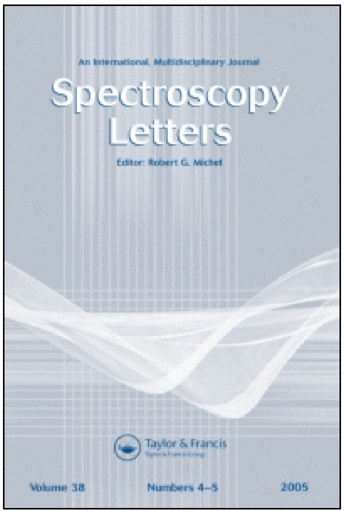

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Photoluminescence of Novel Zinc Complexes with Long-Chain Mono (Hexadecyl, Octadecyl, Eicosyl, and Docosyl) Phthalate

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Photoluminescence of Novel Zinc Complexes with Long-Chain Mono (Hexadecyl, Octadecyl, Eicosyl, and Docosyl) Phthalate

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Abstract: Ortho phthalic anhydride was modified with long-chain alcohols (1-hexadecanol, 1-octadecanol, 1-eicosanol, and 1-docosanol) to the corresponding long-chain phthalate monoester, that is, monohexadecyl phthalate (16-Phth), monooctadecyl phthalate (18-Phth), monoeicosyl phthalate (20-Phth), and monodocosyl phthalate (22-Phth), respectively. The zinc complexes with these four long-chain phthalate monoester ligands were synthesized and characterized by elemental analysis, IR and ^{1}H-NMR spectroscopy. The photophysical properties of these complexes were studied in detail with ultraviolet–visible absorption spectra and fluorescent spectra. Fluorescence studies showed that these zinc complexes exhibit intense violet photoluminescence in the solid state and may be potential candidates for photoactive materials or devices.

Keywords: Zinc complexes, long chain mono-phthalate, photoluminescence

INTRODUCTION

Luminescent metal complexes are currently of interest due to their potential application in many areas of chemistry, biology, medicine, and materials sciences.[1–7] Luminescent materials are key color components and may act as fluorescent sensors for certain aromatic molecules that absorb energy in

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the UV region or near the UV region. During the past decades, extensive research on metal complexes based on dipyridylamine,[8–14] azaindole,[11,12] pyridine–phenol,[15–18] oxadiazole,[19,20] and aromatic carboxylate[21] has been established. Aromatic carboxylic acids show many important advantages over other organic ligands in the construction of transition metal–organic complexes. For example, 1,2 or 1,3-benzenedicarboxylates phthalates (or isophthalates) and their analogues, which have two carboxyl groups that may be completely or partially deprotonated to introduce rich coordination sites.[22] Compared with isophthalate, 1,3-phenylenediacetate possesses two longer groups ($-CH_2COO-$ vs. $-COO-$) in the same positions. It is observed that the complex of 1,3-phenylenediacetate ligands possess unique 1D chain structure and less coordination modes while the transition metal complexes based on isophthalate ligands generate frameworks with various structural motifs, such as 1D helical chain and 3D channel-like network,[23] suggesting that the length of ligands may have a significant effect on the construction of coordination framework. Furthermore, the two carboxyl groups of the dicarboxylic acids such as phthalic acid afford the possibility of selective modifications. For example, long-chain alcohols can be used to modify one of the carboxyl groups to achieve the corresponding long-chain ester, which may be endowed with the favorable ability of film formation for practical applications in molecular materials or devices. But it is surprising that little attention has been paid to the relationship of the length of ligands with the photophysical properties of complexes.

In this paper, we use phthalate derivatives as the ligands to investigate the luminescent properties of zinc complexes, because phthalate derivatives with long-chain groups have been found to exhibit favorable properties of luminescence and film formation.[24] Using ortho phthalic anhydride as starting material, four long-chain alcohols (1-hexadecanol, 1-octadecanol, 1-eicosanol, and 1-docosanol) were used to yield their monoesters, that is, monohexadecyl phthalate (16-Phth), monooctadecyl phthalate (18-Phth), monoeicosyl phthalate (20-Phth), and docosyl phthalate (22-Phth), respectively. The corresponding zinc complexes with these ligands were synthesized and characterized. The photophysical properties for these long esters and their zinc complexes were studied in detail and are expected to have practical application to fabricate the functional Langmuir-Blodgett films.

MATERIALS AND METHODS

The synthesis of mono-L phthalates was achieved by a procedure similar to that described in Ref. 25: Ortho phthalic anhydride (2.96 g/mmol) was mixed with equimolar amount of long-chain alcohols [1-hexadecanol (4.85 g), 1-octadecanol (5.41 g) 1-eicosanol (5.97 g) and 1-docosanol (6.53 g)] in a flask. Then the solid mixtures were placed in an oil bath at the temperature 110∼115°C and reacted for 15 hr. Finally, the samples were recrystallized

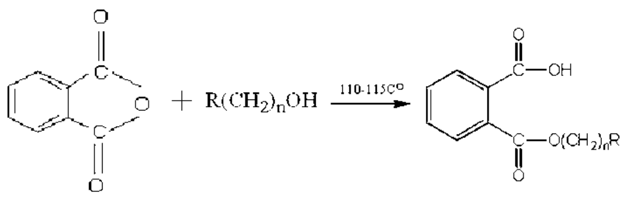

Figure 1. Synthesis of long-chain ester via reaction of ortho phthalic anhydride with alcohols (R=CH$_3$, n = 15, 17, 19, 21).

with *n*-hexane three times to afford the products (as shown in Fig. 1). The product composition was confirmed by elemental analysis: Calcd. for $C_{24}H_{38}O_4$ (mp: 65°C) C, 73.81; H, 9.81; Found: C 74.21; H, 9.20; ^{1}H-NMR 7.97 (1H), 7.75 (1H), 7.60 (2H), 4.33 (2H), 3.49 (2H), 2.24 (26H), 1.18 (3H), 10.50 (–COOH). For $C_{26}H_{42}O_4$ (mp: 68°C): C, 74.60; H, 10.11; Found: C, 74.95; H, 9.59; ^{1}H-NMR 7.97 (1H), 7.76 (1H), 7.59 (2H), 4.34 (2H), 3.35 (2H), 2.24 (2H), 1.73 (2H), 1.25 (26H) 0.89 (3H), 10.90 (–COOH). For $C_{28}H_{46}O_4$ (mp: 70°C): C, 75.20; H, 10.38; Found: C, 75.61; H, 9.58; ^{1}H-NMR 7.97 (1H), 7.70 (1H), 7.60 (2H), 4.30 (2H), 3.47 (2H), 2.17 (2H), 1.77 (2H), 1.26 (28H), 0.88 (3H), 11.20 (–COOH). Calcd. for $C_{30}H_{50}O_4$ (mp: 78°C): C, 75.90; H, 10.62; Found: C 77.74; H, 10.18: ^{1}H-NMR 7.97 (1H), 7.71 (1H), 7.60 (2H), 4.35 (2H), 3.50 (2H), 2.21 (2H), 1.75 (2H), 1.25 (30H), 0.98 (3H), 10.30 (–COOH).

Long-chain phthalate monoester (0.195 g for 16-Phth, 0.209 g for 18-Phth, 0.223 g for 20-Phth, and 0.237 g for 22-Phth; 0.5 mmol) was dissolved into the aqueous solution of Na_2CO_3 (0.1 mol L^{-1}). Then an aqueous solution of $Zn(CH_3COO)_2 \cdot 2H_2O$ (110 mg, 0.5 mmol) was added very slowly to the above solution with vigorous stirring, resulting in white solid products (as shown in Fig. 2). After 1 hr of stirring, the product was filtered, washed

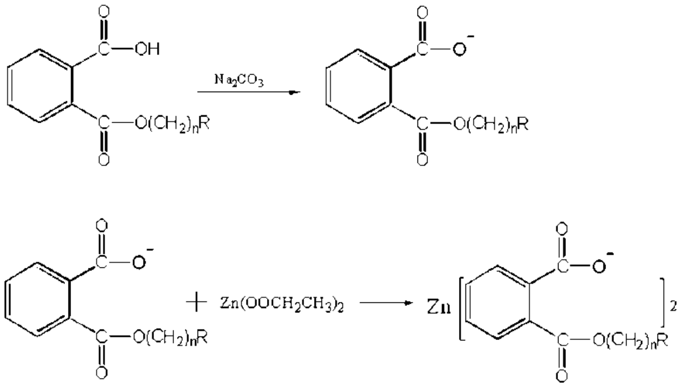

Figure 2. Synthesis of zinc complexes with long-chain esters (R=CH$_3$, n = 15, 17, 19, 21).

with ethanol and water repeatedly, and dried in a desiccator. The composition of the complexes was confirmed by elemental analysis: Calcd for Zn(16-Phth)$_2$: C, 68.27; H, 8.83; Found: C, 68.00; H, 8.59. Calcd for Zn(18-Phth)$_2$: C, 69.35; H, 9.18; Found: C, 69.08; H, 8.86. Calcd for Zn(20-Phth)$_2$: C, 70.30; H, 9.48; Found: C, 70.03; H, 9.21. Calcd for Zn(22-Phth)$_2$: C, 71.15; H, 9.75; Found: C, 71.42; H, 9.53.

Elemental analyses (C, H) were carried out by the Elementar Vario EL elemental analyzer (Germany). Infrared spectroscopy with KBr pellets was performed on a Nicolet Nexus 912 AO446 model spectrophotometer (USA) in the $4000 \sim 400\,\mathrm{cm}^{-1}$. ^{1}H-NMR spectra were recorded in chloroform on a Bruker AVANCE-500 spectrometer (Switzerland) with tetramethylsilane (TMS) as internal reference. Ultraviolet absorption spectra were obtained with an Agilent 8453 spectrophotometer (USA). The fluorescence (excitation and emission) spectra were determined with Perkin-Elmer LS-55 spectrophotometer (USA): excitation slit width = 10 nm, emission slit width = 5 nm.

RESULTS AND DISCUSSION

Table 1 gives the data of IR spectra. All the IR spectra of these zinc complexes show similar features. For example, for the Zn(16-Phth)$_2$, the characteristic absorption peaks of the carboxylic group COO$^-$ appear at $1593\,\mathrm{cm}^{-1}$ for ν_{as}(COO−) and $1405\,\mathrm{cm}^{-1}$ for ν_s(COO−), respectively, whereas these peaks are not present in the spectra of 16-Phth ligand, suggesting that the oxygen atoms of mono 16-Phth carbonyl group are coordinated with Zn^{2+}. Both the free ligand and its complexes show characteristic absorption bands of the carbonyl group at $1743\,\mathrm{cm}^{-1}$ (s) for 16-Phth and $1724\,\mathrm{cm}^{-1}$ for Zn(16-Phth)$_2$ except for a weaker absorption intensity of the complex than that of 16-Phth, which indicates that there still exists one long-chain ester carbonyl group, and this carbonyl groups coordinates with zinc ions. For the Zn(18-Phth)$_2$ system, the characteristic absorption peaks of the carboxylic group appear at $1589\,\mathrm{cm}^{-1}$ for ν_{as}(COO−) and $1401\,\mathrm{cm}^{-1}$ for ν_s(COO−), respectively, these peaks are not observed in the spectra of free 18-Phth ligand, indicating that the oxygen atoms of mono 18-Phth carbonyl group are coordinated with Zn^{2+}. The characteristic absorption band of carbonyl group shifts from high wave number of $1744\,\mathrm{cm}^{-1}$ for 18-Phth to a lower value of $1724\,\mathrm{cm}^{-1}$ for Zn(18-Phth)$_2$, and the absorption intensity is weaker for complexes than that of 18-Phth, which suggests that there still exists one carboxyl group of long-chain ester. For the Zn(20-Phth)$_2$, the characteristic absorption peaks of the carboxylic group appear at $1585\,\mathrm{cm}^{-1}$ for ν_{as} (COO−) and $1405\,\mathrm{cm}^{-1}$ for ν_s(COO−), respectively. These peaks are not present in the spectrum of the free 20-Phth ligand, which indicates that the oxygen atoms of mono 20-Phth carbonyl group are coordinated with Zn^{2+}. The free ligand and its complexes show the characteristic absorption bands of the carbonyl group at $1749\,\mathrm{cm}^{-1}$ (strong) and $1724\,\mathrm{cm}^{-1}$, respectively,

Table 1. The IR spectra and band assignments of the zinc complexes

Compounds	$\nu_{c=o}$ (cm^{-1})	ν_s(COO−) (cm^{-1})	ν_{as}(COO−) (cm^{-1})	ν_s(C−O−C) (cm^{-1})	ν_{as}(C−O−C) (cm^{-1})
16-Phth	1742.70(s)			1074.00	1262.76
Zn(16-Phth)$_2$	1723.95(w)	1405.22	1593.19	1082.41	1274.46
18-Phth	1743.78(s)			1126.49	1282.16
Zn(18-Phth)$_2$	1723.95(m)	1401.14	1589.10	1082.41	1274.46
20-Phth	1749.19(s)			1074.59	1249.37
Zn(20-Phth)$_2$	1723.95(m)	1405.22	1585.02	1078.32	1270.37
22-Phth	1742.70(s)			1074.59	1288.65
Zn(22-Phth)$_2$	1719.86(w)	1405.22	1589.10	1082.41	1278.55

and the intensity of the latter is much weaker than that of complexes, verifying that there still exists one carboxyl group of long-chain ester that also takes part in the coordination with zinc ions. For the Zn(22-Phth)$_2$, the characteristic absorption peaks of the carboxylic group appear at 1589 cm^{-1} for ν_s(COO−) and 1405 for ν_{as}(COO−), respectively, while these peaks do not appear in the spectra of free ligand 22-Phth, suggesting that the oxygen atoms of mono 22-Phth carbonyl group are coordinated with Zn^{2+}. The characteristic absorption bands of the carbonyl group for complexes (1720 cm^{-1}) show blue shift compared with its corresponding ligand (1742 cm^{-1}, strong), which indicates that there still exists one carboxyl group of long-chain ester.

Figure 3 shows the ultraviolet–visible absorption spectra for the four long-chain esters (16-Phth, 18-Phth, 20-Phth, and 22-Phth). They all exhibit domain absorption peaks in the ultraviolet region in the range 200–400 nm, and the maximum absorption peaks are located at 249 nm (16-Phth), 247 nm (18-Phth), 243 nm (20-Phth), and 243 nm (22-Phth), respectively. Besides, one shoulder peak is observed at around 277 nm for three long-chain ligands (16-Phth, 20-Phth, and 22-Phth), which is not apparent for the 18-Phth. Both absorptions are ascribed to the characteristic peaks of phenyl ring with long-chain substitutes. The small difference in the band position and intensity is due to the different chain length (16-Phth–18-Phth–20-Phth). It can be observed that the maximum absorption bands exhibit small blue shifts with the increase

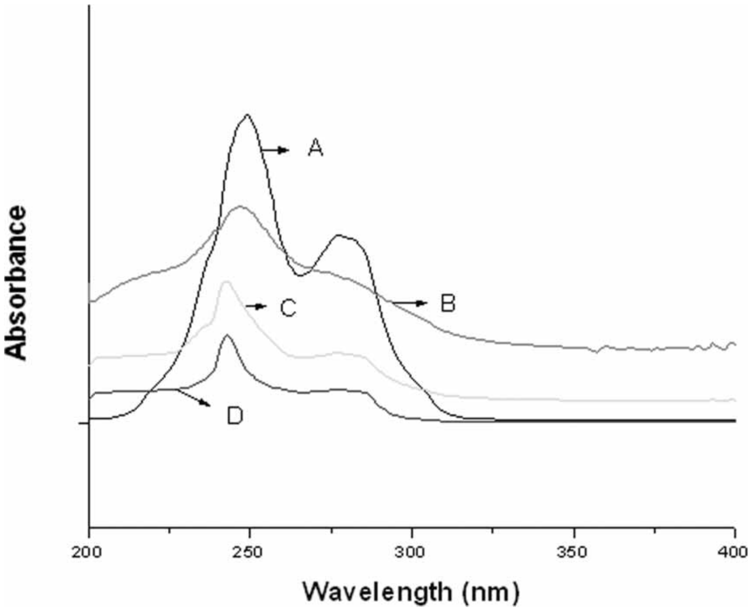

Figure 3. Ultraviolet–visible absorption spectra of 16-Phth (A), 18-Phth (B), 20-Phth (C), and 22-Phth (D).

of chain length, suggesting that the increase of substituted alcohol chain length influences the electron distribution in along the phenyl ring, but this effect becomes not distinct when the length of the alcohol chain increases to some extent (22-Phth). Figure 4 presents the absorption spectra of the four zinc complexes: the maxima 244 nm for Zn(16-Phth)$_2$, 245 nm for Zn(18-Phth)$_2$, 242 nm for Zn(20-Phth)$_2$, and 243 nm for Zn(22-Phth)$_2$, are ascribed to $\pi \rightarrow \pi^*$ transition.

The excitation spectra of these zinc complexes show that they have effective absorption in the region 200–400 nm and mainly located around 240 and 330 nm. As shown in Fig. 5, the maximum excitation peaks under the violet emission of 420 nm are located around 243 nm for Zn(16-Phth)$_2$, 241 nm for Zn(18-Phth)$_{2,}$, 240 nm for Zn(20-Phth)$_2$, and 239 nm for Zn(22-Phth)$_2$, respectively. It can also be observed that the maximum absorption bands exhibit slight blue shift with the increase of chain length, suggesting that the increase of substituted alcohol chain affects the electron distribution along the phenyl ring. Besides, a broad band from 308 to 360 nm, which may arise from charge transfer and is observed for Zn(16-Phth)$_2$ and Zn(18-Phth)$_2$, whereas it is not apparent in spectra of Zn(20-Phth)$_2$ and Zn(22-Phth)$_2$. So the charge transfer may have some relationship with the length of the alcohol chain. We recorded the corresponding emission spectra by selective excitation. The striking feature of the four zinc complexes is that

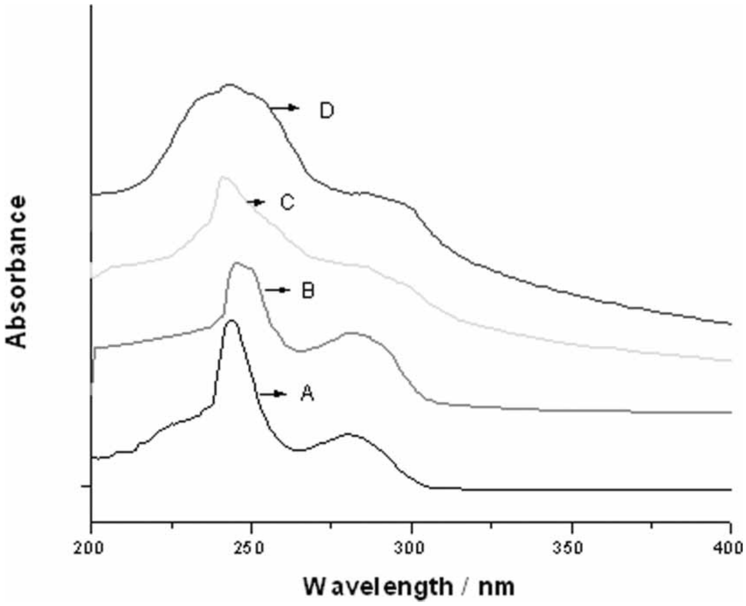

Figure 4. Ultraviolet–visible absorption spectra of Zn(16-Phth)$_2$ (A), Zn(18-Phth)$_2$ (B), Zn(20-Phth)$_2$ (C), and Zn(22-Phth)$_2$ (D).

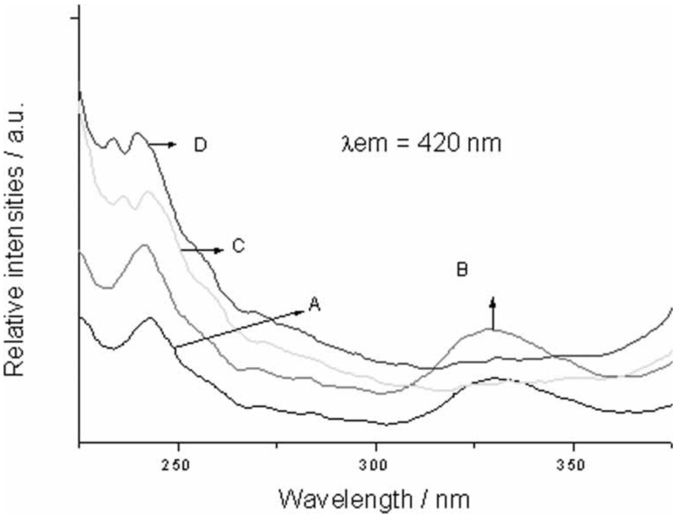

Figure 5. Excitation spectra of Zn(16-Phth)$_2$ (A), Zn(18-Phth)$_2$ (B), Zn(20-Phth)$_2$ (C), and Zn(22-Phth)$_2$ (D) ($\lambda_{em} = 420$ nm).

they exhibit intense violet photoluminescence upon the irradiation of UV light in the solid state at room temperature, as shown in Fig. 6. They all exhibit broad emission band from 375 to 450 nm with maxima at 390 nm for Zn(16-Phth)$_2$, 391 nm for Zn(18-Phth)$_2$, 395 nm for Zn(20-Phth)$_2$, and 391 nm for Zn(22-Phth)$_2$. Figure 7 shows the emission spectra of 16-Phth and Zn(16-Phth)$_2$. The emission broad band of Zn(16-Phth)$_2$ mainly ranges from 375 to 450 nm with the maximum emission wavelengths at 390 nm while the emission spectrum of the free 16-Phth molecule shows one emission at 334 nm, indicating that the 16-Phth ligand has no emission in the visible region, but when it is bound to a zinc center, violet luminescence is observed. The lower energy band would be assigned to the ligand-to-metal charge transfer (LMCT),[23] and the observed luminescence of the complex is attributed to the coordinate 16-Phth ligand. Furthermore, the luminescence intensity of the Zn(16-Phth)$_2$ is greatly increased compared with that of the free ligand due to the ligand chelating to the metal ion, which effectively increases the rigidity of the 16-Phth. The same results can also be obtained for the other three complexes. As shown in Table 2, The red shift (Δ) between complex and ligand is 54, 63, 68, and 71 nm for Zn(16-Phth)$_2$, Zn(18-Phth)$_2$, Zn(20-Phth)$_2$, and Zn(22-Phth)$_2$, respectively, enhancing with the increase of chain length, which may suggest that the increase of substituted alcohol chain affects the electron distribution along the phenyl ring.

In summary, four long-chain ester ligands have been synthesized by the modification of ortho phthalic anhydride with long-chain alcohols, and their complexes with Zn^{2+} have been prepared. The photophysical properties

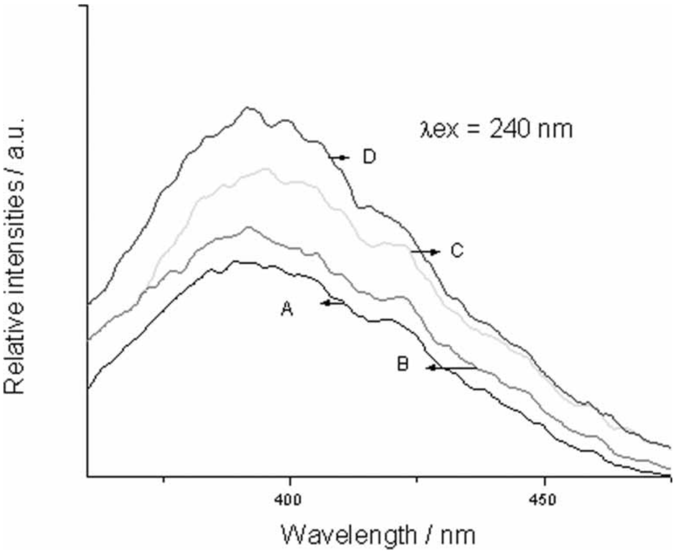

Figure 6. Emission spectra of Zn(16-Phth)$_2$ (A), Zn(18-Phth)$_2$ (B), Zn(20-Phth)$_2$ (C), and Zn(22-Phth)$_2$ (D) ($\lambda_{ex} = 240\,\text{nm}$).

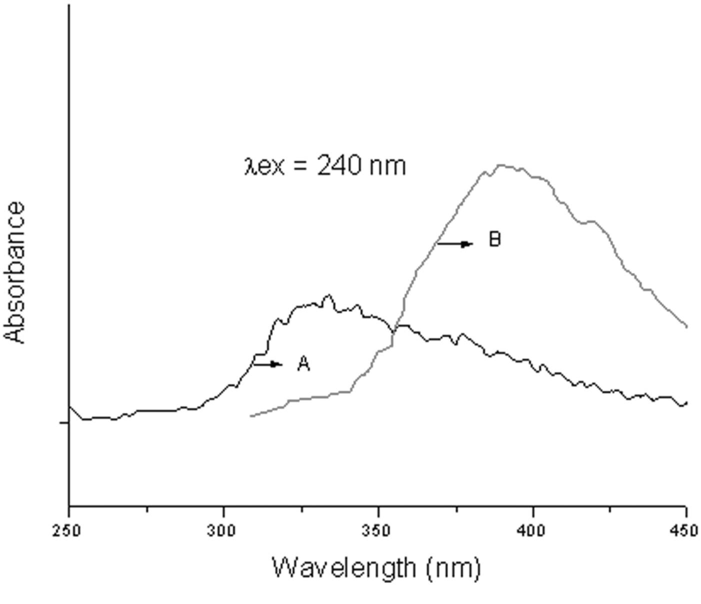

Figure 7. Emission spectra of 16-Phth (A), and Zn(16-Phth)$_2$ (B) ($\lambda_{ex} = 240\,\text{nm}$).

Table 2. The maximum emission wavelengths of the four ligands and their corresponding complexes

Compounds	λ_{max} (nm)	$\Delta\lambda$
16-Phth	334	56
Zn(16-Phth)$_2$	390	
18-Phth	329	63
Zn(18-Phth)$_2$	392	
20-Phth	327	68
Zn(20-Phth)$_2$	395	
22-Phth	321	71
Zn(22-Phth)$_2$	392	

have been studied by ultraviolet as well as excitation and emission spectroscopy. The fluorescence excitation and emission spectra studies reveal that the four compounds exhibit a strong violet emission in the solid state at room temperature, and the increase of substituted alcohol chain has an effect on the photophysical properties of the complexes. The four zinc complexes may be potential candidates as photoactive materials.

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