Synthesis of Photoactive Polyfunctional Molecules Based on 1-Aryloxyanthraquinone Aminoderivatives

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Abstract—By alternating stages of condensation of 1-(4-*tert*-butylphenoxy)-2-amino-9,10-anthraquinone with 5-nitroisophthaloyl dichloride, cyanuric chloride, and 4-aminobenzocrown ethers with stages of catalytic reduction of nitro group photoactive polyfunctional compounds were prepared containing both several photoactive groups and complex-forming crown ether moieties.

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Recently a considerable interest grew in the chemistry of dendrimeric molecules resulting from a discovery of new specific features inherent to the dendrimeric architecture of highly efficient catalysts, pharmaceuticals, and polymeric materials [1-5]. Every year increases the number of various dendrimers synthesized, but only single instances are known of preparation of photoactive dendrimeric molecules. For instance, certain examples exist of incorporation of photosensitive azobenzene fragments into macrocyclic, catenane, and dendrite structures [6–9]. A unique feature of these compounds is first of all their capability to light-induced reversible structural transformations, molecular photo-switching arrangement, that can significantly extend their applicability, e.g., in materials for molecular electronic devices, for recording and storage of optical information.

The most promising procedure in designing photoactive dendrimers is application of photochromic substances as building blocks. The photochromic properties of 1-aryloxyand 1-acyloxy-9,10-anthraquinones are fairly well documented [10]. Different derivatives of 1-aryloxy- and 1-acyloxy-9,10-anthraquinones were shown to suffer under irradiation with light a reversible migration of aryl and acyl groups to the *peri*-oxygen giving 9-aryloxy- and 9-acyloxy-1,10-anthraquinones accompanied with significant spectral changes. We did not find any examples of application at these derivatives to building dendrimeric structures. From two possible procedures of building up dendrimeric molecules we chose the convergent one involving a primary synthesis of the dendrimer branches, dendrones, followed by their linking to the stem of the dendrimer. The target of this study was a development of synthetic methods for previously unknown photosensitive polyfunctional molecules including two or more fragments of the photochromic 1-arloxy-9,10-anthraquinone aminoderivative which could serve as dendrones in building up photoactive dendrimers.

In the framework of this approach condensations were carried out of 1-(4-tert-butylphenoxy)-2-amino-9,10-anthraquinone (**I**) with 5-nitroisophtaloyl dichloride (**II**), cyanuric chloride, and 4-aminobenzocrown ether. The latter was brought into the condensation to endow the molecule with complexing properties. It should be noted that the use of compound **I** with a *tert*-butyl substituent in the aryloxy group improves the solubility of compounds obtained in organic solvents.

In the first stage reaction of compounds **I** and **II** gave isophthalamide **III** in 85% yield. Then on catalytic reduction with hydrazine hydrate amino derivative **IV** was obtained in 60% yield.

In the third stage amine **IV** was acylated with acyl dichloride **II** into compound **V** in 72% yield. Thus isophthalamide **V** containing four photoactive centers was prepared in three stages. To get more complex branched molecules containing eight and more anthraquinone





moieties the reduction and acylation stages can be repeated (Scheme 1).

In the same manner photoactive polyfunctional molecules with a crown ether fragment were prepared. On mixed condensation of equimolar amounts of anthraquinone I and 4-aminobenzo-15-crown-5 (VI) with acyl dichloride II isophthalamide VII was isolated in 67% yield. Then by the pattern described above amino derivative **VIII** was prepared whose acylation with dichloride **II** led to the formation of polyfunctional molecule **IX** in 75% yield (Scheme 2).

By condensation of 1-aryloxy-2-amino-9,10-anthraquinone I with 2,4,6-trichloro-1,3,5-triazine (X) in phenol at 200°C was obtained in 76% yield photoactive triazine XI (Scheme 3).

Scheme 2.



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The study of photochemical activity of synthesized macromolecular compounds III, V, IX, and XI containing several fragments of 1-aryloxy-9,10-anthraquinone showed that they are endowed with photochromic qualities. Like in case of their monomeric analog, 1-(4tert-butylphenoxy)-2-(4-nitrobenzoylamino)-9,10anthraquinone (XII) [11], on irradiation of benzene solutions of compounds obtained ($\lambda_{max} < 400 \text{ nm}$) in their electron absorption spectrum gradually disappeared the longwave absorption maximum of the initial compound concurrently with the appearance of the absorption band of photoproduct. Therewith the solution color changed from light yellow to red-violet, and the maximum shift of the longwave absorption band into the visible region attained 180 nm. The photochemical isomerization of the newly synthesized derivatives is reversible. On irradiation with a longwave visible light ($\lambda_{max} > 500$ nm) the photoinduced form completely disappeared, and the spectrum of the initial compound is recovered. The direct and reverse photoisomerisation can be repeated many times; thus synthesized macromolecular compounds III, V, IX, and XI are typical photochromic substances. They are characterized by high photo-sensitivity, good color contrast, low rate of reverse dark reaction at room temperature ensuring long dark storage of the photoform. Interestingly, the quantum yield of the reverse photorearrangement in these compounds reduced about twice compared to compound XII.

In the study of the photochemical activity of the polyfunctional compounds obtained a reasonable problem arises whether all the photoactive fragments of 1-aryloxy-9,10-anthraquinone present take part in the photoinduced isomerization, and also whether the process occurs by stages. To clear the problem we carried out a photholysis of compound **III** in the presence of an equiv amount of *p*-toluidine. The reaction with amines was used as a certain test on *ana*-quinoid structure formation in the course of photomigration of aryl group. We showed

formerly [10–12] that on irradiation the 1-aryloxy-2(4)-R-9,10-anthraquinones easily react with water and amines yielding the corresponding hydroxy and amino derivatives. For instance, on photolysis in the presence of alkyl- and arylamines we isolated 1-hydroxy-2-(4)-R-9,10-anthraquinone 9-imines[12]. The ready formation of such compounds on photolysis originated from the high reactivity of the intermediate derivatives of 1,10-anthraquinone (*ana*-quinone) with respect to nucleophilic agents.

The photolysis of a benzene solution containing a mixture of isophthalamide **III** and *p*-toluidine resulted in formation of diimine **XIII** in virtually quantitative yield (Scheme 4). Chromatographic monitoring of the reaction progress showed that in the first minutes of the process formed two products, and then one of them disappeared. Presumably we succeeded in observing the formation of a product of monosubstitution of the aryloxy group in compound **III**, suggesting that the photoisomerisantion in the molecule with two photoactive centers was sequential.

For the sake of comparison we carried out the photolysis of 1,4- and 1,5-di(4-*tert*-butylphenoxy)-9,10anthraquinones (**XIV** and **XV**) in the presence of *p*-toluidine. These compounds also contain two aryl groups capable of migration under irradiation, but unlike compound **III** both groups are attached to the same anthraquinone framework.

Presumably in compounds **XIV** and **XV** both groups could take part in the sequential migration to give diimino derivatives, but the corresponding experiments yielded only the monosubstituted products: 1-hydroxy-4- and 5(4*tert*-butylphenoxy)-9,10-anthraquinone 9-imines (**XVI** and **XVII**).

The reason of this difference is probably the presence in compounds **XVI** and **XVII** of a chromophoric quinone imine system that governs the position of the longwave maximum in the electron absorption spectra (442 nm). Therefore at further irradiation no redistribution of electron



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Scheme 5.



XIV, XVI, R = OAr, R' = H; **XV, XVII**, R = H, R' = OAr; Ar = 4-t-BuC₆H₄.

density on quinone oxygens occurs that is required for the migration of the second aryl group (cf. [13]). Whereas the experiments with dendrones **III**, **V**, **IX**, and **XI** suggest that the irradiation with light causes presumably a sequential isomerization in all photoactive centers present in the molecule.

Thus we have developed a convenient preparation method for a wide range of photochromic dendrones containing alongside the photoactive groups crown ether fragments capable of complexing. Compounds obtained can be used for designing photoactive dendrimers.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Bruker Vector-22 from KBr pellets, electron absorption spectra were taken on a spectrophotometer Hewlett-Packard Agilent-8453 from ethanol solutions (10^{-4} mol/l) . ¹H NMR spectra were registered on a spectrometer Bruker WP-200SY, internal reference SiMe₄. Mass spectra (electron impact) were measured on Finnigan MAT-8200 instrument; the m/z values for molecular ion $[M]^+$ are presented. Mass spectrometric analysis of compounds with a molecular mass >500 was carried out using a liquid chromatograph with a mass-selective detector Agilent (1100 Series LC/MSD). The samples admission mode directly into the flow of liquid (FIA) was used. The samples ionization was performed by electrostatic atomization (API-ES). The scanning of positive or negative ions was performed in the range m/z 350–1500 at three values of voltage on the atomizer: 40, 70, and 100 V. Photolysis was carried out using DRSh-500 lamp with a filter UFS-1 (280-400 nm), and also with the total light of the mercury lamp. The desired spectral intervals were separated by standard filters: UFS-2, ZhS-3, SS-5, OS-11 (State Standard GOST 9411-66). The 313 nm line was isolated from the spectrum of the mercury lamp combining the filters UFS-2 and ZhS-3. TLC analysis was carried out on Silufol UV-254 plates, eluent a mixture toluene–ethanol, 9:1. For column chromatograpy silica gel was used (140–350 μ m). The solvents were dried before use.

Initial compounds **I**, **X**, **XI**, **XIV**, and **XV** were synthesized by a protocol from [14], compound **II**, by procedure [15], **VI**, by [16].

N,N'-Bis[1-(4-tert-butylphenoxy)-9,10-anthraquinone-2-yl]-5-nitroisophthalamide (III). A mixture of 0.37 g (1 mmol) of compound I and 0.15 g (0.6 mmol) of compound II was boiled in 20 ml of toluene for 30 min (TLC monitoring). The reaction mixture was evaporated to a volume of 3-4 ml, and ethyl ether was added. The precipitate was filtered off, washed with ethyl ether, and subjected to column chromatography on SiO₂ (eluent CHCl₃). Yield 0.75 g (85%), mp 326–328°C. IR spectrum, v, cm⁻¹: 3412 (N–H), 2963, 2928 (C–H), 1675 (C=O), 1590 (C=C). Electron absorption spectrum, λ_{max} , nm (log ε): 262 (4.71), 372 (3.98). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.23 s [18H, 2C(CH₃)₃], 6.91 d (4H, H^{2',6'}, J9 Hz), 7.35 d (4H, H^{3',5'}, J9 Hz), 7.75 m (4H, H^{6,7}), 8.21 m (4H, H^{5,8}), 8.40 d (2H, H³, J 8.5 Hz), 8.51, 8.66, 8.90 br.s (3H, C₆H₃), 9.05 d (2H, H⁴, J 8.5 Hz), 9.08 m (2H, NH). Found, %: C 73.36; H 4.66; N 4.50. [M]⁺ 917. C₅₆H₄₃N₃O₁₀. Calculated, %: C 73.28; H 4.69; N 4.58. M 917.98.

5-Amino-*N*,*N***'-bis**[**1-(4-***tert*-**butylphenoxy)-9,10anthraquinon-2-yl]isophthalamide (IV).** To a mixture of 0.92 g (1 mmol) of compound **III** and 0.02 g of moist Pd/C in 50 ml of ethanol was added dropwise 2 ml of hydrazine hydrate within 20 min. The mixture was boiled for 1 h and on cooling was subjected to column chromatography on Al_2O_3 (eluent CHCl₃), separating the main yellow zone. Eluate was evaporated, hexane was added, the separated precipitate was filtered off, and dried. Yield 0.53 g (60%), mp 271–274°C. IR spectrum, v, cm⁻¹: 3425, 3375 (NH₂), 2960, 2868 (C–H), 1674 (C=O), 1564 (C=O). Electron absorption spectrum, λ_{max} , nm (log ϵ): 268 (4.84), 388 (4.07). Found [*M*]⁺ 887. C₅₆H₄₅N₃O₈. Calculated *M* 887.99.

N,*N*'-Bis{*N*,*N*'-di[1-(4-*tert*-butylphenoxy)-9,10anthraquinon-2-yl]-5-isophthalamido}-5-nitroisophthalamide (V) was prepared similarly to compound III from 0.45 g (0.5 mmol) of compound IV and 0.08 g (0.3 mmol) of 5-nitroisophthaloyl dichloride. Yield 0.35 g (72%), mp 308–312°C. IR spectrum,v,cm⁻¹: 3420 (N– H), 2962, 2903 (C–H), 1676 (C=O), 1585 (C=O). Electron absorption spectrum, λ_{max} , nm (log ε): 266 (5.28), 378 (4.41). Found, %: C 73.33; H 4.51; N 4.56. C₁₂₀H₉₁N₇O₂₀. Calculated, %: C 73.88; H 4.67; N 5.03.

N-[1-(4-tert-butylphenoxy)-9,10-anthraquinon-2yl]-N'-(2,3,5,6,8,9,11,12-octahydrobenzo[b][1,4,7, 10,13]pentaoxacyclopentadecen-15-yl)-5-nitroisophthalamide (VII). To a solution of 0.5 g (2.1 mmol) of compound II in 20 ml of anhydrous benzene at 0°C was added dropwise within 30 min at stirring a solution of 0.74 g (2 mmol) of compound I in 15 ml of benzene containing several drops of pyridine (TLC monitoring). The reaction mixture was kept at room temperature in the dark for 5 h. Then 0.6 g (2.1 mmol) of 4-aminobenzo-15-crown-5 in 15 ml of THF containing several drops of pyridine was added, the mixture was heated at 40°C for 1 h. The reaction mixture was evaporated to a volume of 3–4 ml, and ethyl ether was added. The precipitate was filtered off, washed with ethyl ether, and subjected to column chromatography on SiO₂ (eluent CHCl₃). Yield 1.1 g (67%), mp 220–223°C. IR spectrum, v, cm⁻¹: 3421 (N-H), 3071, 2956, 2870 (C-H), 1673 (C=O), 1590 (C=C). Electron absorption spectrum, λ_{max} , nm (log ε): 256 (4.38), 370 (3.80). ¹H NMR spectrum (CDCl₃), δ, ppm: 1.23 s [9H, C(CH₃)₃], 3.79–4.08 m (16H, OCH₂CH₂), 6.83 d (2H, H^{2',6'}, J 9 Hz), 6.97 d (1H, H⁶ê, J 9 Hz), 7.21 d (2H, H^{3',5'}, J 9 Hz), 7.36 d.d (1H, H⁵ê, J₁) 9, J₂ 2 Hz), 7.47 d (1H, H³ê, J 2 Hz), 7.89 m (2H, H^{6,7}), 8.13 m (2H, H^{5,8}), 8.27 d (1H, H³, J 8.5 Hz), 8.41 d (2H, H⁴, J 8.5 Hz), 8.51, 8.66, 8.89 br.s (3H, C₆H₃), 10.50, 10.65 br.s (2H, 2NH). Found $[M]^+$ 829. $C_{46}H_{43}N_3O_{12}$. Calculated M 829.87.

5-Amino-N-[1-(4-*tert*-butylphenoxy)-9,10anthraquinon-2-yl]-N'-(2,3,5,6,8,9,11,12-octahydrobenzo[b][1,4,7,10,13]pentaoxacyclopentadecen-15yl)isophthalamide (VIII) was prepared in the same way as compound IV from 0.42 g (0.5 mmol) of compound VII, 0.01 g of moist Pd/C, and 1 ml of hydrazine hydrate in 30 ml of ethanol. Yield 0.30 g (75%), mp 198–202°C. IR spectrum, v, cm⁻¹: 3470, 3415 (NH₂), 2962, 2910 (C– H), 1675 (C=O), 1587 (C=C). Electron absorption spectrum, λ_{max} , nm (lgɛ): 256 (4.37), 380 (3.82). Found, %: C 69.53; H 5.51; N 5.35. C₄₆H₄₅N₃O₁₀. Calculated, %: C 69.09; H 5.63; N 5.26.

N,*N*'-Bis{*N*-[1-(4-*tert*-butylphenoxy)-9,10anthraquinone-2-yl]-*N*'-(2,3,5,6,8,9,11,12octahydrobenzo[*b*][1,4,7,10,13]pentaoxacyclopentadecen-15-yl)-5-isophthalamido}-5-nitroisophthalamide (IX) was prepared in the same way as compound III from 0.40 g (0.5 mmol) of compound VIII, and 0.08 g (0.3 mmol) of compound II. Yield 0.35 g (72%), mp 238–241°C. IR spectrum, v, cm⁻¹: 3420 (N–H), 2960, 2930 (C–H), 1675 (C=O), 1585 (C=C). Electron absorption spectrum, λ_{max} , nm (log ε): 266 (4.83), 376 (4.09). Found, %: C 67.30; H 5.11; N 5.65. C₁₀₀H₈₉N₇O₂₄. Calculated, %: C 67.76; H 5.03; N 5.53.

N,N',N''-Tri[1-(4-tert-butylphenoxy)-9,10-anthraquinon-2-yl]-2,4,6-amino-1,3,5-triazine (XI). A mixture of 1.1 g (3 mmol) of compound I, 0.2 g (1 mmol) of cyanuric chloride (X), and 5 g of phenol was heated at stirring to 200°C for 40 min (TLC monitoring). The reaction mixture was cooled, and 50 ml of 10% water solution of KOH was added. The precipitate was filtered off, washed with hot water, and dried. Then it was subjected to column chromatography on Al₂O₃ (eluent CHCl₃), separating the main yellow zone. Eluate was evaporated, hexane was added, the separated precipitate was filtered off, and dried. Yield 0.91 g (76%), mp 236-238°C. IR spectrum, v, cm⁻¹: 3406 (N–H), 2962 (C–H), 1676 (C=O), 1593 (C=C). Electron absorption spectrum λ_{max} , nm (log ϵ): 255 (5.06), 395 (4.27). Found, %: C 75.33; H 5.03; N 7.11. C₇₅H₆₀N₆O₉. Calculated, %: C 75.76; H 5.05; N 7.07.

Synthesis of compounds XIII, XVI, and XVII. In 0.5 liter of anhydrous benzene was dissolved 1 mmol of initial compound **III, XIV**, or **XV**, 2 mmol of *p*-toluidine was added, and the reaction mixture was subjected to the radiation of a mercury lamp SVD-120A for 3–4 h or to the sunlight for 10–12 h. Photholysis was carried out at 20–25°C till disappearance of the initial compound (TLC monitoring). The solvent was distilled off in a vacuum to dryness at 30°C, the residue was washed with hexane, filtered off, purified by column chromatography (eluent CHCl₃), and recrystallized from a mixture benzene–ethanol, 1:1.

N,*N*'-Bis[1-hydroxy-9-(4-methylphenyl)imine-9,10-anthraquinon-2-yl]-5-nitroisophthalamide (XIII). From 0.92 g of compound III and 0.22 g of *p*-toluidine we obtained 0.76 g (92%) of compound XIII, mp 215–218°C. IR spectrum, v, cm⁻¹: 3432 (N–H), 3067, 2962 (C–H), 1676, 1630 (C=O, C=N), 1593 (C=C). Electron absorption spectrum, λ_{max} , nm (log ε): 260 (4.78), 432 (4.04). ¹H NMR spectrum (CDCl₃), δ , ppm: 2.39 s (6H, CH₃), 7.01 d (4H, H^{2',6'}, *J* 9 Hz), 7.20 d (4H, H^{3',5'}, *J* 9 Hz), 7.31 d.d (2H, H⁸, *J*₁ 9, *J*₂ 2 Hz), 7.53 m (4H, H^{6,7}), 7.80 d (2H, H³, *J* 8.5 Hz), 8.31 d.d (2H, H⁵, *J*₁ 9, *J*₂ 2 Hz), 8.68 d (2H, H⁴, *J* 8.5 Hz), 8.83, 8.86, 8.90 br.s (3H, C₆H₃), 9.00 br.s (2H, NH), 10.65 br.s (2H, OH). Found, %: C 72.68; H 4.05; N 8.37. C₅₀H₃₃N₅O₈. Calculated, %: C 72.20; H 3.97; N 8.42.

1-Hydroxy-4-(4-*tert***-butylphenoxy)-9-(4-tolylimino)-9,10-anthraquinone (XVI).** From 0.5 g of compound **XIV** and 0.22 g of *p*-toluidine we obtained 0.41 g (90%) of compound **XVI**, mp 157–158°C. IR spectrum, v, cm⁻¹: 3431 (O–H), 3068, 2926, 2864 (C–H), 1671, 1636 (C=N, C=O), 1592 (C=C). Electron absorption spectrum, λ_{max} , nm (log ε): 268 (4.46), 442 (3.86). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.28 s [9H, C(CH₃)₃], 2.34 s (3H, CH₃), 6.83 d (2H, H^{2',6'}, *J* 9 Hz), 7.00 d (2H, H^{2"6"}, *J* 9 Hz), 7.15 d (2H, H^{3',5'}, *J* 9 Hz), 7.24 d (2H, H^{3",5"}, *J* 9 Hz), 7.34 m (2H, H^{6,7}), 7.44 d (1H, H³, *J* 8.5), 8.10 d.d (1H, H⁸, *J*₁ 9, *J*₂ 2 Hz), 8.35 d.d (1H, H⁵, *J*₁ 9, *J*₂ 2 Hz), 8.95 d (1H, H², *J* 8.5 Hz), 15.20 brs (1H, OH). Found, %: C 80.18; H 5.65; N 3.04.

1-Hydroxy-5-(4-*tert***-butylphenoxy)-9-(4-tolylimino)-9,10-anthraquinone (XVII).** From 0.5 g of compound **XV** and 0.22 g of *p*-toluidine we isolated 0.40 g (87%) of compound **XVII**, mp 180–182°C. IR spectrum, v, cm⁻¹: 3423 (O–H), 3073, 2926, 2872 (C– H), 1670, 1635 (C=N, C=O), 1591 (C=C). Electron absorption spectrum, λ_{max} , nm (log ε): 278 (4.48), 440 (3.87). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.28 s [9H, C(CH₃)₃], 2.34 s (3H, CH₃), 6.90 d (2H, H^{2',6'}, *J* 9 Hz), 7.05 d (2H, H^{2'',6''}, *J* 9 Hz), 7.12 d (2H, H^{3',5'}, *J* 9 Hz), 7.20 d (2H, H^{3'',5''}, *J* 9 Hz), 7.32 m (1H, H⁷), 7.38 m (1H, H⁶), 7.41 d (1H, H³, *J* 8.5 Hz), 8.10 m (1H, H⁸), 8.79 d (1H, H², *J* 8.5 Hz), 15.12 br.s (1H, OH). Found, %: C 80.51; H 5.76; N 3.09. C₃₁H₂₇NO₃. Calculated, %: C 80.69; H 5.86; N 3.04.

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