

Complex formation of 2,2-diphenyl-2*H*-benzo[*f*]chromene containing the aza-18-crown-6-ether fragment in the polymeric layer

V. B. Nazarov,^{a*} O. A. Fedorova,^b S. B. Brichkin,^a T. M. Nikolaeva,^a
S. P. Gromov,^b A. V. Chebun'kova,^b and M. V. Alfimov^b

^a*Institute of Problems of Chemical Physics, Russian Academy of Sciences,
142432 Chernogolovka, Moscow Region, Russian Federation.*

E-mail: nazarov@icp.rssi.ru

^b*Center of Photochemistry, Russian Academy of Sciences,
7a ul. Novatorov, 119421 Moscow, Russian Federation*

The synthesis of 2,2-diphenyl-2*H*-benzo[*f*]chromene containing the aza-18-crown-6-ether fragment was described. Its complex formation with alkaline-earth metal ions in dibutyl phthalate and the polymeric gelatin film was investigated. The treatment of the layers containing the ionophore mentioned with aqueous solutions of the corresponding salts in the presence of NaBPh₄ as the phase transfer catalyst results in the extraction of the metal cation into the polymeric layer due to complex formation with crown-containing benzo[*f*]chromene. The complex formation is accompanied by changes in the spectroscopic characteristics of chromene in the closed and open forms and an increase in the lifetime of the merocyanine form. The effects obtained depend on the metal cation concentration in a solution and the time of layer treatment.

Key words: 2,2-diphenyl-2*H*-benzo[*f*]chromene, aza-18-crown-6 ether, complex formation, alkaline-earth metal cations, gelatin, emulsion, polymeric layers.

Photochromic crown ethers demonstrate an optical response to complex formation due to changes in the structure caused by phototransformations.^{1–7} Systems based on the latter are promising for use in various practical applications (photoswitchable extractions of metal cations, molecular electronics, systems of optical recording of information, and nonlinear optical devices).

Benzo[*f*]chromenes also comprise a promising class of compounds.^{8–13} Intense works on the development of new representatives of this series with a high coloration level and stable upon introduction into polymeric matrices are evoked by their use in the production of photochromic lenses and coatings.

We have recently^{6,7} reported the synthesis of benzo[*f*]chromene containing the aza-15-crown-5-ether fragment as the substituent. It is shown that in an MeCN solution this compound can react with alkaline-earth metal cations, and complex formation is accompanied by considerable spectral changes. The photoinduced formation of the open form of benzo[*f*]chromene decreases the ability of the ligand to bind metal cations.

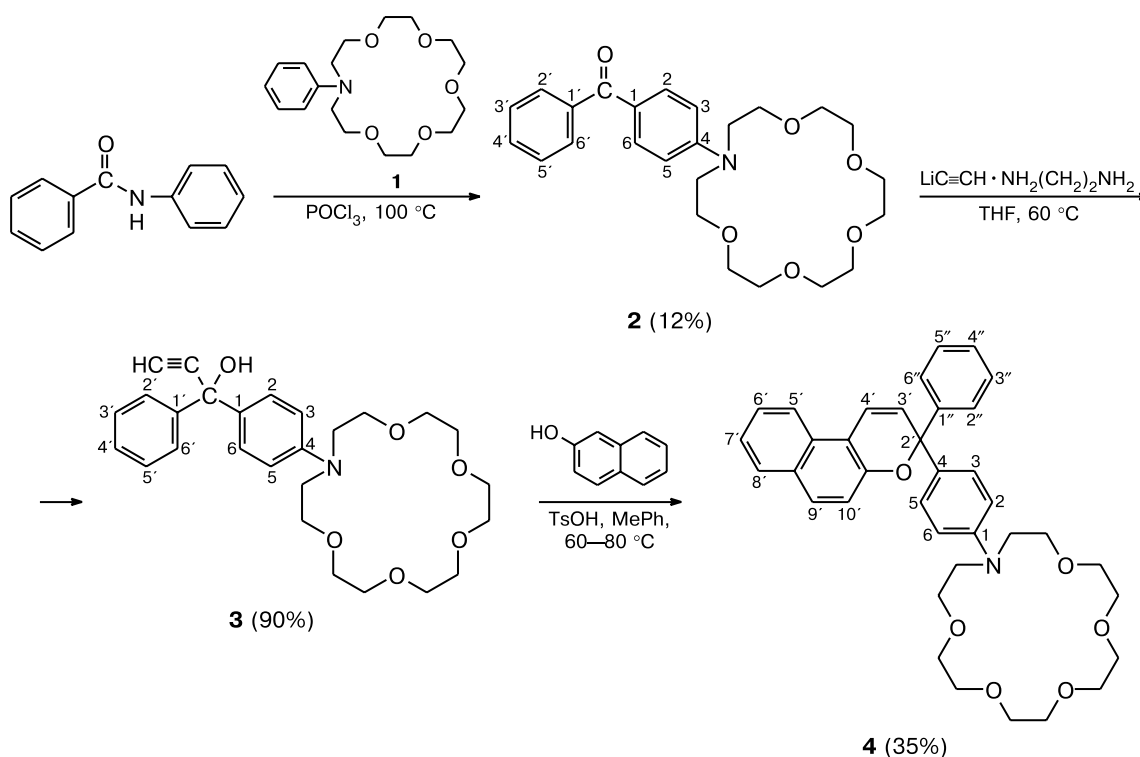
The purpose of the present work is to study the complex formation of benzo[*f*]chromene containing the crown-ether fragment in the polymeric film with alkaline-earth metal cations in aqueous solutions of the corresponding salts. This process gives an optical response.

For this purpose, we synthesized benzo[*f*]chromene similar in structure with the compound synthesized and obtained previously^{14,15} but containing the aza-18-crown-6-ether fragment as the ionophoric substituent with higher binding constants with alkaline-earth metal cations compared to those of aza-18-crown-6 ether.¹⁶ Comparison of the results of this study with the data obtained by us previously for MeCN solutions enables the analysis of processes in the complicated system of gelatin layers.

Results and Discussion

Synthesis of 2,2-diphenyl-2*H*-benzo[*f*]chromene containing the aza-18-crown-6-ether fragment. The multistep process of the synthesis of benzo[*f*]chromene **4** studied in this work is presented in Scheme 1. Diaryl ketone **2** containing the aza-18-crown-6-ether fragment was synthesized by the acylation of benzanilide with phenylaza-18-crown-6 ether (**1**) in the presence of POCl₃ via the Vilsmeier reaction.¹⁷ The corresponding propargyl alcohol **3** was synthesized from diaryl ketone **2** using the ethylenediamine complex of lithium acetylenide LiC≡CH·NH₂(CH₂)₂NH₂. The reaction of compound **3** with 2-naphthol in the presence of TsOH in toluene afforded crown-containing benzo[*f*]chromene **4** in 35% yield.

Scheme 1



Complex formation of chromene **4 in solutions of dibutyl phthalate.** Since in the obtained polymeric layer (see Experimental) chromene **4** exists as an emulsion of its solution in dibutyl phthalate (DBP), complex formation in pure DBP was preliminarily studied. The absorption spectra of a solution of chromene **4** in DBP in the presence of the Ba^{2+} cations at different cation : chromene **4** molar ratios (curves 1–7) and the absorption spectrum of

the complex with the Mg^{2+} ion in a 20-fold excess of the latter (curve 8) are presented in Fig. 1. It is well seen that an increase in the cation concentration results in systematic changes in the absorption spectrum, and the influence of the Mg^{2+} and Ba^{2+} cations is almost the same.

Scheme 2

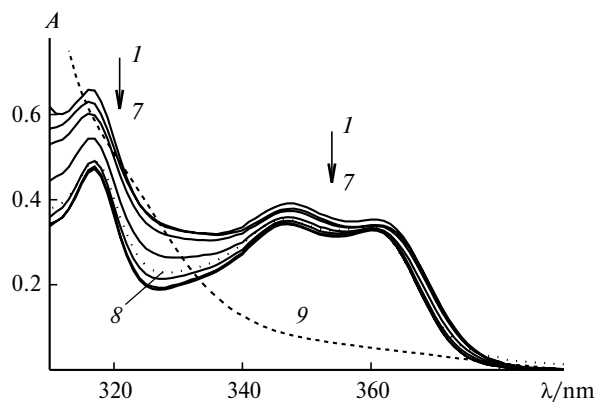
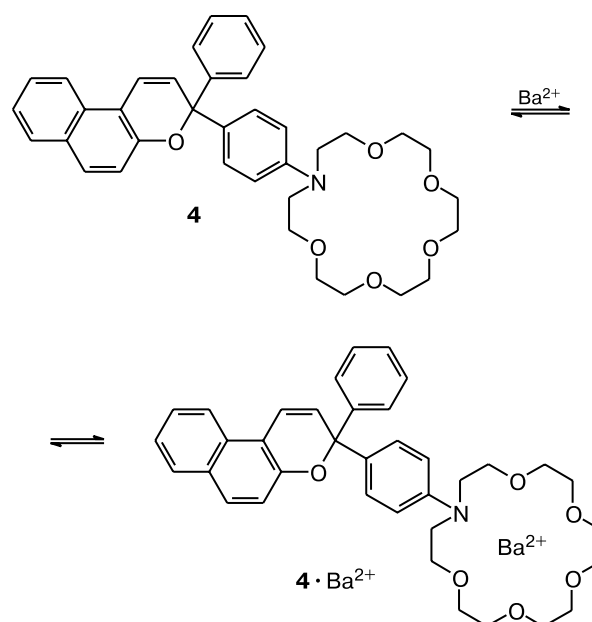


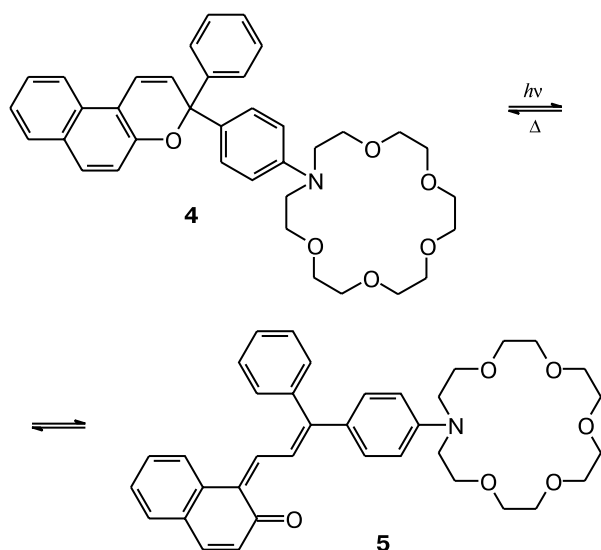
Fig. 1. Absorption spectra of solutions of chromene **4** in DBP ($C = 7.62 \cdot 10^{-5} \text{ mol L}^{-1}$) at different Ba^{2+} : chromene **4** molar ratios: 0 (1), 0.1 (2), 0.2 (3), 0.4 (4), 1 (5), 5 (6), and 20 (7). Curve 8 corresponds to the addition of 20-fold Mg^{2+} excess to a solution of chromene **4**. Curve 9 is the absorption spectrum of DBP in a cell with $l = 1 \text{ cm}$.

Therefore, only the barium cation was used in further studies.

The changes observed in the absorption spectrum of the closed form of benzo[f]chromene **4** in the presence of metal cations are caused by complex formation involving the crown-ether fragment (Scheme 2). These spectral changes are similar to those observed previously^{14,15} by the study of the complex formation of aza-15-crown-5-containing benzo[f]chromene with alkaline-earth metal cations in a MeCN solution.

Irradiation of a solution of compound **4** results in the formation of the photoinduced form **5** (Scheme 3).

Scheme 3



The photostationary absorption spectrum of compound **5** also changes in the presence of the Ba²⁺ cations (Fig. 2). The open form **5**, unlike starting benzo[f]chromene **4**, has two coordination sites capable of complex

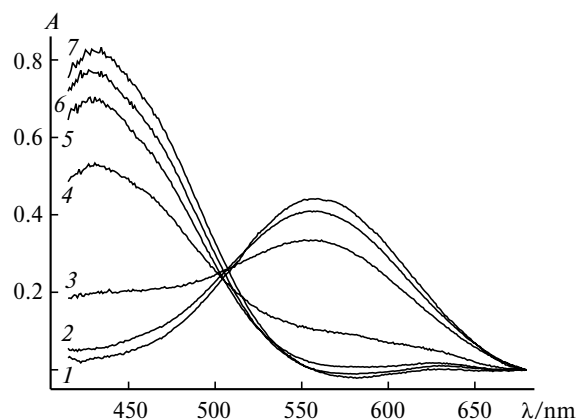
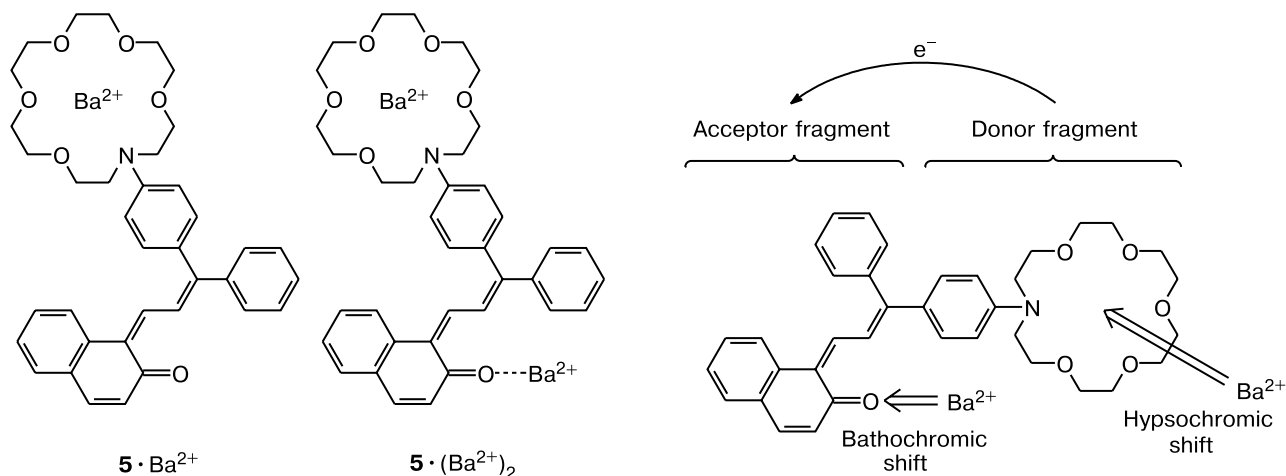


Fig. 2. Photostationary absorption spectra of a solution of compound **5** ($C = 7.5 \cdot 10^{-5} \text{ mol L}^{-1}$) in DBP at different [Ba²⁺] : [**4**] molar ratios: 0 (**1**), 0.1 (**2**), 0.25 (**3**), 0.8 (**4**), 1.25 (**5**), 5 (**6**), and 10 (**7**). To obtain a solution of compound **5**, sample **4** was excited with the light from a DRS-1000 mercury lamp through the UFS-6 light filter in a cell with $l = 1 \text{ cm}$.

formation: the crown-ether fragment and O atom of the merocyanine form.

As shown by our studies, in the case of benzo[f]chromene containing the aza-15-crown-5 fragment, in a MeCN solution the formation of a metal cation complex to the crown-ether fragment is accompanied by the hypsochromic shift of the long-wave absorption band, whereas the coordination of the cation with the O atom, on the contrary, results in the bathochromic shift in the absorption spectrum of the merocyanine form.¹⁵ These spectral changes can be explained if the molecule of the open form of benzochromene can be presented as consisting of two moieties (Scheme 4): donor (crown-ether) and acceptor. The absorption spectrum of the compound is a characteristic of the charge transfer from the donor to the acceptor moiety. Complex formation to the crown-ether fragment should prevent the electron density transfer, re-

Scheme 4



sulting in the hypsochromic shift. By contrast, the coordination of the O atom with the cation enhances the withdrawing properties of this part of the molecule facilitating the easier charge transfer and, as a consequence, it is accompanied by the bathochromic shift of the long-wave absorption band.¹⁵

The observed considerable hypsochromic shift in the UV spectrum of the open form **5** in a DBP solution in the presence of the Ba^{2+} cations indicates that the formation of the $5 \cdot \text{Ba}^{2+}$ complex involving the crown-ether fragment of the compound is the main process. The stability of this complex is higher than that of the complex formed by the coordination of the O atom of the merocyanine form with the metal cation, because the macrocycle contains several heteroatoms for binding with the metal cation due to the so-called "macrocyclic effect."^{14,15}

Coordination with the metal cation results in the electron density redistribution along the whole conjugation chain, which increases considerably the lifetime of the open form **5**. This is confirmed by the data presented in Fig. 3: the lifetime of the open form increases significantly with an increase in the cation concentration. However, this dependence is explicitly nonlinear with some induction period (see Fig. 3). At the $[\text{Ba}^{2+}] : [\mathbf{5}]$ ratios from 0 to 0.5, the influence is very low followed by the region of efficient response ($[\text{Ba}^{2+}] : [\mathbf{5}] = 0.5\text{--}1.5$) and, finally, the saturation region is observed, which also can be considered linear but with much lower sensitivity to changes in the Ba^{2+} ion concentration. Most likely, sharp changes in the spectra correspond to the formation of the $5 \cdot \text{Ba}^{2+}$ complex, and the subsequent smooth changes correspond to the formation of the $5 \cdot (\text{Ba}^{2+})_2$ complex. It is known¹⁵ that the stability of the complex with the O--Ba^{2+} coordination is insignificant, and its formation requires high cation excess.

Study of complex formation of chromene **4 in gelatin layers.** In order to study the influence of metal cations on

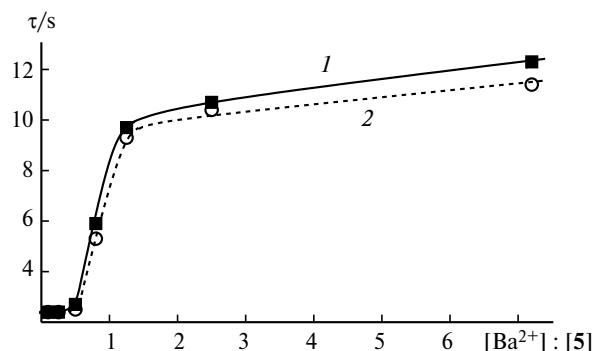


Fig. 3. Changes in the lifetime (τ) of the open chromene form with an increase in the concentration of the Ba^{2+} cations in DBP: $\lambda = 550$ (1) and 440 nm (2) (cell with $l = 1$ cm, chromene concentration $7.5 \cdot 10^{-5}$ mol L^{-1} ; the sample was excited with the light from a DRSh-1000 mercury lamp through the UFS-6 light filter).

the properties of chromene **4** in the gelatin layer, the prepared samples were treated with aqueous solutions of the corresponding metal perchlorates in the presence of NaBPh_4 used as a phase transfer catalyst, which accelerates the ion equilibration, after which the layer was rinsed with water and dried under standard conditions. Reference experiments showed that the treatment of the layers with water and aqueous solutions of the SV-81 surfactant exerted no effect on the spectroscopic and photochromic properties of chromene, whereas the treatment with aqueous solutions containing the Ba^{2+} ion results in effects similar to those observed in solutions. However, note that the solubility of alkaline-earth metal perchlorates in water exceeds their solubility in DBP, and the volume of the organic phase (DBP) in the film is much smaller than the water volume in the treating solution. Therefore, only a very small portion of the Ba^{2+} ions can diffuse into DBP microdrops from an aqueous solution. For this reason the absorption spectrum changes insignificantly. The photostationary absorption spectra and plots of the lifetime of the open form are much more sensitive. The photostationary absorption spectra of the layer treated with an aqueous solution of $\text{Ba}(\text{ClO}_4)_2$ for 5 h (curve 1) and the reference layer stored in water for 5 h (curve 2) are presented in Fig. 4. For clarity, curve 3 represents the differential spectrum of curves 1 and 2, which is analogous to the photostationary spectra obtained in DBP solutions at high Ba^{2+} cation excess (see Fig. 2).

The plot of the lifetime of the merocyanine form **5** vs. duration of treatment of the sample with a solution of $\text{Ba}(\text{ClO}_4)_2$ is presented in Fig. 5. The lifetime increases almost threefold at longer duration of treatment and is a very sensitive parameter indicating the presence of the Ba^{2+} cation in an aqueous solution.

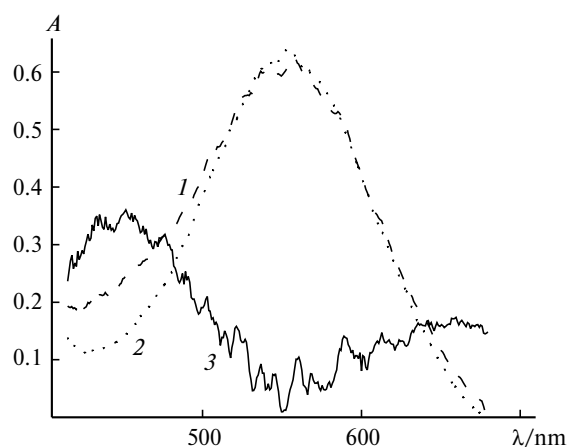


Fig. 4. Photostationary absorption spectra of the film sample treated with an aqueous solution containing the Ba^{2+} cations (1) and with water (2) and the differential photostationary spectrum of compounds **4** and **5** (multiplied by the coefficient 2.5 for clarity). The layer was excited with the light from a DRSh-1000 mercury lamp through the UFS-6 and BS-7 light filters.

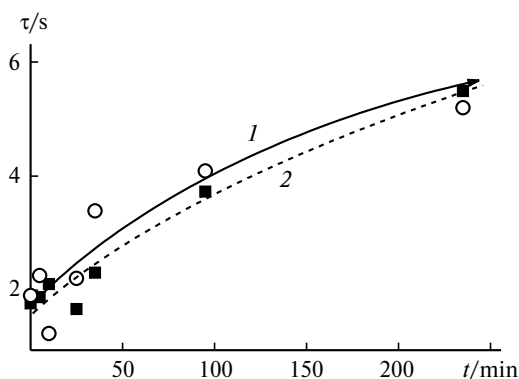


Fig. 5. Plots of the lifetime (τ) of the open form of chromene **4** in the film vs. time of treatment of the sample (t) with an aqueous solution of $\text{Ba}(\text{ClO}_4)_2$ in the presence of NaBPh_4 ($C = 4.87 \cdot 10^{-4} \text{ mol L}^{-1}$): $\lambda = 550$ (1) and 440 nm (2).

Thus, benzo[f]chromene **4** introduced into the gelatin layer as an emulsion of microdrops of its solution in DBP retains the photochromic properties. UV irradiation in the absorption region of the closed form induces the merocyanine isomer with the characteristic absorption band at $\lambda = 550 \text{ nm}$ and a lifetime of $\sim 2 \text{ s}$. The treatment of such layers with aqueous solutions of alkaline-earth metal salts in the presence of the phase transfer catalyst (NaBPh_4) changes both the spectroscopic characteristics of chromene in the closed and open forms and the lifetime of the merocyanine form, increasing the latter by almost three times depending on the metal cation concentration in a solution and duration of the layer treatment. These changes are qualitatively the same as those occurred in homogeneous solutions of chromene in DBP when alkaline-earth metal perchlorates are added. The main conclusion is that chromene in the polymeric layer reacts to the presence of metal cations in the aqueous phase as in the case of homogeneous solutions. Reactions of the future sensor polymeric layer can easily be simulated using this property.

Experimental

^1H NMR spectra were recorded on a Bruker DRX-500 spectrometer (working frequency 500.13 MHz) in CDCl_3 . Chemical shifts were measured with an accuracy of 0.01 ppm, and the accuracy of measuring spin-spin coupling constants was 0.1 Hz. Mass spectra were obtained on a Varian MAT 311A instrument. High-resolution mass spectra were obtained on a Finnigan MAT 8430 instrument (ionization energy 70 eV, direct injection of the sample into the ionization region). The course of the reaction was monitored by TLC on 25 DC-Alufolien Kieselgel 60 F_{254} and DC-Alufolien Aluminiumoxid 60 F_{254} neutral (Typ E, Merck) plates. Commercial reagents and solvents (Merck) were used in the syntheses of compounds **2–4**.

The following reagents were used for the preparation of gelatin layers with benzo[f]chromene **4**: distilled water, NaBPh_4 (Aldrich), photoinert bone gelatin (trade mark A), surfactant

SV-81, and DBP (Reakhim) purified by double vacuum distillation. Absorption spectra were recorded on Specord M40 and Perkin—Elmer Lambda EZ210 spectrophotometers. Photo-stationary absorption spectra and lifetimes of the open form were measured on an original laboratory setup. Emulgation was carried out with an UZDN-A ultrasonic dispersator. Gelatin films were supported using a special spray stage, whose temperature was maintained constant and the horizontal position of the surface was controlled.

[4-(1,4,7,10,13-Pentaoxa-16-azacyclooctadec-16-yl)phenyl]phenylmethanone (2). A mixture of benzanilide (0.2 g, 1 mmol) and phenylaza-18-crown-6 ether (**1**) (0.7 g, 2 mmol) was heated with stirring to melting and then cooled to $\sim 20^\circ\text{C}$. POCl_3 (0.3 g, 2 mmol) was added dropwise to the mixture along with cooling with ice. Then the mixture was stored at 100°C for 4 h in an oil bath and dissolved in 15% HCl on heating ($\sim 30^\circ\text{C}$). The resulting solution was filtered to remove an orange insoluble precipitate, and a 10% solution of NaOH was added to the filtrate until an alkali reaction was shown by litmus paper. The reaction mixture was extracted with benzene, the extract was washed with water, and the solvent was evaporated. The product was purified by column chromatography on silica gel (silica gel L 40—100 μm (Chemapol) using a benzene—MeOH (from 20 : 1 to 10 : 1) mixture as eluent or Kieselgel 60 (Merck) using a heptane—AcOEt (from 2 : 1 to 1 : 3) mixture as eluent). The yield of ketone **2** was 0.053 g (12%, oil). ^1H NMR, δ : 3.67 (br.s, 16 H, 8 CH_2O); 3.74 (m, 8 H, 4 CH_2O); 6.70 (d, 2 H, H(3), H(5), $J = 9.0 \text{ Hz}$); 7.46 (t, 2 H, H(3'), H(5'), $J = 7.6 \text{ Hz}$); 7.53 (t, 1 H, H(4'), $J = 7.4 \text{ Hz}$); 7.73 (d, 2 H, H(2'), H(6'), $J = 7.3 \text{ Hz}$); 7.78 (d, 2 H, H(2), H(6), $J = 9.0 \text{ Hz}$). Mass spectrum, m/z (I_{rel} (%)): 443 [M] $^+$ (100), 267 (28), 266 (34), 254 (40), 224 (53), 210 (65), 209 (48), 132 (62), 105 (74), 76 (48). High-resolution mass spectrum, found: m/z 443.2301 [M] $^+$. $\text{C}_{25}\text{H}_{33}\text{NO}_6$. Calculated: $M = 443.2307$.

1-[4-(1,4,7,10,13-Pentaoxa-16-azacyclooctadec-16-yl)phenyl]-1-phenylprop-2-yn-1-ol (3). A solution of diaryl ketone **2** (0.44 g, 1 mmol) in anhydrous THF (25 mL) was added dropwise to a suspension of $\text{LiC}\equiv\text{CH}\cdot\text{NH}_2(\text{CH}_2)_2\text{NH}_2$ (0.46 g, 5 mmol) in anhydrous THF (25 mL) for 1 h at 60°C with stirring. The reaction mixture was stirred for 2 h at 60°C , then water was added, and the product was extracted with AcOEt. The organic fraction was washed with water to the neutral reaction by litmus paper, and the solvent was evaporated. The residue was extracted with hot hexane, and a light yellow product was precipitated on cooling. The yield of compound **3** was 0.42 g (90%, oil). ^1H NMR, δ : 2.67 (s, 1 H, OH); 2.85 (s, 1 H, $\text{C}\equiv\text{CH}$); 3.65 (br.s, 24 H, 12 CH_2O); 6.63 (d, 2 H, H(3), H(5), $J = 8.8 \text{ Hz}$); 7.27 (m, 1 H, H(4')); 7.34 (m, 2 H, H(3'), H(5')); 7.39 (d, 2 H, H(2), H(6), $J = 8.7 \text{ Hz}$); 7.63 (d, 2 H, H(2'), H(6'), $J = 7.0 \text{ Hz}$). Mass spectrum, m/z (I_{rel} (%)): 469 [M] $^+$ (7), 443 (40), 266 (35), 254 (32), 236 (30), 224 (56), 210 (65), 132 (52), 105 (100), 77 (19). High-resolution mass spectrum, found: m/z 469.2457 [M] $^+$. $\text{C}_{27}\text{H}_{35}\text{NO}_6$. Calculated: $M = 469.2464$.

16-[4-(2-Phenyl-2H-benzo[f]chromen-2-yl)phenyl]-1,4,7,10,13-pentaoxa-16-azacyclooctadecane (4). A mixture of alcohol **3** (0.47 g, 1 mmol) and 2-naphthol (0.15 g, 1 mmol) in anhydrous toluene (10 mL) was prepared on stirring and heating (to $60\text{--}80^\circ\text{C}$). $\text{TsOH}\cdot\text{H}_2\text{O}$ (0.02 g, 0.1 mmol) was added, and the mixture was stirred for 3 h. Then water was added to the mixture, the solvent was evaporated, and the residue was purified by column chromatography on Al_2O_3 (Aluminiumoxid

aktiviert basisch (Typ 5016A, Merck), benzene—AcOEt (3 : 1) mixture as eluent). The yield of chromene **4** was 0.21 g (35%), m.p. 78–80 °C (from hexane). Found (%): C, 74.48; H, 6.98; N, 2.31. $C_{37}H_{41}NO_6$. Calculated (%): C, 74.60; H, 6.94; N, 2.35. 1H NMR, δ : 3.52 (m, 4 H, 2 CH_2O); 3.54 (m, 8 H, 4 CH_2O); 3.56 (br.s, 8 H, 4 CH_2O); 3.60 (m, 4 H, 2 CH_2O); 6.25 (d, 1 H, $H(3')$, $J = 10.0$ Hz); 6.66 (d, 2 H, $H(2)$, $H(6)$, $J = 8.9$ Hz); 7.22 (d, 1 H, $H(10')$, $J = 8.8$ Hz); 7.26 (m, 2 H, $H(3)$, $H(5)$); 7.27 (m, 1 H, $H(4'')$); 7.37 (m, 4 H, $H(3'')$, $H(5'')$, $H(7')$, $H(4')$); 7.52 (m, 1 H, $H(6'')$); 7.53 (d, 2 H, $H(2'')$, $H(6'')$, $J = 8.9$ Hz); 7.74 (d, 1 H, $H(9')$, $J = 8.9$ Hz); 7.79 (d, 1 H, $H(8')$, $J = 8.0$ Hz); 8.04 (d, 1 H, $H(5')$, $J = 8.5$ Hz). Mass spectrum, m/z (I_{rel} (%)): 595 [M] $^+$ (100), 257 (57), 168 (26), 105 (22), 100 (24), 87 (28), 82 (29), 77 (26), 71 (40), 69 (37).

Preparation of gelatin layers with benzo[*f*]chromene **4.** Since the chromene content in the polymeric layer permissible for aqueous solutions of metal cations should remain constant during treatment and chromene **4** is soluble in both organic solvents and water, to prepare the working sample, we used a procedure, according to which benzo[*f*]chromene **4** was first dissolved in the organic hydrophobic solvent DBP and then emulgated in an aqueous solution of the natural polymer gelatin. The drop size was determined on a CAPA-500 analyzer of particle size. The resulting emulsion was supported on a glass support. A similar procedure is widely used in the production of colored photographic materials to introduce protected color-forming components into layers and provides reliable, well reproducible results.¹⁸

In order to obtain layers with the optimum characteristics (absorption, thickness, transparency), we studied the dependences of the size of emulsion drops on the ratio of the hydrophobic and aqueous phases, amount and concentration of a surfactant, gelatin concentration, and duration of ultrasonication of the emulsion. The results are presented in Figs. 6 and 7, which show that at concentrations of gelatin of 5% and SV-81 surfactant $\geq 6\%$ the average drop size remains unchanged during sonication of the emulsion for 10 min and longer.

Taking into account the data obtained, we formulated the following procedure of layer preparation: a solution (0.8 mL) of benzo[*f*]chromene **4** in DBP ($C = 1.6 \cdot 10^{-2}$ mol L^{-1}) and a 6.5% solution (0.48 mL) of the SV-81 surfactant in an EtOH—water

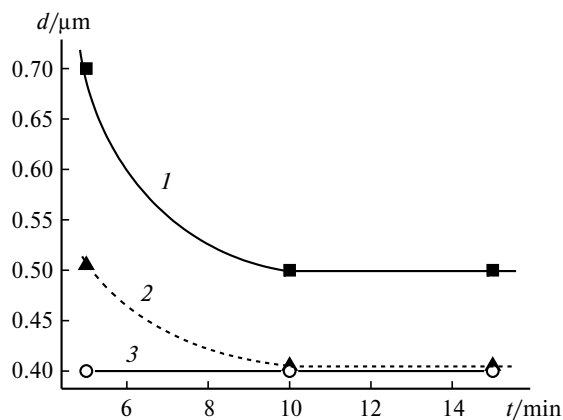


Fig. 6. Plots of the average diameter of microdrops (d) of the DBP emulsion in a 5% solution of gelatin vs. time of sonication (t) at different concentrations of the SV-81 surfactant: 5 (1), 6 (2), and 10% (3).

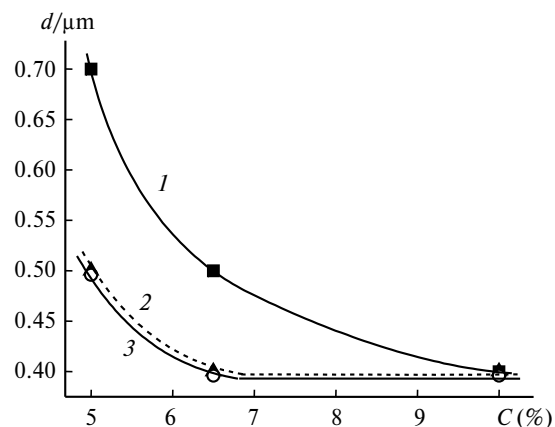


Fig. 7. Plots of the average diameter of microdrops (d) of the DBP emulsion in a 5% solution of gelatin vs. concentration of the SV-81 surfactant (C) at different times of sonication: 5 (1), 10 (2), and 15 min (3).

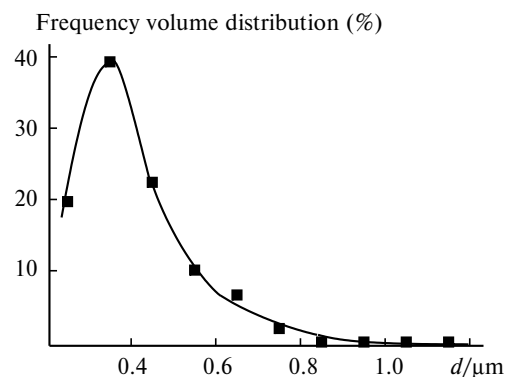


Fig. 8. Particle volume distribution of DBP microdrops in the emulsion (d is the diameter of microdrops).

(1 : 1) mixture was added to a 5% (wt.%) aqueous solution (5.12 mL) of gelatin. The mixture was sonicated for 10 min. As a result, an emulsion with the average diameter of microdrops $d = 0.4$ μm and a rather narrow particle volume distribution (Fig. 8) was obtained with a good reproducibility.

The prepared emulsion was supported at 40 °C onto a glass support on a horizontal spray stage and dried at ~ 20 °C. A solution of benzo[*f*]chromene **4** in DBP with a concentration of $1.6 \cdot 10^{-2}$ mol L^{-1} should be used to prepare the layer with the absorption $A = 0.5$ at $\lambda = 347$ nm (when a noticeable effect of the solvent (DBP) absorption is absent). The surface area of the supports was 20 cm^2 , and the emulsion volume on the plate was 1 cm^3 . Under these conditions of supporting, the total thickness of the dry layer was ~ 85 μm . The prepared layers sustain a prolonged treatment (at least 8 h) in aqueous solutions of metal salts without layer decomposition and without losses of introduced chromene. The $NaBPh_4$ phase transfer catalyst ($C = 4.87 \cdot 10^{-4}$ mol L^{-1}) was added to a solution containing metal ions to accelerate the penetration of the metal cations from the aqueous phase into the DBP microdrops.

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