnY$_2$(NBu$_2$bipy)] (Y = Cl, CF$_3$CO$_2$), [Zn(2,2′-bipyridine)$_2$(NBu$_2$bipy)][PF$_6$]$_2$) and octupolar ([Zn(NBu$_2$bipy)$_3$][PF$_6$]$_2$) complexes were investigated by the two-photon emission (TPE) technique in a femtosecond regime, working in the 730–930 nm spectral range. We found, in contrast with previous literature data, that the TPA enhancement upon coordination of a TPA active ligand to a metal center may be larger in the dipolar rather than the corresponding octupolar complex, the response being easily modulated by the choice of the ancillary ligands.

1. Introduction

Molecular nonresonant two-photon absorption (TPA) has attracted growing interest over recent years due to the many applications in material science and biological imaging [1–8]. For such reason, organic molecular engineering was directed towards TPA optimization leading to a range of molecules of various symmetries including dipoles [9], quadrupoles [10–18], octupoles [19–22], and branched structures [23–31]. As a general trend, it was evident that dipolar chromophores usually exhibit a smaller TPA cross-section with respect to quadrupolar and octupolar derivatives. However, the judicious combination of dipolar branches within a three-branched octupolar structure can induce a very intense TPA response [7]. Surprisingly, although coordination chemistry is a powerful tool to build up various symmetries including octupolar arrangements, using a wide range of metals with different oxidation states and ligands, only recently the potential of organometallic and coordination compounds in the design of TPA materials was highlighted [32–41]. Theoretical calculations (ZINDO-SOS) predicted that, working in the long wavelength region, the TPA response of 4,4′-bis(*para*-dimethylaminostyryl)-2,2′-bipyridine (NMe$_2$bipy) increases upon coordination to a Zn(II) center, the enhancement being huge in the octupolar complex [Zn(NMe$_2$bipy)$_3$][PF$_6$]$_2$ but lower in a dipolar complex such as [ZnCl$_2$(NMe$_2$bipy)] (Table 1) [38,39]. In contrast, experimentally, both Z-scan and two-photon emission (TPE) measurements evidenced that the TPA cross section of the structurally related octupolar [Zn(NBu$_2$bipy)$_3$][PF$_6$]$_2$ (NBu$_2$bipy = 4,4′-bis(*para*-di-*n*-butylaminostyryl)-2,2′-bipyridine) complex is not as high as expected (Table 1) [40,41]. Therefore, in order to get a detailed experimental evidence on the effect of various kinds of coordination on the TPA properties of NBu$_2$bipy (**1**), we prepared and characterized not only such ligand and its octupolar Zn(II) complex (**2**) but also the corresponding dipolar [ZnCl$_2$(NBu$_2$bipy)] (**3**), [Zn(CF$_3$CO$_2$)$_2$(NBu$_2$bipy)] (**4**), and [Zn(2,2′-bipyridine)$_2$(NBu$_2$bipy)][PF$_6$]$_2$ (**5**) complexes and we investigated their TPA properties (Fig. 1).

2. Experimental

Ligand **1** and complexes **2** and **3** were prepared according to the literature [42]; the new complexes **4** and **5** were obtained by reaction of NBu$_2$bipy with Zn(CF$_3$CO$_2$)$_2$·nH$_2$O and [Zn(2,2′-bipyridine)$_2$][PF$_6$]$_2$, respectively. All compounds were fully characterized by elemental analyses, ^{1}H NMR, UV–vis, and emission spectroscopies (Table 1).

*Synthesis of [Zn(CF$_3$CO$_2$)$_2$(NBu$_2$bipy)] (**4**, NBu$_2$bipy = 4,4′-bis(para-di-n-butylaminostyryl)-2,2′-bipyridine):* Zn(CF$_3$COO)$_2$·2H$_2$O (36 mg,

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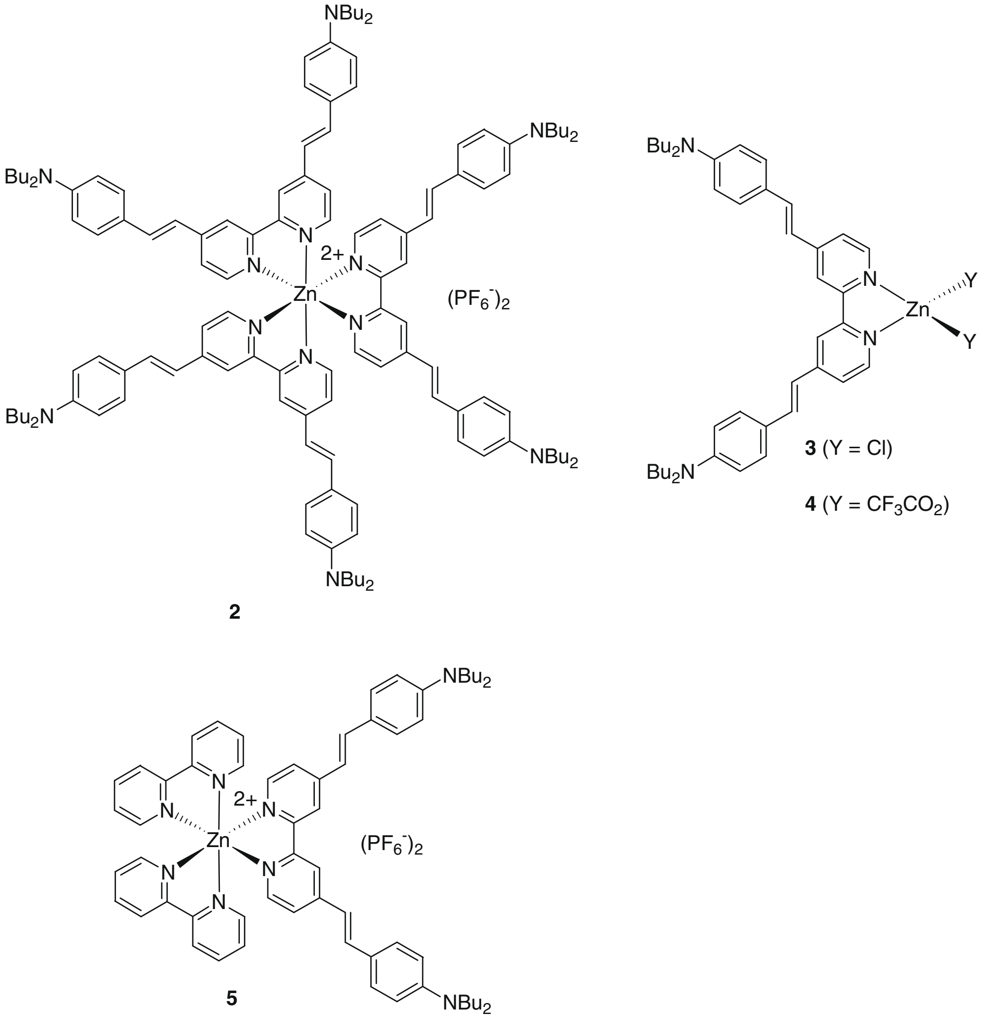
Table 1
Absorption and emission properties of bipyridines and related Zn(II) complexes.

Compound	λ_{abs} (nm)	λ_{em} (nm)	FQY	λ_{TPA} (nm)	σ_{TPA} (GM)	EF[a]	Method	Refs.
NMe_2bipy	395	–	–	736.2	493.6	–	ZINDO-SOS	[38]
$[Zn(NMe_2bipy)_3]Y_2$	463	–	–	949.2	4132.1	2.8[b]	ZINDO-SOS	[38]
Y = PF_6				822.0	9932.9	6.7[b]		
$ZnCl_2(NMe_2bipy)$	445	–	–	830.6	770.3	1.6[b]	ZINDO-SOS	[38]
1	400	493	0.087 ± 0.015	780	278 ± 57[c]	–	TPE[d]	This work
2	474	644	–	965	860 ± 20	–	Z-scan[d]	[40]
				765	1700 ± 500			
2	473	640[c]	0.18 ± 0.02	900	1018 ± 160[c]	1.2[e]	TPE[d]	This work
3	462	628	0.36 ± 0.04	920	602 ± 99	2.2[e]	TPE[d]	This work
4	463	633	0.26 ± 0.02	920	637 ± 78	2.3[e]	TPE[d]	This work
5	476[f]	640	0.12 ± 0.03	920	299 ± 84	1.1[e]	TPE[d]	This work

[a] Enhancement factor = (σTPA of the complex)/(σTPA of the NR_2bipy ligand)(number of NR_2bipy ligands in the complex).
[b] With respect to NMe_2bipy at 736 nm.
[c] Data are different from those previously reported due to a defective calibration of the fluorescence spectrometer in Ref. [41].
[d] In CH_2Cl_2.
[e] With respect to NBu_2bipy at 780 nm.
[f] There is a shoulder at 571 nm.



2

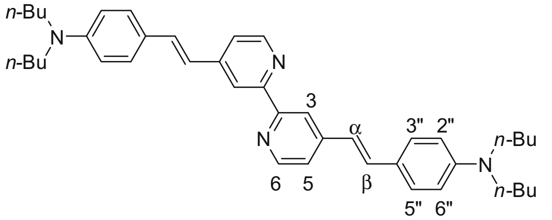
3 (Y = Cl)

4 (Y = CF_3CO_2)

5

Fig. 1. Chemical Structures of Zn(II) complexes.

0.11 mmol) was dissolved in 3 ml of EtOH. The ligand NBu_2bipy (67 mg, 0.11 mmol) was dissolved in 15 ml of CH_2Cl_2 and this solution was slowly added to the solution containing the metal salt, at room temperature. The resulting red solution was stirred at room temperature for 24 h. The solvent was partially removed in vacuum and the solution was cooled at −18 °C overnight. A red powder was filtered off, washed with *n*-hexane and cold EtOH and then dried under vacuum. Yield: 32 mg (32%). $C_{46}H_{54}O_4N_4F_6Zn$



Scheme 1. Structure of NBu_2bipy ligand (**1**) with site numbering for NMR assignment.

(906.38): calcd. C, 60.96; H, 5.96; N, 6.18; found: C, 60.76; H, 5.87; N, 5.97. ^1H NMR ($CDCl_3$): δ 8.70 (broad s, 2H, 3-H), 8.09 (broad s, 2H, 6-H), 7.55–7.48 (m, 6H, 5-H, 3′-H and 5′-H), 7.43 (d, 2H, $J = 16.1$ Hz, β-H), 6.83 (d, 2H, $J = 16.1$ Hz, α-H), 6.69 (d, 4H, $J = 8.9$ Hz, 2′-H and 6′-H), 3.37 (t, 8H, $J = 7.9$ Hz, NCH_2), 1.65 (quintet, 8H, $J = 7.6$ Hz, NCH_2CH_2), 1.42 (sextet, 8H, $J = 7.6$ Hz, $NCH_2CH_2CH_2$), 1.02 (t, 12H, $J = 7.4$ Hz, CH_3) (see Scheme 1).

*Synthesis of [Zn(2,2′-bipyridine)$_2$(NBu_2bipy)][PF_6]$_2$ (**5**, NBu_2bipy = 4,4′-bis(para-di-n-butylaminostyryl)-2,2′-bipyridine):* The complex was obtained in two steps. Step 1, synthesis of [Zn(2,2′-bipyridine)$_2$][PF_6]$_2$: 2,2′-bipyridine (70 mg, 0.45 mmol) was dissolved in 7 ml of CH_2Cl_2. $Zn(CH_3COO)_2$ (49 mg, 0.23 mmol) was dissolved in 3 ml of MeOH and added to the solution of 2,2′-bipyridine at room temperature. $NaPF_6$ (378 mg, 2.26 mmol) was added and the resulting solution was stirred overnight at room temperature. The solvent was removed in vacuum and the resulting solid was washed with H_2O to remove the excess of $NaPF_6$ and NaCl. The resulting pink powder was then washed with *n*-hexane and dried under vacuum. Yield: 81 mg (67%). $C_{20}H_{16}N_4P_2F_{12}Zn$ (667.638): calcd. C, 35.98; H, 2.40; N, 4.20; found: C, 35.56; H, 2.28; N, 4.37. ^1H NMR (CD_3CN): δ 8.53 (d, 4H, $J = 8.2$ Hz, 3-H), 8.27 (t, 4H, $J = 7.9$ Hz, 4-H), 7.96 (d, 4H, $J = 4.4$ Hz, 6-H), 7.58 (t, 4H, $J = 5.8$ Hz, 5-H). ^{31}P NMR(CD_3CN): δ −144.06 (2P).

Step 2, synthesis of [Zn(2,2′-bipyridine)$_2$(NBu_2bipy)][PF_6]$_2$: [Zn(2,2′-bipyridine)$_2$][PF_6]$_2$ (52 mg, 0.078 mmol) was dissolved in 5 ml of CH_3CN at room temperature. At the resulting pink solution was added NBu_2bipy (48 mg, 0.78 mmol) dissolved in 5 ml of CH_2Cl_2. The resulting orange solution was heated for few minutes in order to completely dissolve the ligand and the reaction mixture was stirred overnight at room temperature and then dried under vacuum. The resulting violet powder was washed with n-hexane and diethyl ether. Yield: 78 mg (78%). $C_{58}H_{70}N_8P_2F_{12}Zn$ (1282.553): calcd. C, 56.45; H, 5.67; N, 9.08; found: C, 56.17; H, 5.43; N, 8.71. ^1H NMR (CD_3CN): δ 8.61 (broad s, 2H, 3-H, NBu_2bipy), 8.52 (d, 4H, $J = 8.4$ Hz, 3-H, 2,2′-bipyridine), 8.26 (t, 4H, $J = 7.5$ Hz, 4-H, 2,2′-bipyridine), 8.07 (broad s, 2H, 6-H, NBu_2bipy), 7.95 (d, 4H, $J = 4.9$ Hz, 6-H, 2,2′-bipyridine), 7.70 (d, 2H, $J = 16.1$ Hz, β-H, NBu_2bipy), 7.58 (t, 4H, $J = 5.8$ Hz, 5-H, 2,2′-bipyridine). 7.54–7.51 (m, 6H, 5-H, 3′-H and 5′-H, NBu_2bipy), 7.02 (d, 2H, $J = 16.1$ Hz, α-H, NBu_2bipy), 6.76 (d, 4H, $J = 8.6$ Hz, 2′-H and 6′-H, NBu_2bipy), 3.38 (t, 8H, $J = 7.9$ Hz, NCH_2), 1.61 (quintet, 8H, $J = 7.6$ Hz, NCH_2CH_2), 1.40 (sextet, 8H, $J = 7.6$ Hz, $NCH_2CH_2CH_2$), 1.00 (t, 12H, $J = 7.4$ Hz, CH_3). ^{31}P NMR(CD_3CN): δ −144.06 (2P).

Linear absorption and fluorescence spectra were recorded with a Cary 5 and FluoMax P (Jobin–Yvon), respectively. Fluorescence quantum yields (FQY) were determined in CH_2Cl_2 following the procedure of Demas and Crosby [43]. Fluorescein in water at pH > 10 (FQY = 0.93) [44] and Coumarin 540A in ethanol (FQY = 0.56) were used as reference standard [45]. The TPE technique was used to determine the TPA cross-section (σ_{TPA}) in the 730–930 nm excitation wavelength range employing ultrafast (150 fs) laser pulses [46]. The instrumental set-up has been previously described [41]. Absolute values of the TPA cross section were estimated following the procedure by Xu et al. [46] and using Fluorescein in water at pH > 10 as reference standard.

3. Results and discussion

Absorption and fluorescence emission spectra of **1**, **3**, and **5** in CH_2Cl_2 are shown in Fig. 2, whereas for compound **2** and **4** are given in the Supplementary material. Typical concentrations are 1×10^{-5} M for the one-photon absorption (OPA) spectra, 1×10^{-6} M for the one-photon fluorescence spectra, and between 2×10^{-5} M and 5×10^{-5} M for the TPA spectra.

The one-photon absorption spectrum show a marked red shift of the maximum corresponding to the intraligand charge transfer (ILCT) transition upon coordination of **1** with Zn(II), whereas slighter changes are promoted by the different ancillary ligands. The red shift, also observed in the emission spectrum, is attributed to an increase of the electron-withdrawing ability of the bipyridine moiety of **1** upon coordination to Zn(II). Table 1 collects absorption and emission peaks and FQY values. The free ligand **1** shows the lowest FQY, all the coordination compounds show a net increase of the FQY, with respect to the free ligand, that follows the series: **5 < 2 < 4 < 3**. Since an increase of the FQY can be interpreted as a sign of a more rigid structure with less internal degrees of freedom, we expect that the free ligand is less rigid than all the coordination compounds, meaning that it can assume different conformations in solution that can interchange rapidly among themselves. The absorption and emission properties of compound **2** are almost identical to those of **5**, whereas **3** behaves similarly to **4.** These findings can be considered as an evidence of similar geometry and electronic properties of ligand **1** in the Zn(II) complexes when the complex is neutral (**3** and **4**) or cationic (**2** and **5**).

OPA and TPA spectra in CH_2Cl_2 for all compounds are depicted in Fig. 3, with the abscissa scale of the TPA spectra divided by two to have a direct comparison with the OPA spectra. The σ_{TPA} are expressed in Göppert-Mayer units ($1\ \text{GM} = 1 \times 10^{-50}\ \text{cm}^4\ \text{s photon}^{-1}\ \text{molecule}^{-1}$). The TPA maximum wavelengths together with the corresponding σ_{TPA} are collected in Table 1. In the TPA spectra of compounds **2**, **3**, **4**, and **5** there is a shoulder around 420–430 nm. This shoulder can be a consequence of the presence of a similar feature in the reference TPA spectrum of fluorescein in water at pH > 10 [46]. Therefore we will not discuss it any further, whereas we concentrate our attention on the main TPA maxima.

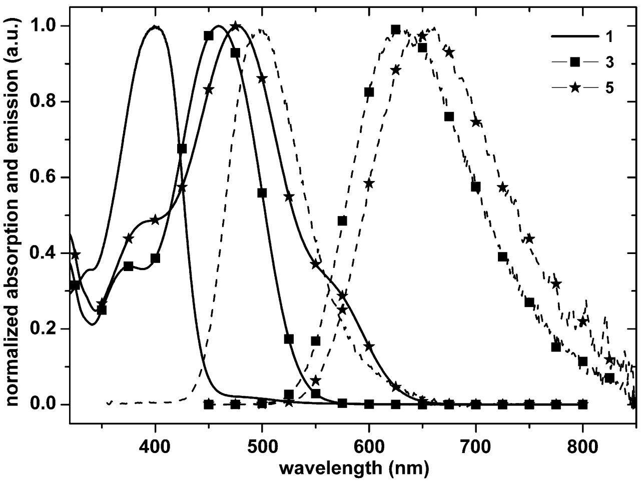


Fig. 2. Normalized linear absorption (full lines) and fluorescence (dashed lines) emission spectra of **1** (no symbol), **3** (squares) and **5** (stars) in CH_2Cl_2.

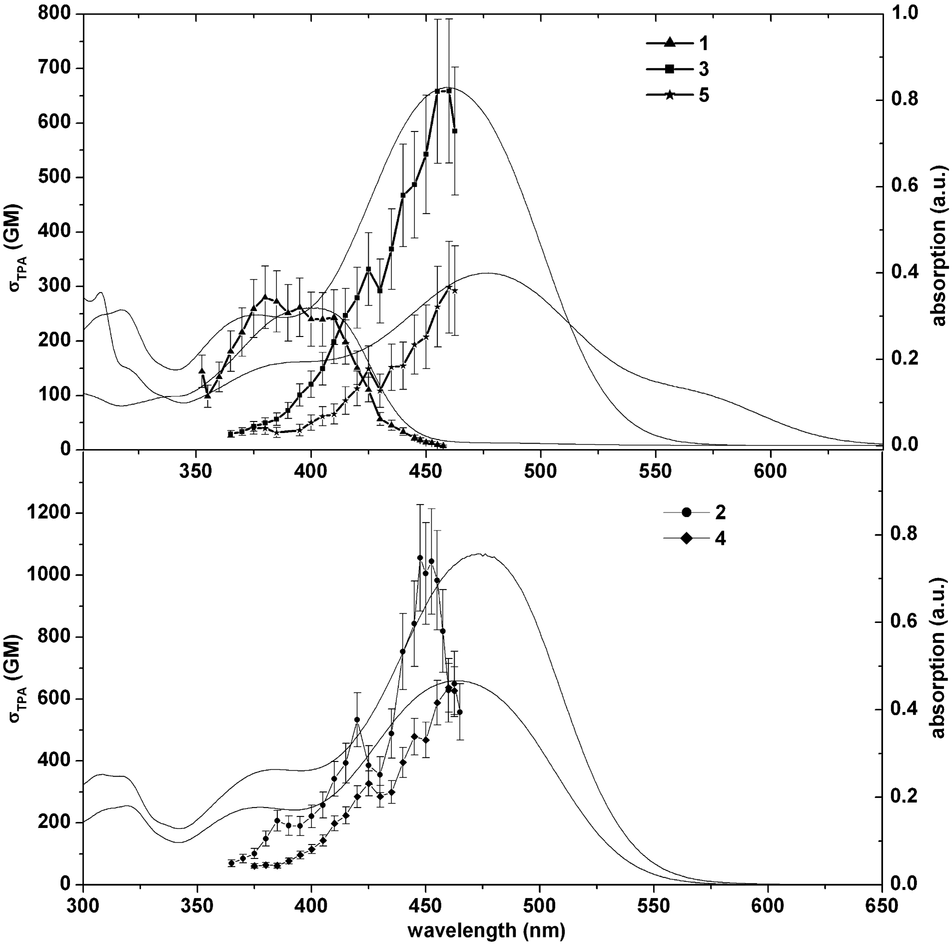


Fig. 3. One-photon absorption (full line) and two-photon absorption (line + symbol) spectra of **1** (triangles), **3** (squares) and **5** (stars) in the upper panel, and of **2** (circle) and **4** (diamonds) in CH_2Cl_2 in the lower panel. The abscissa scale of the TPA spectra is divided by two to have a direct comparison with the OPA spectra.

The strong overlap of the OPA and TPA spectra of the ligand **1** (Fig. 3) is a clear evidence that the one- and two-photon allowed excited electronic state is the same in both cases. Theoretical calculations for **1** in vacuum consider a C_{2h} symmetry; therefore the calculated OPA and TPA maxima are not coincident, as reported also in Table 1 [38]. Our experimental data do not confirm this theoretical prediction, and this can be a consequence of the ability of this ligand to easily rotate around the 2,2′-bipyridine bond, consequently it does not have a rigid C_{2h} structure in solution and is preferentially arranged in a non-centrosymmetric structure. The TPA spectrum of **2** shows a slight blue shift with respect to the OPA. Assuming a D_3 symmetry for this complex, one should expect a first excited state both one- and two-photon allowed and a second excited state which is only TPA active, as demonstrated by theoretical calculations for a similar compound and for other octupolar systems [20,38]. In the investigated spectral range (730–930 nm) the TPA spectrum of the octupolar compound **2** shows a single maximum, shifted just of approximately 20 nm with respect to the OPA maximum, suggesting that the interaction among the ligands is weak. As expected for non-centrosymmetric structures, the TPA maxima for all dipolar complexes: **3**, **4** and **5** are almost coincident with the OPA one, although for compound **5** we could only approach it, because of instrumental limits. On the other hand, since the TPA maximum is expected near to the OPA maximum, the highest value of σ_{TPA} measured should be close to the maximum TPA cross section.

In order to compare the increased TPA properties of the dipolar **3**, **4**, and **5** and the octupolar **2** complexes with that of **1**, we define as enhancement factor (EF) the maximum σ_{TPA} of the complex divided by the product of the maximum σ_{TPA} of **1** times the number of ligands **1** present in the complex. The EF shows that neutral dipolar complexes **3** and **4** display a net increase of the TPA response, whereas σ_{TPA} for the cationic complex **5** is almost equal to that of **1**. For the cationic octupolar complex **2**, there is a slight enhancement (EF = 1.2) of the TPA response with respect to ligand **1**, but there is no significant enhancement if one, more appropriately, compares the σ_{TPA} of **1** with that of complex **5** that has only one ligand **1**. This latter evidence confirms the absence of noticeable intermolecular interactions between the ligands **1** in the octupolar complex **2**, as above suggested discussing the spectral features of the TPA maxima.

Comparison of the maximum σ_{TPA} values for compound **1**, **2** and **3** with the one computed with ZINDO-SOS calculations for structurally related compounds (see Table 1) leads to the following considerations [38,39]. The smaller experimental value found for σ_{TPA} of **1** can be attributed to a theoretical value computed just for the C_{2h} conformer, while, as above discussed, in solution **1** is present as a mixture of conformers whose TPA cross section can be lower than the one arising solely from the C_{2h} conformer. For the dipolar compound **3**, instead, the experimental and calculated σ_{TPA} values cannot be compared because they refer to different excited electronic states. In our experimental data, corresponding to an excited state at 920 nm, we observe a substantial coincidence of the OPA and TPA maxima, whereas the calculated TPA values are referred to an excited state that falls at 830 nm, which we do not observe in the experimental TPA spectrum. The σ_{TPA} value observed for the octupolar complex **2** is in complete contrast with the theoretical predictions. The ZINDO-SOS calculations predict two maxima (see Table 1): the first one belongs to the same transition observed in the OPA spectrum and shows a cross section that is eight times more intense than the one calculated for the free ligand **1** (EF = 2.8), the second one is due to a TPA allowed transition and has an even more intense cross section (EF = 6.7). The theoretical prediction of a tremendous enhancement of the TPA cross section, due to a strong interaction among the ligands in the octupolar compound, is totally disattended by our experimental data which show only one TPA maximum whose cross section is just three time more intense than the one of ligand **1**. The TPA cross section of compound **2** in CH_2Cl_2 has been also measured with the Z-scan technique at two wavelengths: 765 and 965 nm, as reported in Table 1 [40]. The TPA cross section at 965 nm is outside the range that we can explore, whereas the one at 765 nm can be compared with our measurements. At 765 nm there is not a maximum in our spectrum, the σ_{TPA} is just 200 GM, strongly lower than the value of 1700 GM obtained with the *Z*-scan technique. However it is known that the TPA cross section measured with these two techniques can differ markedly when different two-photon processes can contribute to the σ_{TPA} value. The TPE technique is sensitive to just the purely coherent two-photon absorption process, while *Z*-scan can also be affected by sequential two-photon absorption, arising from the first excited state. The probability of the latter mechanism increases as the one-photon transition is approached [47].

4. Concluding remarks

This study puts in evidence the large unexpected TPA activity of the simple and easily prepared dipolar neutral $[ZnY_2(NBu_2bipy)]$ complexes, which appear as very attractive building blocks for the design of TPA materials with practical applications. It opens a new route for the design of TPA active convenient dipolar metal complexes and it represents a springboard even for the design of photoswitchable materials. In fact, an efficient photoswitching of the TPA properties of dipolar photochromic Zn(II) complexes is expected on the basis of their second order nonlinear optical properties [48] and on the prediction that an increase of either the conjugated chain length of the bipyridine ligands or the acceptor properties of the Zn(II) center by a careful choice of the ancillary ligands would improve the TPA properties of these Zn(II) complexes.

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Appendix A. Supplementary material

One photon absorption and emission spectra of the octupolar complex **2** (Fig. A1) and of the dipolar complex **4** (Fig. A2) in dichloromethane.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.cplett.2009.05.033.

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