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A novel metal-free and metallophthalocyanines containing four 19-membered dithiadiazadioxa macrocycles by microwave irradiation: Synthesis and characterization

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Abstract

The novel tetrasubstituted metal-free phthalocyanine (5) and metallophtalocyanines (6, 7 and 8) bearing four 19-membered dithiadiazadioxa macrocyclic moieties on peripheral positions have been synthesized by cyclotetramerization reaction of phthalonitrile derivative (4) in a multi-step reaction sequence. The new compounds were characterized by a combination of IR, ¹H NMR, ¹³C NMR, UV–Vis, elemental analysis and MS spectral data.

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Keywords: Phthalocyanine; Dithiadiazamacrocycle; Phthalonitrile; Macrocyclization; Tosylation

1. Introduction

Phthalocyanines and metallophthalocyanines have been investigated for many years owing to their range applications in many fields, including use and investigation in chemical sensors, liquid crystals, Langmuir–Blodgett films, non-linear optics, optical data storage and as carrier generation materials in near-IR devices. Substituted derivatives can be used for photodynamic cancer therapy and other processes driven by visible light. A disadvantage of phthalocyanines and metal phthalocyanines is their low solubility in organic solvents or water [1,2].

Phthalocyanines have become noted recently for their special optical and electrical properties such as electrical conductivity, photovoltaic effects, and electrochromism [3,4]. Also, their use as efficient is becoming important in photodynamic therapy, as well [5].

Phthalocyanines have a two dimensional $18-\pi$ electron conjugated system, in which more than 70 different metal

and also non-metal ions can be incorporated. A number of modifications can be made in the macrocycle either by introduction of different central ions or by substitution of functional groups at the peripheral sites of the ring [6]. Microwaves have been previously used for the synthesis of phthalocyanines and include a wider range of references on the topic [7–15].

We have previously synthesized phyhalocyanines containing 14-membered tetraaza macrocyclic [13], four pyridyl groups [14] polymeric group [15] by microwave irradiation. In this paper, we have rapidly prepared metal-free and metallophthalocyanines by microwave irradiation and we have described characterization of the new metal-free and metallophthalocyanines, bearing dithiadiazadioxa macrocyclic moieties (Fig. 1).

2. Results and discussion

The preparation of the target metal-free 5 and metallophthalocyanines (6, 7 and 8) is shown in Scheme 1. The structures of novel compounds were characterized by the combination of ¹H NMR, ¹³C NMR, IR, UV–Vis,

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Fig. 1. UV–Vis spectra of compounds $5~(\cdots),~6~(-\!-\!\!-\!)$ and $7~(-\!-\!\!-\!)$ in chloroform.

elemental analysis and MS spectral data. N-[2-({3-[(2-aminophenyl)thio]propyl}thio)phenyl]-4,4'-di(toluene-*p*-sulfo-nylamino) **1** [16], 1,2-dibromo-4,5-bis(2-iyodoethoxy) benzene **2** [17] were prepared according to the literatures.

Compound 1 [16] was reacted with 1,2-dibromo-4,5bis(2-iyodoethoxy)benzene 2 [17] in dry CH₃CN containing finely ground anhydrous Cs₂CO₃ as a template agent in a Schlenk system under an argon atmosphere to give the new dithiadiazadioxa macrocycle 3 in 58% yield. Tosylation of aza groups, which are used as protective groups of aza functions in the cyclotetramerization, is effective in the cyclization. Analytical and spectroscopic data of 3 clearly confirm the success of the cyclization reaction. In the ¹H NMR spectrum of **3** the chemical shifts of NH protons in precursor compound 1 disappear after the macrocyclization reaction. In the ¹H NMR. spectrum of **3**, the chemical shifts due to Ar-H, CH2-O, CH2-N protons were observed at $\delta = 7.17$, 3.66, 3.50 ppm, respectively. ¹³C NMR spectral data were also in good agreement with the formation of 3. The chemical shifts for aromatic carbon atoms, CH₂–O and CH₂–N were observed at $\delta = 147.44$, 119.36, 114.67, 64.66 and 49.85 ppm, respectively. The difference between in infrared spectra of the precursor compounds and the macrocycle is clear from the presence of characteristic vibrations belonging to the starting materials, such as Ar–H (3054 cm^{-1}), SO₂ ($1348-1161 \text{ cm}^{-1}$), Ar–O–C (1089 cm⁻¹) and the absence of N–H stretching vibrations. The MS mass spectrum of 3, which shows a peak at m/z = 918 [M]⁺ support the proposed formula for this compound.

4,5'-Dicyano substituted macrocycle was synthesized by treating the dibromo compound **3** with three equivalents of CuCN according to Rosenmund von Braun reaction [18] in a moderately high-boiling solvent such as DMF at 145 °C under argon atmosphere. The ¹H NMR spectrum of **4** closely resembles that of the precursor compound **3** as expected. The ¹³C NMR spectrum of **4** shows the presence of nitrile carbon atoms at $\delta = 113.61$ ppm which indicates the completion of conversion of **3–4**. The MS mass spectrum of compound **4**, which shows a peak at m/z = 812 [M+1]⁺ support the proposed formula for this compound.

Metal-free phthalocyanine **5** was synthesized by microwave irradiation [13–15] of the corresponding dicyano compound **4** in dry quinoline for 15 min. The IR spectrum of metal-free phthalocyanine **5** shows the 3288 (NH), 3054 (Ar–H) vibrations. In the ¹H NMR spectrum of **5**, the typical shielding of inner core protons could not be observed due to the probable strong aggregation of the molecules [19]. The signals related to aromatic protons and aliphatic protons in the macrocyclic moieties and phthalocyanine skeleton gave significant absorbance characteristic of the proposed structure. The disappearance of the C=N stretching vibration on the IR spectra of **4** suggested the formation of compound **5**. The MS mass spectra of compound **5**, which shows a peak at m/z = 3246 [M]⁺ support the proposed formula for this compound.

The metallophthalocyanines 6, 7 and 8 were synthesized in moderate yield 40%, 44% and 39%, respectively. The metallophthalocyanines (6, 7 and 8) were obtained from dicyano derivative 4 and corresponding anhydrous metal salts NiCl₂, CoCl₂ and CuCl₂ respectively, by microwave irradiation in dry quinoline for 15 min. The IR spectra of metallophthalocyanines (6, 7 and 8) the disappearance of strong $C \equiv N$ stretching vibration of **4** is an evidence for the formation of metallophthalocyanines 6, 7 and 8. The rest of the IR spectra of metallophthalocyanines are very similar to those of the metal-free phthalocyanine 5. In the ¹H NMR spectra of these compounds are almost identical to those of metal-free phthalocyanine 5. Also, it should be mentioned that the other differences in the ¹H NMR spectra of metal-free phthalocyanine and metallophthalocyanines were the broad signals encountered in the case of compounds 6, 7 and 8, owing to the aggregation of planar phthalocyanine molecules at the considerably high concentration used for NMR measurements. They are in agreement with the structural information. In the mass spectrum of compounds 6, 7 and 8, the presence of molecular ion peaks at m/z = 3302 [M]⁺, 3303 [M]⁺ and 3307 $[M]^+$, respectively, confirmed the proposed structures.

In general, phthalocyanines show typical electronic spectra with two strong absorption regions, one in the UV region at about 300–500 nm related to the B band and the other in the visible region at 600–700 nm related to the Q band [20]. The split Q bands in 5, which are characteristic for metal-free phthalocyanines were observed at $\lambda_{\text{max}} = 707$ and 689 nm. These Q band absorptions show the monomeric species with D_{2h} symmetry and due to the phthalocyanine ring related to the fully conjugated 18- π electron system [21–23]. The presence of strong absorption bands in 5 in the near UV region at $\lambda_{\text{max}} = 334$ and 281 nm also shows Soret region B bands which have been ascribed to the deeper π - π^* levels of LUMO transitions.

The UV–Vis absorption spectra of metallophthalocyanines (6, 7 and 8) in chloroform show intense Q absorption at $\lambda_{max} = 680$, 686 and 691 nm, with weaker absorptions at 611, 617 and 622 nm, respectively. The single Q bands in metallo derivatives 6, 7 and 8 are characteristic. This result is typical for metal complexes of substituted and unsubsti-



Scheme 1. The synthesis of the metal-free phthalocyanine and metallophthalocyanines (Ts = p-toluenesulphonyl).

tuted metallophthalocyanines with D_{4h} symmetry [24] B band absorptions of compound **6**, **7** and **8** were observed at $\lambda_{\text{max}} = (342 \text{ and } 270)$, (351 and 276), (354 and 282) nm as expected, respectively.

3. Experimental

N-[2-({3-[(2-aminophenyl)thio]propyl}thio)phenyl]-4,4'di(toluene-*p*-sulfonylamino) (1) [16] and 1,2-dibromo-4,5bis(2-iyodoethoxy)benzene (2) [17] were prepared according to the literatures. All reagents and solvents were of reagent grade quality and were obtained from commercial suppliers. All solvents were dried and purified as described by Perrin and Armarego [25]. The IR spectra were recorded on a Perkin–Elmer 1600 FT-IR Spectrophotometer, using KBr pellets or NaCl disc. ¹H and ¹³C NMR spectra were recorded on a Varian Mercury 200 MHz spectrometer in CDCl₃, DMSO, and chemical shifts were reported (δ) relative to Me₄Si as internal standard. Mass spectra were measured on a Micromass Quatro LC/ULTIMA LC-MS/MS spectrometer. Elemental analyses were determined by a LECO Elemental Analyser (CHNS O932) and Unicam 929 AA spectrophotometer, respectively. Melting points were measured on an electrothermal apparatus and are uncorrected. Optical spectra in the UV–Vis region were recorded with a Unicam UV2-100 spectrophotometer, using 1 cm pathlength cuvettes at room temperature.

3.1. 10,11-Dibromo-5,16-bis[(4-methylphenyl)sulfonyl]-6,7,15,16,23,24-hexahydro-5H,14H,-22H-tribenzo[b,h,o]-[1,4,10,14,7, 17]dioxadithiadiazacyclononadecine (**3**)

N-[2-({3-[(2-aminophenyl)thio]propyl}thio)phenyl]-4,4'di(toluene-*p*-sulfonylamino) (1) (3 g, 5.01 mmol) and in dry acetonitrile (120 ml) containing finely ground anhydrous Cs₂CO₃ (4.07 g, 12.52 mmol) and purged under argon in a Schlenk system connected to a vacuum-line was heated and stirred under argon until 1 was dissolved. This solution was stirred at 110 °C and a solution of 1,2-dibromo-4,5bis(2-iyodoethoxy)benzene 2 (2.88 g, 5.01 mmol) in dry acetonitrile (40 ml) was added dropwise over a period of 4 h. The reaction was monitored by TLC using chloroform/petroleum ether/methanol (7:2:1) and was completed in 120 h at the same temperature. At the end of this period, the reaction mixture was filtered and washed with dry acetonitrile and then the combined solution was evaporated to dryness under reduced pressure. The oily product was mixed with water (50 ml) and then extracted with chloroform $(3 \times 70 \text{ ml})$. The combined extract was washed with water, dried over MgSO₄ and then filtered and evaporated to dryness to give crude product. The crude product was chromatographed on silica gel with chloroform as eluents. Yield: 2.66 g (58%), m.p.: 85-87 °C. Anal. Calc. for C₃₉H₃₈N₂O₆S₄Br₂(918.80): C, 50.98; H, 4.17; N, 3.04. Found: C, 50.40; H, 4.10; N, 3.31%. IR (KBr tablet) v_{max}/cm⁻¹: 3054 (Ar-H), 2924–2853 (Aliph. C-H), 1597, 1468, 1348–1161 (SO₂), 1089, 907, 814, 759, 673, 650, 575. ¹H NMR. (200 MHz, CDCl₃), (δ : ppm): 7.48 (m, 4H, tosyl Ar-H), 7.32 (m, 4H, tosyl Ar-H), 7.25 (t, 2H, Ar-H), 7.21 (d, 2H, Ar-H), 7.15 (t, 2H, Ar-H), 7.09 (d, 2H, Ar-H), 7.17 (s, 2H, Ar-H), 3.66 (m, 4H, CH2-O), 3.50 (t, 4H, CH₂-N), 2.87 (t, 4H, CH₂-S), 2.38 (s, 6H, CH₃), 1.53 (m, 2H, CH₂-CH₂-CH₂). ¹³C NMR. (200 MHz, CDCl₃), (δ: ppm): 147.44, 143.77, 138.14, 135.58, 130.00, 129.70, 129.28, 127.89, 124.63, 119.36, 114.67, 64.66, 49.85, 34.44, 29.69, 21.64. MS (FAB), (m/z): 918 [M]⁺.

3.2. 5,16-Bis[(4-methylphenyl)sulfonyl]-6,7,15,16,23,24hexahydro-5H,14H,22H- tribenzo[b,h,o] [1,4,10,14,7,17]dioxadithiadiazacyclononadecine-10,11-dicarbonitrile (**4**)

A mixture of 10,11-dibromo-5,16-bis[(4-methylphenyl)sulfonyl]-6,7,15,16,23,24-hexahydro-5*H*,14*H*,22*H*tribenzo[*b*,*h*,*o*][1,4,10,14,7,17]dioxadithiadiazacyclononadecine (**3**) (2.30 g, 2.50 mmol), CuCN (0.67 g, 7.5 mmol), and dry pyridine (0.12 ml) in dry DMF (25 ml) was refluxed under nitrogen for 48 h. The mixture was cooled to room temperature and then poured into aqueous ammonia. After stirring for 24 h, the mixture was extracted with chloroform. The combined organic layers were washed with

water, dried over anhydrous magnesium sulfate and then evaporated to dryness. Methanol 10 ml was added to this section and then stirred at room temperature for 6 h. At the end of this period, the precipitation occured. The resulting brown precipitate was filtered off, washed with cold methanol and diethyl ether and dried in vacuo. Yield: 1.38 g (68%), m.p.: 175–177 °C. Anal. Calc. for C₄₁H₃₈N₄O₆S₄(811.02): C, 60.71; H, 4.72; N, 6.90. Found: C, 60.78; H, 4.89; N, 6.71%. IR (KBr tablet) v_{max}/cm^{-1} : 3059 (Ar-H), 2922-2851 (Aliph. C-H), 2229 (C=N), 1594, 1515, 1468, 1347-1161 (SO₂), 1088, 914, 815, 756, 723. ¹H NMR. (200 MHz, CDCl₃), (δ : ppm): 7.52 (s, 2H, Ar-H), 7.48 (m, 4H, tosyl Ar-H), 7.23 (m, 4H, tosyl Ar-H), 7.19 (t, 2H, Ar-H), 7.16 (d, 2H, Ar-H), 7.12 (t, 2H, Ar-H), 7.01 (d, 2H, Ar-H), 3.74 (m, 4H, CH₂-O), 3.66 (t, 4H, CH2-N), 2.81 (t, 4H, CH2-S), 2.36 (s, 6H, CH₃), 1.80 (m, 2H, CH₂-CH₂-CH₂). ¹³C NMR. (200 MHz, CDCl₃), (δ: ppm): 151.73, 144.28, 139.45, 134.27, 130.82, 129.84, 128.26, 125.71, 123.55, 117.73, 115.29, 113.61, 111.48, 68.05, 49.93, 32.11, 29.94, 21.89. MS (FAB), (m/z): 812 $[M+1]^+$.

3.3. Metal-free phthalocyanine (5)

А mixture of 5,16-bis[(4-methylphenyl)sulfonyl]-6,7,15,16,23,24-hexahydro-5H,14H,22H-tribenzo[b,h,o] [1,4, 10,14,7,17]dioxadithiadiazacyclononadecine-10,11-dicarbonitrile (4) (0.3 g, 0.37 mmol) and dry quinoline (2.5 ml) was irradiated in a microwave oven at 220 °C, 350 W for 15 min. After cooling to room temperature the reaction mixture refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (40 ml) and dried in vacuo. The solid product was chromatographed on silica gel with chloroform:methanol (9.5:0.5) as eluents. Yield: 151 mg (41%), m.p.: >300 °C. Anal. Calc. for $C_{164}H_{154}N_{16}O_{24}S_{16}$ (3246.08): C, 60.68; H, 4.78; N, 6.90. Found: C, 60.34; H, 4.59; N, 6.73%. IR (KBr tablet) v_{max}/cm^{-1} : 3288 (N–H), 3054 (Ar-H), 2923-2857 (Aliph. C-H), 1618, 1594, 1500, 1467, 1346–1161 (SO₂), 1088, 1050, 810, 724, 673, 575. UV–Vis (CHCl₃): λ_{max}/nm : $[(10^{-5} \varepsilon dm^3 mol^{-1} cm^{-1})]$: 281 (4.83), 334 (4.68), 689 (5.08), 707 (5.11). ¹H NMR. (200 MHz, DMSO- d_6), (δ : ppm) 8.42 (s, 8H, Ar–H), 7.68-7.32 (m, 32H, tosyl Ar-H), 7.57-6.95 (m, 16H, Ar-H), 7.51–7.23 (t, 16H, Ar-H), 3.90 (m, 16H, CH₂–O), 2.99 (t, 16H, CH₂-S), 2.95 (m, 16H, CH₂-N), 2.40 (s, 24H, CH₃), 2.19 (m, 8H, CH₂-CH₂): MS (FAB), (m/z): 3246 [M]⁺.

3.4. Nickel(II) phthalocyanine (6)

A mixture of 5,16-bis[(4-methylphenyl)sulfonyl]-6,7,15,16,23,24-hexahydro-5H,14H,22H-tribenzo[b,h,o][1,4, 10,14,7,17]dioxadithiadiazacyclononadecine-10,11-dicarbonitrile (4) (0.3 g, 0.37 mmol) anhydrous NiCl₂ (11.92 mg, 0.09 mmol) and dry quinoline (2.5 ml) was irradiated in a microwave oven at 220 °C, 350 W for 15 min. After cooling to room temperature the reaction mixture refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (30 ml) and dried in vacuo. The green solid product was chromatographed on silica gel with chloroform:methanol (9.5:0.5) as eluents. Yield: 150 mg (40%), m.p.: >300 °C. Anal. Calc. for C₁₆₄H₁₅₂N₁₆O₂₄S₁₆Ni (3302.75): C, 59.64; H, 4.63; N, 6.78; Ni, 1.77. Found: C, 59.07; H, 4.88; N, 6.82; Ni, 2.02%. IR (KBr tablet) $v_{\text{max}}/\text{cm}^{-1}$: 3054 (Ar-H), 2923-2851 (Aliph. C-H), 1632, 1595, 1500, 1468, 1346-1162 (SO₂), 1089, 1045, 815, 724, 675, 575. UV-Vis (CHCl₃): $\lambda_{\text{max}}/\text{nm}$: [(10⁻⁵ ε dm³ mol⁻¹ cm⁻¹)]: 270 (4.88), 342 (4.76), 611 (4.58), 680 (5.16). ¹H NMR. (200 MHz, DMSO- d_6), (δ : ppm) 8.58 (s, 8H, Ar-H), 7.74–7.44 (m, 32H, tosyl Ar-H), 7.65-7.03 (m, 16H, Ar-H), 7.59-7.32 (t, 16H, Ar-H), 3.94 (m, 16H, CH₂-O), 3.06 (m, 16H, CH2-S), 2.98 (t, 16H, CH2-N), 2.49 (s, 24H, CH3), 2.23 (m, 8H, CH₂–CH₂). MS (FAB), (m/z): 3302 [M]⁺.

3.5. Cobalt(II) phthalocyanine (7)

A mixture of 5,16-bis[(4-methylphenyl)sulfonyl]-6,7, 15,16,23,24-hexahydro-5H,14H,22H-tribenzo[b,h,o][1,4,10, 14,7,17]dioxadithiadiazacyclononadecine-10,11-dicarbonitrile (4) (0.3 g, 0.37 mmol) anhydrous cobalt(II) chloride (11.94 mg, 0.09 mmol) and dry quinoline (2.5 ml) was irradiated in a microwave oven at 220 °C, 350 W for 15 min. After cooling to room temperature the reaction mixture refluxed with ethanol to precipitate the product which was filtered off. The green product was washed with hot ethanol (35 ml) and dried in vacuo. The solid product was chromatographed on silica gel with chloroform:methanol (9.5:0.5) as eluents. Yield: 164 mg (44%), m.p.: >300 °C. Anal. Calc. for C₁₆₄H₁₅₂N₁₆O₂₄S₁₆Co (3303.00): C, 59.63; H, 4.63; N, 6.78; Co, 1.78. Found: C, 59.36; H, 4.41; N, 6.88; Co, 2.06%. IR (KBr tablet) v_{max}/cm^{-1} : 3035 (Ar-H), 2945-2851 (Aliph. C-H), 1619, 1595, 1501, 1469, 1370–1140 (SO₂), 1117, 938, 804, 736, 627. UV–Vis (CHCl₃): $\lambda_{\text{max}}/\text{nm}$: [(10⁻⁵ ε dm³ mol⁻¹ cm⁻¹)]: 276 (4.75), 351 (4.64), 617 (4.52), 686 (5.04). ¹H NMR. (200 MHz, DMSO-d₆), (δ: ppm) 8.55 (s, 8H, Ar-H), 7.70-7.41 (m, 32H, tosyl Ar-H), 7.61-7.00 (m, 16H, Ar-H), 7.54-7.27 (t, 16H, Ar–H), 3.92 (m, 16H, CH₂–O), 3.04 (m, 16H, CH₂-S), 2.99 (m, 16H, CH₂-N), 2.47 (s, 24H, CH₃), 2.21 (m, 8H, CH₂–CH₂). MS (FAB), (m/z): 3303 [M]⁺.

3.6. Copper(II) phthalocyanine (8)

A mixture of 5,16-bis[(4-methylphenyl)sulfonyl]-6,7, 15,16,23,24-hexahydro-5H,14H,22H-tribenzo[b,h,o][1,4,10, 14,7,17]dioxadithiadiazacyclononadecine-10,11-dicarbonitrile (4) (0.3 g, 0.37 mmol) anhydrous copper(II) chloride (12.10 mg, 0.09 mmol) and dry quinoline (2.5 ml) was irradiated in a microwave oven at 220 °C, 350 W for 15 min. After cooling to room temperature the reaction mixture refluxed with ethanol to precipitate the product which was filtered off. The light green product was washed with hot ethanol (45 ml) and dried in vacuo. The green solid product was chromatographed on silica gel with chloroform:methanol (9.5:0.5) as eluents. Yield: 145 mg (39%), m.p.: >300 °C. Anal. Calc. for $C_{164}H_{152}N_{16}O_{24}S_{16}Cu$ (3307.61): C, 59.55; H, 4.63; N, 6.77; Cu, 1.92. Found: C, 59.48; H, 4.45; N, 6.89; Cu, 2.10%. IR (KBr tablet) $v_{max}/$ cm⁻¹: 3054 (Ar–H), 2923–2854 (Aliph. C–H), 1629, 1593, 1497, 1465, 1341–1161 (SO₂), 1083, 1048, 806, 721, 573. UV–Vis (CHCl₃): $\lambda_{max}/$ nm: [(10⁻⁵ ε dm³ mol⁻¹ cm⁻¹)]: 282 (4.71), 354 (4.60), 622 (4.40), 691 (4.96). MS (FAB), (m/z): 3307 [M]⁺.

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