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Effect of non-peripheral alkyloxy substituents on the structure and spectroscopic properties of metal-free phthalocyanines

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Abstract

Four non-peripheral alkyloxy substituted metal-free phthalocyanines have been synthesized and characterized with X-ray crystal structure, IR, UV–vis and ^{1}H NMR. It is found that the center caves of Pc skeletons were expanded but their ring currents were reduced by the introduction of alkyloxy substituents on the non-peripheral positions.

Keywords: Substituted phthalocyanines; Structure; Spectra

1. Introduction

Phthalocyanines (Pcs) have been studied widely with respect to their importance in application as dyes, catalysts, molecular materials, chemical sensors, non-linear optical materials and photosensitizes for photodynamic therapy [1–3]. To fit these applications, it is necessary to modify the structure of Pcs, such as varying the central metal atom, changing the size of the π-conjugated system of Pcs, and alternating the type, number, and positions of the substituents on the macrocycle ligands.

It is known that the non-peripheral alkyloxy substituted Pcs always exhibit a longer wavelength Q band absorption in the near IR region than the unsubstituted Pcs for they have the larger size of effective π system [4]. However, there is no direct structure data reported to support this assume. In the present article, we describe the effect of non-peripheral alkyloxy substituents on the structure and spectroscopic properties of metal-free phthalocyanines (H_2 Pcs) based on the studies of X-ray crystal structure, IR, UV–vis and ^{1}H NMR spectra.

2. Experimental section

2.1. Materials

The substituted phthalonitriles [3-(*i*-propoxy)phthalonitrile, 3-(2,2,4-trimethyl-3-pentoxy)phthalonitrile, 3,6-dibutyloxyphthalonitrile] were synthesized following the procedure in literature [5], and 1,3,3-trichloroisoindolenine was synthesized as reported previously [6,7]. THF was dried with 4 Å molecular sieve for 24 h before used. All other reagents and solvents were of reagent grade and used without further purification and all reactions were performed under nitrogen atmosphere.

2.2. Measurements

The infrared spectra were recorded in the range of 400–4000 cm^{-1} on Perkin-Elmer FT–IR spectrophotometer. And elemental analyses were carried out on a Vario EL III elemental analyses instrument. ^{1}H NMR spectra were measured with a UNITY-500 spectrometer at

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500 MHz and UV–vis spectra were recorded on PE-Lambda9 UV–vis spectrophotometer using 1 cm path length cuvettes at room temperature.

2.3. The synthesis of 5-(2,2,4-trimethyl-3-pentoxy)-1,3-diiminoisoindole [6]

In a dried 100-mL round-bottom flask equipped with a magnetic stirrer, a reflux condenser, and an ammonia gas inlet, a solution of 3-(2,2,4-trimethyl-3-pentoxy)phthalonitrile (3 g, 11.7 mmol) and sodium (0.2 g, 8.7 mmol) in 30 mL dry methanol was placed. Ammonia was blowed through, and the solution was stirred at room temperature for 1 h. The solution was then heated to reflux for 5 h with continued addition of ammonia. After cooling to room temperature, the solvent was distilled. The residue was dissolved with toluene, washed three times with water to remove any remaining NaCl, and dried. The product was recrystallized several times with the mixing solvent of acetic ether/*n*-hexane. Yield 2.0 g, 63.0%; mp 186.0–187.8 °C; IR(KBr): 1651, 1614.7 cm^{-1} ($\nu_{C=N}$), 3303.3, 3321.3 cm^{-1} (ν_{N-H}), 1272.7, 1095.5 cm^{-1} (ν_{Ar-O-C}), 3488.6 cm^{-1} (ν_{Ar-H}), 1614.7, 1651 cm^{-1} ($\nu_{C=C}$), 2966.1, 2873.0 cm^{-1} (ν_{CH_3}).

2.4. General process of the synthesis of the symmetry metal-free phthalocyanines

A solution of substituted phthalonitriles (4 mmol) in 1-pentanol (6 mL) was heated to 120 °C, and then lithium (28 mg, 4 mmol) was added to this solution and the reaction was continued at the same temperature for 4 h. After the reaction mixture was cooled to room temperature, methanol (100 mL) and HCl (3 mL) were added to precipitate the rude product which was then purified on a silicon column with dichloromethane/acetic ether as eluent and recrystallized several times with the mixing solvent of dichloromethane/ethanol.

2.5. Tetrakis(i-propoxy)phthalocyanine (**1**)

Yield 8.4%; Anal. Calcd (%) for $C_{64}H_{82}N_8O_4$: C, 70.76; H, 5.67; N, 15.00; found C, 71.15; H, 5.68; N, 13.78; IR(KBr): 1585.6, 1491.5 cm^{-1} ($\nu_{C=N}$), 3296.4 cm^{-1} (ν_{N-H}), 1269.8, 1104.05 cm^{-1} (ν_{Ar-O-C}), 1450.77 cm^{-1} ($\nu_{C=C}$), 2971.34 cm^{-1} (ν_{CH_3}); ^{1}H NMR (ppm, $CDCl_3$): $\delta = 8.887$–8.969 (t, Ph-H, 4H), 7.924–8.067 (m, Ph-H, 4H), 7.261–7.532 (m, Ph-H, 4H), 5.1–5.5 (m, —CH—, 4H), 1.8–2.0 (m, —CH_3, 24H), −1.423 (br, N—H, 8H); UV–vis (nm, $CHCl_3$): 316.92, 695.37, 728.21.

2.6. Tetrakis(2,2,4-trimethyl-3-pentoxy)phthalocyanine (**2**)

Yield 9.1%; Anal. Calcd (%) for $C_{64}H_{82}N_8O_4$: C, 74.82; H, 8.04; N, 10.91; found C, 74.82; H, 8.07; N, 10.78; IR(KBr), 1586.9, 1491.0 cm^{-1} ($\nu_{C=N}$), 3298.8 cm^{-1} (ν_{N-H}), 1241.5, 1109.9 cm^{-1} (ν_{Ar-O-C}), 1481.3 cm^{-1} ($\nu_{C=C}$), 2956.8 cm^{-1} (ν_{CH_3}); ^{1}H NMR (ppm, $CDCl_3$): $\delta = 9.146$ (s, Ph-H, 4H), 8.047 (s, Ph-H, 4H), 7.716 (s, Ph-H, 4H), 4.762 (m, —CH—, 8H), 2.589 (m, —CH_3, 60H), −0.046 (br, N—H, 8H); UV–vis (nm, $CHCl_3$): 223.34, 319.14, 635.56, 667.38, 702.02, 731.95.

2.7. Octakisbutyloxyphthalocyanine (**3**)

Yield 16.1%; Anal. Calcd (%) for $C_{64}H_{82}N_8O_8$: C, 70.43; H, 7.574; N, 10.27; found C, 70.81; H, 7.21; N, 10.26; IR(KBr): 1597.46, 1498.67 cm^{-1} ($\nu_{C=N}$), 3298.29 cm^{-1} (ν_{N-H}), 1267.93, 1037.76 cm^{-1} (ν_{Ar-O-C}), 1464.17 cm^{-1} ($\nu_{C=C}$), 2954.64 cm^{-1} (ν_{CH_3}); ^{1}H NMR (ppm, $CDCl_3$): $\delta = 7.587$ (s, Ph-H, 8H), 4.845 (m, —CH_2—, 16H), 2.223–2.260(m, —CH_2—, 16H), 1.61–1.681 (m, —CH_2—, 16H), 1.070–1.098 (m, —CH_3, 24H), −0.80 (br, N—H, 8H); UV–vis (nm, $CHCl_3$): 330.1, 751.26, 774.68

2.8. The synthesis of dikis(2,2,4-trimethyl-3-pentoxy)phthalocyanine (**4**) [6]

A solution of 5-(2,2,4-trimethyl-3-pentoxy)-1,3-diiminoisoindole (0.6214 g, 2.27 mmol), triethylamine (0.64 mL) in dry THF (60 mL) was cooled to 0 °C, then another solution of 1,3,3-trichloroisoindolenine (0.5 g, 2.27 mmol) in dry THF (50 mL) was gradually added under a slow stream of nitrogen. The reaction was carried out with stirring for 1 h at approximately 0 °C then slowly warmed to room temperature over a 5-h period. After the insoluble triethylamine hydrochloride was removed, hydroquinone (0.25 g, 2.27 mmol) and sodium methoxide (0.2 g Na in 5 mL menthol) was added to the reaction vessel. Then a reflux condenser was equipped and the reaction solution was refluxed under nitrogen for 6 h. After being cooled to room temperature, the dark blue-black residue was filtered out. The residue was washed by boiling water and extracted in SOXHLET extractor with menthol and acetone until the extraction was clear. Then, it was further purified by recrystallization several times with chloroform. Yield 0.1949 g, 22.3%; IR(KBr):1583.1, 1490.9 cm^{-1} ($\nu_{C=N}$), 3274.9 cm^{-1} (ν_{N-H}), 1266.5, 1108.3 cm^{-1} (ν_{Ar-O-C}), 1583.1 cm^{-1} ($\nu_{C=C}$), 2955.6, 2866.4 cm^{-1} (ν_{CH_3}); ^{1}H-NMR (ppm, $CDCl_3$): $\delta = 9.213$; 7.993; 7.676 (m, m, m, 14H, Pc-H), 4.759 (m, 2H, O—CH—), 2.608 (m, 2H, —CH—), 1.346–1.318 (t, 12H, —$C(CH_3)_2$), 1.507–1.496 (s, 18H, —$C(CH_3)_3$), −1.423 (m, 2H, H—N); MS: *m/z* (M+) 771.8, 1541.8; UV–vis (nm, $CHCl_3$): 333.97, 618.21, 650.96, 680.89, 714.87.

2.9. Crystal structure determination of compounds **2**

A suitable single crystal of **2** with dimensional $0.25 \times 0.25 \times 0.12$ mm^3 was carefully selected under a polarizing microscope and glued to the tip of a glass fiber. Collection of diffraction data for compound **2** was performed on Rigaku Raxis-Rapid Imaging Plate Diffractometer with graphite-monochromated Mo—Kα ($\lambda = 0.071069$ nm) in the range of 1.80–27.48° at room temperature. The data

intensity was corrected for LP factors without absorption correction. The structure has been solved using direct methods and refined on F^2 by full matrix least-squares methods using the SHELX97 program package. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms of the Pc peripheral ring were placed on the calculated positions and hydrogen atoms on the branches of each substituent were not added. The disorders of the branches were disposed by putting six methyl groups in the structure with the sum of their occupation factors to be restrained at five. The final $R = 0.1500$ $[I > 2\sigma\ (I)]$ for 386 parameters and 7237 reflections with $I > 2\sigma\ (I)$. CCDC reference number 259449. The crystallographic data are listed in Table 1.

Table 1
Crystal data and structure refinement for compound **2**

Empirical formula	$C_{64}H_{82}N_8O_4$
Formula weight	1027.38
Temperature	293(2) K
Wavelength	0.71069 Å
Crystal system, space group	Monoclinic, $P\ 21/n$
Unit cell dimensions	$a = 13.8183(4)$ Å, $\alpha = 90.00$
	$b = 14.5996(4)$ Å, $\beta = 98.54(0)°$
	$c = 16.320(2)$ Å, $\gamma = 90.00$
Volume	3255.88(50) Å^3
Z, calculated density	2, 1.048 g/cm^3
Absorption coefficient	0.066
Crystal size	$0.25 \times 0.25 \times 0.12$ mm^3
$F(000)$	1012.0
θ range for data collection	1.80–27.48°
Limiting indices	$h = 0 \to 17$, $k = 0 \to 18$, $l = -21 \to 20$
Reflections collected/unique	21961/7237
Refinement method	Full-matrix least squares on F^2
Data/restraints/parameters	7237/386/3
Goodness-of-fit on F^2	1.019
Final R indices $[I > 2\sigma\ (I)]$	$R_1 = 0.1500$, $wR_2 = 0.3068$
Largest diff. peak and hole	0.323 and −0.310

3. Results and discussion

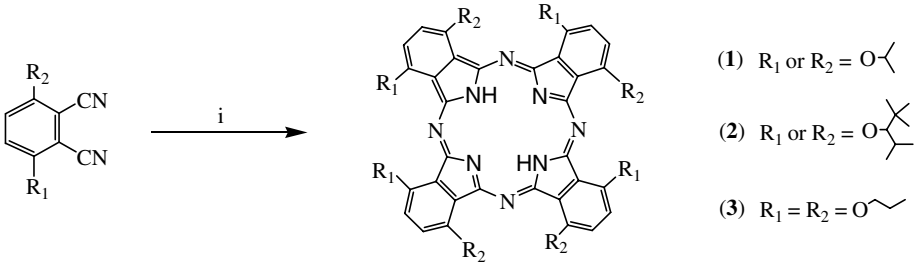
3.1. Synthesis of metal-free phthalocyanines

As illustrated in Scheme 1, preparation of the symmetry metal-free phthalocyanines (**1**–**3**) begins with the Li (I) template cyclization of substituted phthalonitriles. In the case of the tetrasubstituted **1** and **2**, the composition and distribution of their isomer mixture depend on the bulk of the substituents [8,9]. Compound **1** with *i*-propoxy showed four peaks in an HPLC chromatogram, while compound **2** with bulky 2,2,4-trimethyl-3-pentoxy only showed one peak which indicates that it has only one isomer.

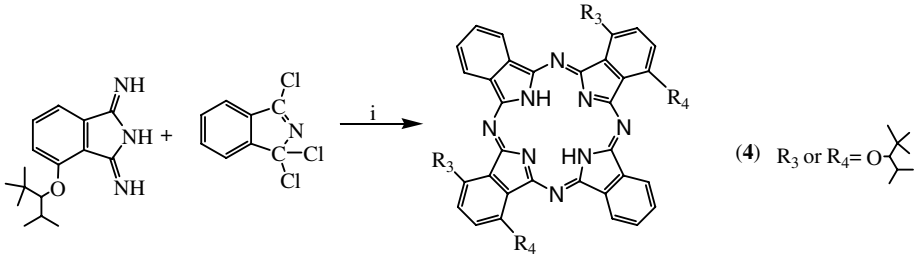
According to the synthesis routine of compound **4** shown in Scheme 2, there are two possible isomers with *cis*- and *trans*-symmetry for the production. But in fact, only one peak was found in its HPLC chromatogram and single crystal with bright purple luster was obtained from the mixture solution of THF/menthol.

3.2. X-ray crystal structures

The crystal structure of compound **1** was not determined for it is a mixture of four isomers and those of compound **3** and **4** have been reported in an earlier presentation [10,11]. However, the reflection data of **2** can only be collected in a sealed vessel for it is easily efflorescent in air, which leads to the weak reflection. Together with the disorders of the substituent branches, it results in a somewhat large deviation factor ($R = 0.1500$). Nevertheless, the ring conformation is confirmed basically, as shown in the Fig. 1a. We can see clearly that the isomer of title Pc derivative is assigned to the structure isomer C_{4h}, which the four alkoxyl groups arranged in the positions where no two groups are



Scheme 1. Reagents and conditions: (i) pentanol, Li, 120 °C, 4 h, and then conc. HCl.



Scheme 2. Reagents and conditions: (i) THF, $N(C_2H_5)_3$, 0 °C for 1 h and RT for 5 h, and then adding hydroquinone and CH_3ONa, 85 °C for 8 h.

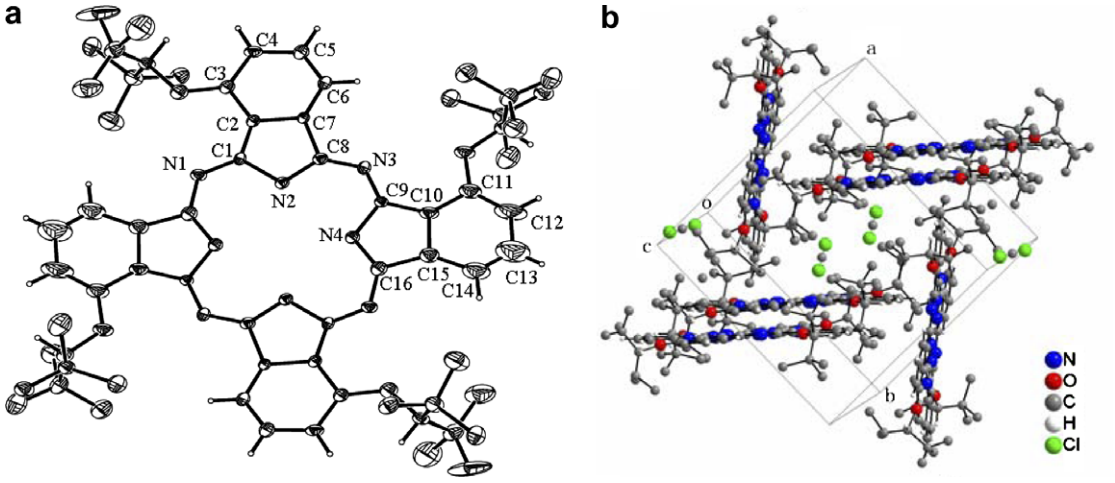


Fig. 1. The molecular structure of compound **2** (a) and its packing in cell (b).

neighbored. The packing of the compound **2** in crystal is shown Fig. 1b. It is found that the neighbored independent molecules are perpendicular each other to form a quadrate cavity with a dichloromethane molecule encapsulated.

Table 2 (the atom labeling as Fig. 2) shows some selected bond lengths and angles of title compounds and the corresponding unsubstituted derivative for comparison. Compared with the unsubstituted Pc [12], it can be found that the distances between the opposite two inner N atoms of substituted Pcs (N_a–N_a and N_p–N_p) are increased, that is, the introduction of the alkyloxy substituents at non-peripheral position expand the cave of the Pc's ring skeleton. This may be comprehended from that the O atoms of alkyloxy substituents enlarged the conjugating system of Pc with their non-bond electrons [13], at the same time, hauled the electrons of conjugated system. And the cave expansion is consistent with the IR spectra where the vibration frequencies of C=N bond of these substituted metal-free phthalocyanines (1585.6 cm^{-1} for **1**, 1586.9 cm^{-1} for **2**, 1597.46 cm^{-1} for **3**, and 1583.1 cm^{-1} for **4**) are decreased compared with that of unsubstituted metal-free phthalocyanines (1600 cm^{-1}) [14].

3.3. UV–vis spectroscopy studies of the metal-free phthalocyanines

It is known that the Q bands of metal-free Pcs always split into two peaks Q_x and Q_y, as listed in Table 3. It is found that the splitting of Q bands ($Q_y - Q_x$) show a wavelength-dependent characteristic (Fig. 3). Namely, the splitting decreases with the wavelength increasing of the Q band what can be expressed with $Q_y - Q_x = 0.2024Q_y - 2278.1$. According to this result, substituted H_2 Pc with the Q band near 800 nm will not show apparent splitting which is also agreed with the metal-free Octakis(*p*-methanoxyphoxy)phthalocyanine reported by Kobayashi [15].

Apparently, the decrease of the splitting of Q bands for metal-free Pcs is attributed to the different shift of Q_x and Q_y by the introduction of substituents, that is, the shift of Q_x is only 0.78 times of Q_y in $CHCl_3$ (the inset of Fig. 3). But in fact, the differentiation of the electron densities between the N_p and N_a atoms become indistinct as a result of the release electron effect of the alkyloxy substituents at non-peripheral position. The Q band splitting of the asymmetry compound **4** agrees with the law of symmetry Pcs, which indicates that the substitution asymmetry in outer benzene rings have not affected the electron distribution of the inner conjugating system. In addition, the analogous wavelength-dependent relations were also found for the metal-free phthalocyanines in different solvents. But compared with substituents, the effect of solvents for the compound **2** is larger with $Q_y - Q_x = 0.5773Q_y - 7629.6$, as shown in Table 4 and Fig. 4.

3.4. ^1H NMR studies of the metal-free phthalocyanines

For **1**, the protons of the Pc skeleton present multi-peaks indicating that there are not only one isomer [16]. On the contrary, the simple proton peaks for other compounds showed they are only one isomer consistent with the HPLC chromatogram. In addition, ^1H NMR of **2** was characterized in dilute benzene solution (1.8 mg/0.5 mL) to obtain better spectra than that in $CDCl_3$. It was supposed to be the decomposition or aggregation of phthalocyanines in $CDCl_3$ for the $CDCl_3$ is always not capable of breaking up the molecular structure completely in solution [17].

Fig. 5 shows the relationship between the position of Q_y band and pyrrole proton signals. It is found the chemical shifts of pyrrole protons are Q band wavelength-dependent too. According to Kobayashi et al. [4], the pyrrole protons appear with more negative chemical shifts due to the higher ring current of the macrocycle. That is, the H_2 Pcs having Q bands at longer wavelength show lower ring currents. Together with the data of crystal structures, IR and UV–vis spectra, we can find that the introduction of non-peripheral alkoxy substituents not only increase the conjugated system, but also expand the cave of the Pc skeletons. As a result, the ring currents of Pc skeletons were reduced instead.

Table 2
The contrastive selected bond lengths (Å) and bond angles (°) of the title compounds

Compounds	$C_\alpha-N_m$ [a]	$C_\alpha-N_p$ ($C_\alpha-N_a$)	$C_\alpha-C_\beta$	$C_\beta-C_\beta$ $C_\alpha-N_m-C_\alpha$	$C_\alpha-N_p-C_\alpha$ ($C_\alpha-N_a-C_\alpha$)	$N_m-C_\alpha-N_p$ ($N_m-C_\alpha-N_a$)	N_a-N_a (N_p-N_p)
H_2 Pc [12]	1.335	1.340	1.490	1.390	115, 119	109	131	3.826
2	1.309	1.461	1.430	1.440	126.42	110.33	122.89	3.950
	1.335	1.306	1.522	1.452	126.36	108.89	133.01	3.949
	1.301	1.469	1.360				120.89	
	1.258	1.369	1.492				129.76	
3	1.348	1.369	1.482	1.406	122.72	106.77	126.29	3.840
	1.349	1.373	1.467		121.92		127.40	4.037
4	1.327	1.363	1.449	1.402	122.83	110.84	129.06	3.845
	1.317	1.384	1.446	1.379	123.23	107.25	126.28	4.013
	1.323	1.368	1.456				128.53	
	1.319	1.369	1.453				128.00	

[a] The marked of phthalocyanine are shown in Fig. 2.

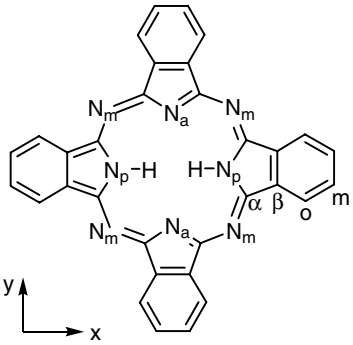


Fig. 2. The Molecular structure of metal-free phthalocyanine. N_p denotes a pyrrole nitrogen and N_a and N_m denote pyrrole aza and meso-bridging aza nitrogenism respectively. The carbon atoms are denoted following their position with respect to pyrrole nitrogen atoms (C_α and C_β) and to the pyrrole moiety (C_o and C_m).

Table 3
Electronic absorption data of the metal free phthalocyanines in $CHCl_3$

Compounds	**1**	**2**	**3**	**4**
Q_x (nm)/ (cm^{-1})	728.21/13732	731.95/13662	774.68/12909	714.87/13989
Q_y (nm)/ (cm^{-1})	695.37/14381	702.02/14245	751.26/13310	680.89/14687
$Q_y - Q_x$	649	583	420	698

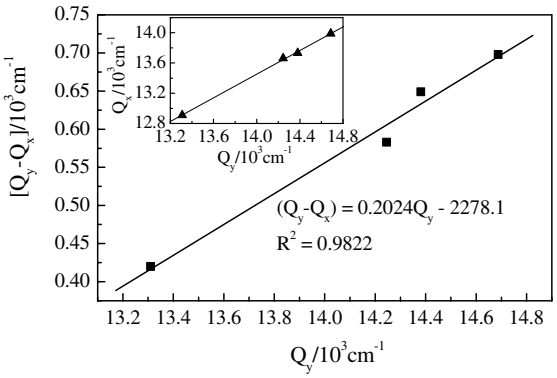


Fig. 3. Correlation between the splitting of Q band $Q_y - Q_x$ and the Q band Q_y in $CHCl_3$ for the different compounds, and the inset are the correlation between the Q_x and Q_y of the Q band ($Q_x = 0.7824Q_y + 2498.2$, $R^2 = 0.999$).

Table 4
The Electronic absorption data of the compound **2** in different solvents

Solvents	Q_x (nm)/(cm^{-1})	Q_y (nm)/(cm^{-1})	$Q_y - Q_x$/(cm^{-1})
Toluene	730.10/13697	697.37/14340	643
CH_2Cl_2	731.67/13667	702.13/14242	575
$CHCl_3$	732.64/13649	700.85/14268	619
THF	731.93/13663	699.25/14301	638
DMF	734.63/13612	706.77/14149	537
DMSO	737.86/13553	713.52/14015	462
Py	735.83/13590	708.71/14110	520

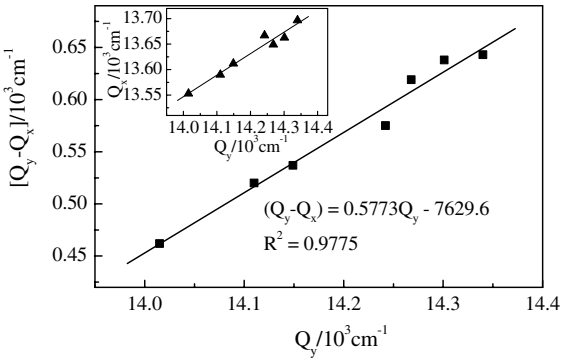


Fig. 4. Correlation between the splitting of Q band $Q_y - Q_x$ and the Q band Q_y in different solvents for compound **2**, and the inset are the correlation between the Q_x and Q_y of the Q band ($Q_x = 0.4227Q_y + 7629.6$, $R^2 = 0.9587$).

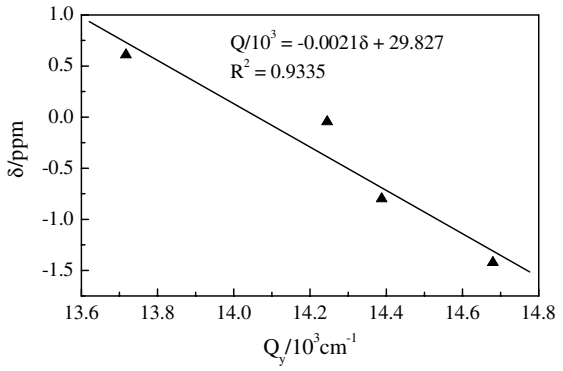
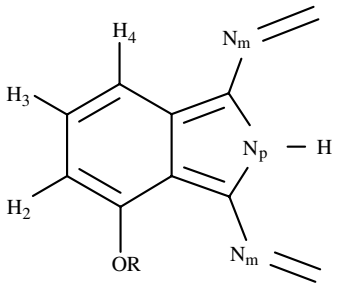


Fig. 5. Correlation between the inner H chemical shift of metal-free phthalocyanines and the Q band Q_y in $CDCl_3$.

Table 5
The ^{1}H NMR data of compound **2** in $CDCl_3$

Protons	δ (Unit: ppm)	Intensity	H number	Intensity/H	Relative intensity[a]
—CH—	2.592	0.29	4	0.0725	1.00
(—CH—O)	4.765	0.29	4	0.0725	1.00
H2—Pc	7.716	0.25	4	0.0625	0.86
H3—Pc	8.047	0.28	4	0.0700	0.97
H4—Pc	9.146	0.22	4	0.0550	0.76
H—N	−0.046	0.09	2	0.0450	0.62

[a] The intensity of —CH— is 1, and the H labeling is shown in Scheme 3.



Scheme 3. The H labeling for ^{1}H NMR spectra of the Pcs.

Furthermore, the average intensities of the inner protons are somewhat larger than the outer protons. As shown in Table 5, the order of the intensity of every proton for compound **2** is N—H < Pc-H4 < Pc-H2 < Pc-H3 < H-substituent. It perhaps can be attributed to the proton–proton nuclear Overhauser effect (NOE) [18]. The observed signals of the protons of outer substituents are increasing for their easy reversal. But contradictorily, the protons of Pc skeleton present a negative NOE because of the large molecular weight and rigidity.

4. Conclusion

The introduction of alkyloxy substituents to the non-peripheral position of phthalocyanine led to a larger center cave, weaker inner bonds, indistinct Q band split, and lower ring current. We believed that this came from the electron donor and electronegativity properties of the alkyloxy groups.

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