Synthesis and Photophysical Properties of Phthalocyanines with 4-(1-Methyl-1-phenylethyl)phenoxy Groups

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This work is devoted to the study of synthesis and investigation of spectroscopic-luminescent and photochemical properties of metal complexes of octa-substituted phthalocyanines containing from four to eight fragments of cumylphenol and 1-benzotriazolyl- or nitro-groups. The objects of study were synthesized by “nitrile” method by heating the corresponding 4,5-substituted phthalonitriles with zinc and magnesium acetate, aluminum chloride.

Keywords: Synthesis, phthalodinitrile, phthalocyanine, phenoxy groups, fluorescence, singlet oxygen.

Синтез и фотофизические свойства фталоцианинов с 4-(1-метил-1-фенилэтил)фенокси группами

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Работа посвящена изучению синтетических подходов и исследованию спектрально-люминесцентных и фотохимических свойств металлокомплексов октазамещенных фталоцианинов, содержащих от 4 до 8 4-(1-метил-1-фенилэтил)фенокси-фрагментов, а также 1-бензотриазол- или нитрогруппы. Объекты исследования синтезировали “нитрильным” методом нагревания соответствующих 4,5-замещенных фталонитрилов с хлоридом алюминия, ацетатом цинка и магния.

Ключевые слова: Синтез, фталоцианин, фталоцианин, феноксигруппы, флуоресценция, синглетный кислород.
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Introduction

Phthalocyanines containing on the periphery oxarylic fragments exhibit wide range of properties useful in an applied sense. Thus, the presence of four 4-(1-methyl-1-phenylethyl)phenoxy- groups on the periphery of phthalocyanine molecule gives to such compounds photosensitivity, liquid-crystal, luminescent and sensor properties. Copper tetra-4-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanates were studied as a component of complex heterogeneous solar fuel. Corresponding phenoxyphthalocyaninates were studied as a component of selective heterogeneous solar fuel.

The study of usage of porphyrins and their synthetic analogues called phthalocyanines is also of interest. Macroheterocyclic compounds using as sensitizers have analogues called phthalocyanines is also of interest. Thus, the presence of four 4-(1-methyl-1-phenylethyl)phenoxy-groups in the molecule structure of both hydrophobic and hydrophilic fragments, absorption maximum at 630–730 nm, intensive fluorescence and so on. By means of this, the development and the investigation of novel phthalocyanine synthetic methods seems to be important work.

Present paper is devoted to synthesis and study of spectroscopic-luminescence and photochemical properties of octasubstituted phthalocyanine metal complexes containing from four to eight 4-(2-phenylisopropyl)phenol fragments, as well as 1-benzenetriazolyl- or nitro groups.

Experimental

Electronic absorption spectra (UV-Vis) were registered on Shimadzu UV-1800 spectrophotometer for solutions of tetraazaphosphorins and their complexes in the concentration range equal to 10⁻⁶–10⁻⁵ mol/L at wavelengths interval from 300 to 900 nm using quartz cells. 1H NMR spectra were recorded on Bruker (200 and 600 MHz) spectrometer in deuterated chloroform (CDCl₃) and deuterated dimethylsulfoxide (DMSO-d6). FT-IR spectra were obtained for the samples pressed with KBr on Avatar 360 FTIR spectrometer. MALDI-TOF mass-spectra were recorded on Shimadzu Biotech Axima confidence spectrometer using 3,5-dihydroxybenzoic acid as matrix and in the absence of it. Fluorescence spectra were registered on RF-6000 SHIMADZU spectrofluorometer using quartz cells (10 mm) in methanol and chloroform. Fluorescence quantum yields (Φ) determination was carried out by comparison method using solution of unsubstituted zinc phthalocyaninate (ZnPc) in 1-propanol as fluorescent standard (Φ(ZnPc) = 0.45 at λₐ = 600 nm) and calculated by equation:

\[
\Phi_F = \frac{G \cdot \eta_{ref} \cdot A' \cdot (\lambda_a) \cdot \Phi_{ref}}{G' \cdot n^2 \cdot (\lambda_c) \cdot A \cdot (\lambda_a) \cdot \Phi_{ref}},
\]

where \(G\) – integral radiation region, \(n\) – solvent refractive index, \(A\) – absorption (0.02) at the excitation wavelength, \(\Phi_F\) – fluorescence quantum yield.

The quantum yields of singlet oxygen generation were determined by comparison method [10] using equation:

\[
\Phi_S = \frac{k_{(\Phi_S)}}{k_{(\Phi_F)}} \cdot \frac{S_{(\Phi_S)}}{S_{(\Phi_F)}},
\]

where \(k_{(\Phi_S)}\) – rate constants of 1,3-diphenylisobenzofuran decomposition reaction in the presence of the investigated Pc and standard (ZnPc, \(\Phi_{(ZnPc)}=67\%\) in DMSO[11]); \(S\) is a total amount of light absorbed by the compound. Chemical reaction rate constants \(k_{(\Phi_S)}\) were found from the kinetic equation of the first order, reduced to a linear form: \(lnC = lnC_0 - kt\), where value of \(k\) was calculated as the tangent of the slope of the linear function to the time axis –tgk = k. The concentration of decolorizing diphenylisobenzofuran was found from the Bouger – Lambert – Beer law: \(A(\lambda) = \varepsilon(\lambda) \cdot C\), where \(A(\lambda)\) – the optical density; \(\varepsilon(\lambda)\) – the molar absorption coefficient; \(C\) – the layer thickness of the test solution. \(C\) – the concentration of the dissolved compound.

The solutions were irradiated with the ALKHT-ELOMED laser apparatus at a wavelength of 670 nm, with continuous operation and an optical radiation power of 1.2 W. The experiment was carried out under standard conditions with bubbling air into the photosensitizer solution.

4-[4-(1-Methyl-1-phenylethyl)phenoxy]-5-nitrophthalonitrile (4). A mixture of 0.25 g (1 mmol) of 4-bromo-5-nitrophthalonitrile (I) and 0.21 g (1 mmol) of 4-(2-phenylisopropyl)phenol was dissolved in 30 mL of DMF, and a solution of 0.14 g (1 mmol) of potash in 2 mL of water was added. The resulting mixture was stirred at 25 °C for 0.5 hours, then poured into water. The precipitate formed was collected on a filter and washed with 2-propanol, then with water and dried in air at 80–90 °C. Yield: 0.32 g (91 %). Found, %: C 71.95, H 5.00, N 10.74. C₁₇₇₆₅N₅O₆. Calculated: C 72.05, H 4.47, N 10.86. m/z (TLC,%): 383.11 (100 %). IR (KBr) νcm⁻¹: 2922, 2850 (CH₃), 2226 (CN), 1556 (asym. NO₂), 1334 (sym. NO₂), 1210 (Ar-O-Ar). 1H NMR (CDCl₃) δ ppm: 8.33 s (H₁, 1H), 7.87 d (H₂, 1H), 7.05 m (H₃, 4H), 7.12 m (H₄, 5H), 1.76 s (CH₃, 6).

4-[1-Benzotriazolyl]-5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile (5). Method 1: A mixture of 0.21 g (1 mmol) of 4-(2-phenylisopropyl)phenol and 0.29 g (1 mol) of 4-(1-benzotriazolyl)-5-nitrophthalonitrile (3) was dissolved in 50 mL of DMF, then 0.138 g (1 mmol) of potash, dissolved in 2 mL of water, was added to the solution. The reaction mixture was kept at room temperature for 1 hour. The precipitate was filtered off, washed with 2-propanol, then with water and dried at 80–90 °C. Yield: 0.39 g (87 %), m.p. 209–212 °C. Found, %: C 76.49, H 4.64, N 15.14. Calculated, %: C 76.42, H 4.65, N 15.37. 1H NMR (DMSO-d₆) δ ppm: 8.70 s (1H, H₁), 8.16 d (1H, H₃, J = 8 Hz), 7.85 s (1H, H₂), 7.8 d (1H, H₄, J = 7.9 Hz), 7.65 t (1H, H₅), 7.15 t (1H, H₆), 7.27 t (2H, H₇, H₈), 7.20 m (4H, H₉, H₁₀, H₁₁, H₁₂), 7.05 d (2H, J = 8.1 Hz, H₁₃, H₁₄), 1.60 s (6H, CH₃).

Compound 6 was synthesized using 4.24 g (0.02 mol) of 4-(1-methyl-1-phenylethyl)phenol. The reaction mass was stirred at 85–90°C for 8 hours. After heating, the reaction mixture was poured into water. The resulting viscous red-brown liquid, which contained the desired product, was separated on a separatory funnel and placed in a 100 mL conical flask, then 10 mL of a mixture of isopropyl alcohol and water (1 : 1 v/v) were added, covered with a glass funnel and left for 3 days. Initially, the unpainted water-alcohol layer became a saturated red-brown color, and the target phthalonitrile precipitated in the form of light beige lumps to the bottom of the flask. The resulting precipitate was filtered off, washed with 2-propanol to colorless filtrates, then with water until the pronounced odor of 2-propanol disappeared and dried in air at 80°C. Yield: 2.40 g (44 %), m.p. 123–125°C, IR spectrum (KBr) ν cm⁻¹: 2967, 2929, 2869 (CH₃), 2233 (CN), 1385 (C=O), 1753 (C=O), 1673 (C=O).
An integral part of this work is the preparation of synthetic precursors of the target octasubstituted phthalocyanines – the corresponding 4-(1-methyl-1-phenylethyl)phthalonitriles. The target products were obtained on the basis of 4-bromo-5-nitrophthalonitrile (1), since this compound is a highly active substrate for the production of both uniformly substituted and bifunctionally substituted phthalocyanines.[12–14] Compound 4 was synthesized by the interaction of 1 with 4-(2-phenylisopropyl) phenol in aqueous DMF containing 10 % water, according to the procedure.[14]

Since we have previously shown[15] that 4-(aryloxy)-5-nitrophthalonitriles may act as the starting materials for the synthesis of the corresponding benzotriazolyl substituted...
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compounds with aryloxy groups, an attempt was made to obtain benzotriazolyl substituted phthalonitrile 5 based on 4 (Scheme 1). It was found the usage of both above and the previously proposed methodologies\(^\text{[12]}\) allowed to obtain phthalonitrile 5 with 73 % yield.

We attempted to synthesize di-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile (6) based on nitro-substituted phthalonitrile 4, but the target compound was not obtained. This can be explained by the significant deactivation of the nitro group of compound 4 in nucleophilic substitution reactions during interaction with a 4-(2-phenylisopropyl)phenol bulk molecule.

Compound 6 was synthesized with 44 % yield in aqueous DMF in the presence of potash as a deprotonating agent\(^\text{[16]}\) at 85–90 °C. An attempt to increase the yield of the target product varying the temperature of the synthesis was also unsuccessful. Thus, the temperature was increased to 120 °C, the reaction mixture was completely ground, and a decrease to 60 °C led to the formation of only nitro-substituted phthalonitrile 4. An increase in the duration of synthesis to 12 and up to 24 hours caused the formation of a mixture of products 4 and 6, which could not be separated.

Finally, an attempt was made to synthesize compound 6 from 1, modifying Scheme 2 by excluding water from the components of the reaction mixture. During the synthesis, the reaction mixture turned to green. Upon completion of heating, when pouring into water, a precipitate of light green color was formed; it can be dissolved in chloroform. In the UV-Vis spectra in the long wavelength region, two intense absorption bands are observed (Figure 1), which is typical for metal-free phthalocyanines.\(^\text{[17]}\) Thus, at 85–90 °C in the absence of water, using potassium carbonate, the metal complex of the corresponding phthalocyanine with potassium is formed and the target phthalonitrile is contaminated with it. This fact can be explained by the low melting point of nitrile 6 (122–125 °C).

All the compounds obtained were identified using elemental analysis data, MALDI-TOF mass spectrometry, \(^1\)H NMR, IR spectroscopy. It was found, that the physicochemical properties of phthalonitrile samples synthesized by various methods are completely identical, which confirms the purity of the obtained compounds and the suitability of all the above approaches.

In the IR spectra of phthalonitriles (4–6) the stretching vibration of C≡N at 2230–2240 cm\(^{-1}\), the vibrations of methyl and methylene groups of 4-(1-methyl-1-phenylethyl)phenoxy groups at 2950–2830 cm\(^{-1}\), as well as symmetric (1340–1385 cm\(^{-1}\)) and asymmetric (1525–1565 cm\(^{-1}\)) vibrations of the NO\(_2\) group\(^\text{[18,19]}\) of compound 4 are observed.

While studying the compound 4 by IR spectroscopy upon heating, it was found to be decomposed at 365 °C.
Fixation in the IR spectra of gaseous degradation products of 4 revealed the presence of carbon monoxide. The next decomposition stage, proceeding at 475 °C, is accompanied by the release of methyl isocyanate, as well as phenol. That is, at this stage the participation in the process of thermal destruction of peripheral substituents: nitro- and 4-(1-methyl-1-phenylethyl)phenoxy groups is observed. The final decomposition of compound 4 is fixed at 630 °C. Carbon monoxide and cyanoacetic acid were recorded as the main gaseous products at this stage, which indicates the complete decomposition of the phthalonitrile molecule 4.

The metal complexes of 4-(1-methyl-1-phenylethyl)-phenoxy substituted phthalocyaninates of zinc, aluminum and magnesium (Scheme 3) were obtained by template co-condensation of the corresponding nitriles.

The process of thermooxidative degradation of the compounds synthesized in the presence of atmospheric oxygen is studied. It was found, that the temperature parameters of the decomposition of phthalonitriles 5 and 6, containing two bulky fragments, are very close (Table 1), while the introduction of the nitro group into 4 leads to a decrease in the temperature at which mass loss begins.

A lot of phthalocyanines exhibit fluorescence in the red spectral region, as well as the ability to generate singlet oxygen. Moreover, the position of the emission band maximum and the quantum yield of fluorescence and generation of singlet oxygen depend on many factors, such as the nature of phthalocyanine (the nature and position of substituents, the nature of the metal complexing agent), and the nature of the solvent. As an example, Figures 2 and 3 show the UV-Vis and fluorescence spectra of the Mg(II), Zn(II), and Al(III) metal complexes of tetra-4-[4-(1-methyl-1-phenylethyl)phenoxy]tetra-5-nitrophthalocyanine in chloroform. Table 2 presents the maxima of the emission of the main fluorescence band, Stokes shifts, quantum yields of fluorescence (ΦF) and generation of singlet oxygen (ΦΔ). Measurements were carried out using dichloromethane solutions to avoid possible protonation.

According to Table 2, the structure of metal complexes of octasubstituted phthalocyanines containing...
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Table 1. Parameters of the thermooxidative degradation of the compounds synthesized.

| No. | Compound | $T_{\text{melt}}, ^{\circ}\text{C}$ | $T_{\text{exo}}, ^{\circ}\text{C}$ | $\Delta T, ^{\circ}\text{C}$ |
|-----|----------|-----------------------------------|---------------------------|-----------------|
|     |          | I stage                           | II stage                  |                  |
| 4   | NC-NC-O-NO$_2$ | 124                              | 280                       | 544             | 475–600    |
| 5   | NC-NC-O-N-N | 128                              | 291, 333                  | 576             | 490–625    |
| 6   | NC-NC-O-NC-NO$_2$ | 148                              | 320                       | 568             | 500–650    |
| 7b  | Zn-NC-NC-O-NO$_2$ | 311                              | 479, 500                  | 390–540        |
| 8b  | Zn-NC-NC-O-N-N | –                                | 492                       | 549             | 520–580    |
| 8c  | ClAl-NC-NC-O-NC | 485                              | 547                       | 560–600        |

The temperature of the onset of mass loss of phthalonitriles 4–6 is naturally lower in comparison with the corresponding phthalocyanines 7, 8. It was noted, that the transition from octa-4,5-[4-(1-methyl-1-phenyl-ethyl)phenoxy]phthalocyaninate of zinc to its 4-nitro substituted analogue is accompanied by a significant decrease in the temperature of the onset of weight loss (Table 1). Replacing a zinc atom with an aluminum atom does not significantly affect the resistance of phthalocyanines to thermal oxidative degradation.

4-(1-methyl-1-phenylethyl)phenoxy groups at the periphery has a noticeable effect on their spectroscopic-luminescent and photochemical properties. Thus, the dependence of the quantum yield of fluorescence and the generation of singlet oxygen on the nature of metals was found; in the series Mg > Al-Cl > Zn, the $\Phi_f$ values decrease,$^{[20–22]}$ while the $\Phi_\Delta$ values increase on the contrary.$^{[28]}$ An analysis of the results showed the presence of a pronounced effect of the heavy atom on the photophysical properties of the studied phthalocyanines. Thus, the highest $\Phi_f$ values are observed for Mg(II) phthalocyanines and the highest $\Phi_\Delta$ values – for Zn(II) phthalocyanines (Figure 4), which indicates an increase in the fraction of nonradiative transition to the triplet state preceding the generation of singlet oxygen.$^{[29]}$

In addition, the quantum yields of fluorescence and singlet oxygen generation depend on the nature and bulk of peripheral substituents. According to the data in Table 2, the highest values of $\Phi_f$ and $\Phi_\Delta$ are shown in the metal complexes of octa-4,5-[4-(1-methyl-1-phenylethyl) phenoxy]phthalocyanine. The presence on the periphery,
Table 2. Data of phthalocyanines fluorescent spectra in dichloromethane solution.

| M       | Q, nm | Fluorescence, nm | Stokes shift, nm | $\Phi_f$ | $\Phi_\Delta$ |
|---------|-------|------------------|------------------|---------|--------------|
| Mg (7a) | 704   | 715              | 11               | 0.4     | 0.07         |
| Zn (7b) | 698   | 711              | 13               | 0.24    | 0.24         |
| Al-Cl (7c)| 705   | 715              | 10               | 0.33    | 0.22         |
| Mg (8a) | 690   | 694              | 4                | 0.49    | 0.12         |
| Zn (8b) | 680   | 695              | 15               | 0.29    | 0.42         |
| Al-Cl (8c)| 698   | 702              | 4                | 0.43    | 0.18         |
| Mg (9a) | 684   | 689              | 5                | 0.6     | 0.2          |
| Zn (9b) | 681   | 687              | 6                | 0.39    | 0.61         |
| Al-Cl (9c)| 696   | 699              | 3                | 0.49    | 0.46         |

along with the residues of 4-(1-methyl-1-phenylethyl)phenoxy fragments, of less bulky benzotriazole groups having significantly smaller sizes and volumes reduces the quantum yields of fluorescence and generation of singlet oxygen.

The lowest quantum yield values are demonstrated by phthalocyanines with nitro groups (Figure 4), which is due to the occurrence of strongly pronounced association processes, which noticeably affect the UV-Vis spectra of these compounds.

**Conclusion**

Thus, derivatives of 4-(1-methyl-1-phenylethyl)phenoxy substituted phthalonitrile containing nitro group, benzotriazolyl-fragment, and also an additional residue of 4-(2-phenylisopropyl)phenol (bifunctionally substituted phthalonitrile) were synthesized in this work. Using the template cyclotetramerization method by fusing the obtained phthalonitriles with Mg(II), Zn(II) acetates and Al(III) chloride, the corresponding metal phthalocyanines were synthesized. All the compounds obtained were identified using elemental analysis data, MALDI-TOF mass spectrometry, $^1$H NMR, IR spectroscopy.

The resistance of the synthesized phthalonitriles and phthalocyanines to thermal oxidative degradation was determined. It was established, that the compounds with nitro groups have lower mass loss temperatures; the replacement of the zinc atom in the coordination cavity of phthalocyanine by an aluminum atom does not significantly affect the thermal stability.
For all synthesized phthalocyanines, the photophysical properties were studied. The quantum yields of fluorescence and singlet oxygen generation were calculated, and the dependence of these indicators on the nature of metals and substituents was revealed.

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