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To the 80th Anniversary of R. A. Cherkasov

Interaction of Poly(benzoyl thiocarbamate)-Modified Hyperbranched Polyester with Co(II) and Cu(II) Nitrates

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Abstract—The interaction of $Co(NO_3)_2$ and $Cu(NO_3)_2$ with hyperbranched polyester containing 7 terminal benzoyl thiocarbamate groups has been studied by IR-Fourier and electron absorption spectroscopy. A new polynuclear complexes of Co(II) and Cu(II) with poly(benzoyl thiocarbamate)-modified hyperbranched polyester have been synthesized. It has been found that the oxygen and sulfur atoms of the peripheral benzoyl thiocarbamate fragments of the macroligand are involved in coordination with metal ions, the metal to ligand ratio being 7 : 1.

Keywords: metal complexes of copper(II) and cobalt(II), hyperbranched polyether, poly(benzoyl thiocarbamate)

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Development of extraction-sorption systems for the recovery of ions of *d*-metals and rare-earth elements from natural and recycling objects is an important scientific and practical issue. Parameters of group separation or individual recovery of the metal ions is determined by the nature and composition of the extracting system as well as the process conditions. The extraction-sorption systems contain the reagents exhibiting coordination activity towards metal ions [1–6]. A remarkable class of them are O-alkyl-N-aroyl carbamates and thiocarbamates which have been successfully applied as highly efficient extracting agents for metal ions [7–11] as well as collectors for the recovery of trace amounts of metals during ore processing [12–14].

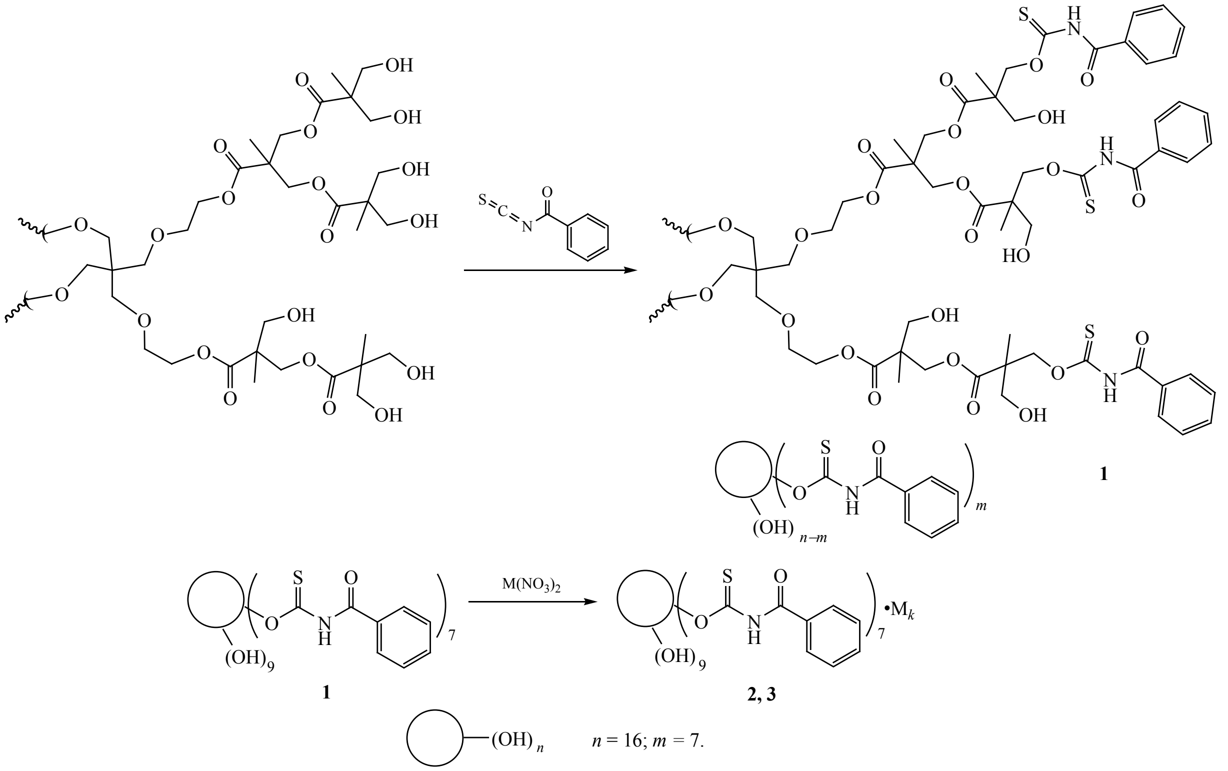
The introduction of macromolecules (including polymers of diverse nature and architecture) in the composition of the extraction-sorption systems can enhance the parameters of metal ions recovery due to a combination of specific and nonspecific interactions [15–21]. Promising reagents for the creation of highly efficient extraction-sorption systems include hyperbranched polyesters of dendrite structure, sparse core, and high concentration of the terminal groups [22, 23] exhibiting good solubility [24, 25], low viscosity [26, 27], biodegradability [26], low toxicity [28, 29], and pronounced complex forming properties [30, 31]. Modification of the terminal groups of polyester with efficiently coordinating (thio)carbamate fragments allows creation of efficient extraction-sorption systems for the recovery and separation of metal ions. The prospects of this approach has been confirmed by the complex formation of hyperbranched polyesters of second and third generation containing different amount of terminal thenylthiocarbamate groups with Cu(II) ions [32]. Biological activity of the thiocarbamate derivatives of polyesterpolyols can be determined by fungicide [32, 33], antitumor, and antiinflammatory properties [34–38] of the modifier.

Evaluation of the complexing activity of hyperbranched polyesterpolyol of the second generation modified with terminal benzoyl thiocarbamate groups towards Co(II) and Cu(II) ions is demanded for further development of colloidal extraction-sorption systems.

Hyperbranced polyester of the second generation **1** (Scheme 1) containing seven terminal benzoyl thiocarbamate groups (functionalization degree 0.44) was synthesized as described elsewhere [39]. Terminal benzoyl thiocarbamate fragments can form coordination nodes in the hyperbranched polyester scaffold.

To evaluate the complexing properties of compound **1**, its interaction with Co(II) and Cu(II) nitrates in

Scheme 1.



M = Co(II), Cu(II).

DMSO was studied by means of spectrophotometry. The components of the absorption spectra were obtained via deconvolution into Gaussian peaks.

Electronic absorption spectra of compound **1** in DMSO (Fig. 1) contained the characteristic bands at 250–430 nm, the bands in visible spectral range being absent. A strong broad band at 273 nm (ε 40531 L·cm^{-1}·mol^{-1}) was due to the π–π*- and *n*–π*-transitions in the benzoyl (255 nm) and carbonyl (270 nm) groups of the polyester core. A weak band at 345 nm (ε = 731 L·cm^{-1}·mol^{-1}) was assigned to the *n*–π*-transitions in the C=S group.

Introduction of $Co(NO_3)_2$ or $Cu(NO_3)_2$ into a solution of compound **1** was accompanied by bathochromic shift of the PhC(O) absorption band to 260 nm and hypochromic effect of the band at 270 nm, pointing at the participation of carbonyl groups in the coordination with Co(II) and Cu(II) ions.

The study of the electronic spectra of the $Co(NO_3)_2$–compound **1** system with the increase in the salt concentration, the ligand concentration being constant (Fig. 2), revealed that the position of the C=S band remained unchanged at the Co(II)–compound **1** molar ratio up to 4 : 1. The increase in the molar ratio to 10 : 1 led to the hypsochromic shift of the band to 338 nm, which could point at the C=S group participation in the coordination of Co(II) ions at the reagents molar ratio above 4 : 1. Introduction of $Cu(NO_3)_2$ into a solution of compound **1**, the Cu(II)–compound **1** molar ratio being varied between 0.5 : 1 and 10 : 1 led to hypsochromic shift of the C=S absorption band to 318 nm accompanied by significant hyperchromic effect up to 1.74 optical units, which could point at the thiocarbonyl group coordination with Cu(II) over the probed range of the reagents molar ratio.

Electronic absorption spectra of the Co salt–compound **1** system showed the increase in the absorption at 538 nm and the appearance of a new band of the *d*→*d*-transitions at 673 nm corresponding to the $^4T_{1g}(P)\rightarrow{}^4T_{1g}$ and $^4T_{2g}\rightarrow{}^4T_{1g}$ terms (Fig. 3). In the case of Cu(II) ion, the appearance of the octahedral coordination nodes led to the appearance of a similar band at λ = 836 nm.

Composition and conditional stability constants of coordination compounds of polyester **1** (ligand L) with Co(II) and Cu(II) ions were calculated using the Jobs method [40, 41]. Polynuclear complexes with M : L = 7 : 1 were formed in both systems, which was further

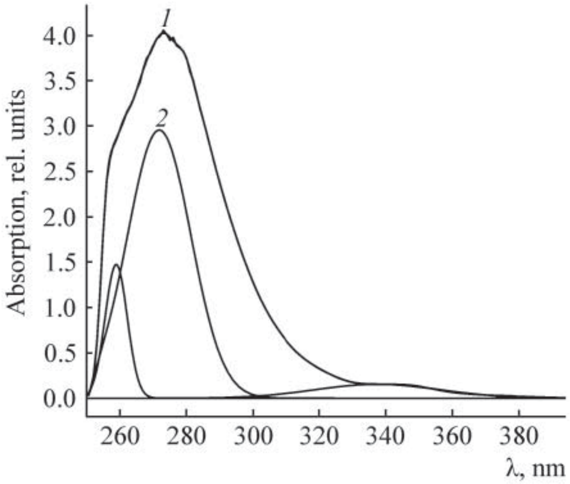


Fig. 1. Electronic absorption spectra of compound **1** in DMSO: (*1*) experiment, (*2*) interpolation from the Gauss distribution ($c_1 = 1\times10^{-4}$ mol/L).

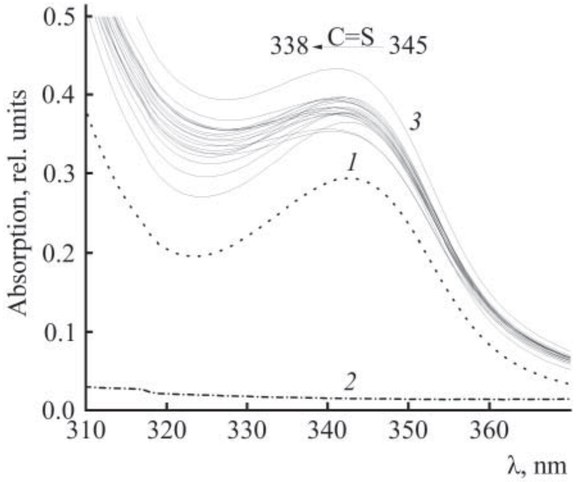


Fig. 2. Electronic absorption spectra of compound **1** (*1*), $Co(NO_3)_2$ (*2*), the $[Co(NO_3)_2$–compound **1**] system (*3*) over 310–370 nm. $c_1 = 1\times10^{-4}$ mol/L, $[Co(NO_3)_2] = 5\times10^{-5}$–$1.0\times10^{-3}$ mol/L.

confirmed by elemental analysis data. Stability of the complex with Co(II) was higher in comparison with Cu(II), logarithms of the conditional stability constants being 17.30±2.77 and 15.92±2.28, respectively.

Basing on the obtained data on the complex formation of Co(II) and Cu(II) with compound **1**, metal-polymer complexes **2** and **3** were synthesized (Scheme 1). Parameters of the electronic absorption spectra of complexes **2**, **3** and Co(II)/Cu(II)–compound **1** in DMSO were similar and confirmed the participation of carbonyl and thioxo peripheral functional groups in the complexes formation. Moreover, the electronic spectrum of complex **3** contained a new band at 805 nm assigned to the charge transfer in the Cu–S group. Cu(II) ion was in the octahedral surrounding with tetragonal distortion [42].

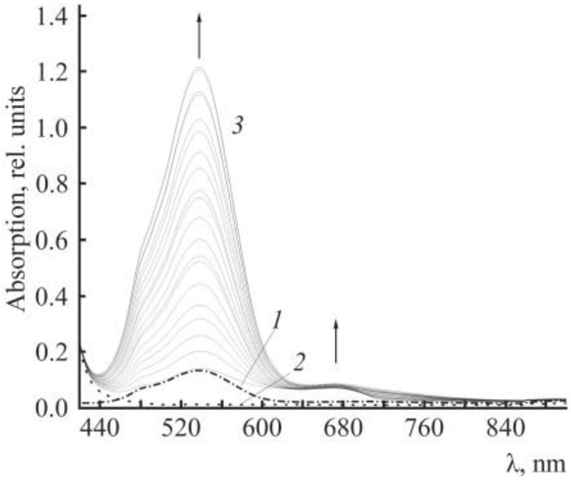
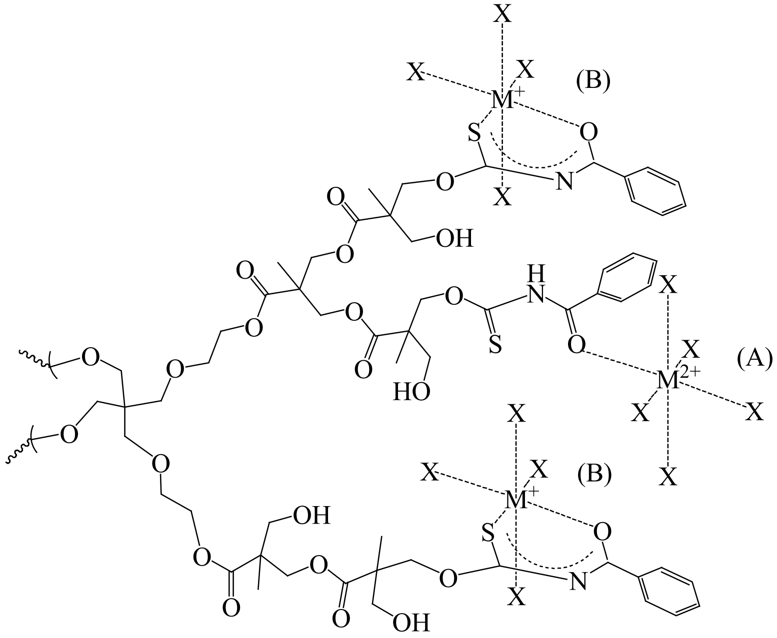


Fig. 3. Electronic absorption spectra of compound **1** (*1*), $Co(NO_3)_2$ (*2*), the $[Co(NO_3)_2$–compound **1**] system (*3*) over 420–900 nm. $c_1 = 1\times10^{-2}$ mol/L, $[Co(NO_3)_2] = 5\times10^{-3}$–$1.0\times10^{-1}$ mol/L.

Comparative analysis of IR spectra of complexes **2**, **3** and polydentate ligand **1** [39] pointed at the participation of the carbonyl C=O and thiocarbonyl C=S groups of the benzoyl thiocarbamate fragment in the coordination with metal ions. IR spectra of complexes **2** and **3** revealed twofold weakening and shift to lower frequency of stretching bands of the C=O bond of the benzoyl fragment to 1658 ($\Delta\nu = 3$ cm^{-1}, **2**) and 1584 cm^{-1} ($\Delta\nu = 77$ cm^{-1}, **3**) as well as strengthening and shift to higher frequency of stretching bands of the C=S bond of the benzoyl thiocarbamate fragment to 1405 (**2**) and 1407 cm^{-1} (**3**). Moreover, the participation of the benzoyl thiocarbamate fragment of the polymer in the formation of the coordination node with Co(II) ion was confirmed by the shift of the amide bands (amide II 1506 cm^{-1}, amide III 1301 cm^{-1}, amide V 817 cm^{-1}, amide IV 615 cm^{-1}). In the case of the copper(II) complex, the amide III band was strengthened twofold, and the amide IV band was shifted to lower frequency by 23 cm^{-1}. The spectra of both complexes contained the stretching bands of the M–O bond: Co–O at 529 cm^{-1} [43, 44] and Cu–O at 685 cm^{-1} [45].

In summary, the formation of coordination compounds of Co(II) and Cu(II) ions with hyperbranched polyesterpolyol of the second generation modified with terminal *N*-benzoyl thiocarbamate groups was stated by a set of physico-chemical methods. Analysis of the spectral

Scheme 2.



X = H_2O, DMSO.

data suggested that the complexes formation involved the donor atoms of oxygen and sulfur of the peripheral benzoyl thiocarbamate fragments of the polydentate ligand which afforded the octahedral coordination nodes A and B (Scheme 2) for cobalt(II) ions (depending on the metal fraction) and mainly node B for copper(II) ions.

The obtained data will be used for the creation of colloidal extraction-sorption systems based on hyperbranched polyesterpolyols functionalized with chelating groups, to be applied in the recovery of metal ions from the model solutions as well as technogenic and ecological monitoring objects.

EXPERIMENTAL

Hyperbranched polyester **1** containing 7 terminal benzoyl thiocarbamate groups (M = 2891 g/mol) was synthesized as described elsewhere [39], $Cu(NO_3)_2 \cdot 3H_2O$, $Co(NO_3)_2 \cdot 6H_2O$, and solvents (1.4-dioxane, ethanol, and petroleum ether) were purchased from Acros.

IR spectra were recorded using a Spectrum 400 spectrometer (PerkinElmer) equipped with an Almaz KRS-5 ATR attachment (4000–400 cm^{-1}, spacing 1 cm^{-1}, 10 scans). Electronic absorption spectra were recorded using a Lambda 750 spectrophotometer (PerkinElmer) (200–900 nm, 25±0.01°C, optical path l = 1 nm); the specimen temperature was maintained constant using a system containing constant-temperature cell holder, a Julabo MB-5A flow thermostat, and a PTP-1 Peltier unit. Absorbance was measured with accuracy of ±1%. 0.1 M $LiClO_4$ was used as background electrolyte.

Composition and stability of the complexes in the studied systems were determined from the spectral data using the Jobs method [40, 41]. The experiment conditions were as follows: for the $Co(NO_3)_2$–compound **1** system – $\lambda = 673$ nm, $c_1 = 1\times10^{-2}$ mol/L, $[Co(NO_3)_2] = 1\times10^{-3}–1.0\times10^{-1}$ mol/L, $\varepsilon_{complex} = 33.84$ L cm^{-1} mol^{-1}; for the $Cu(NO_3)_2$–compound **1** system – $\lambda = 836$ nm, $c_1 = 1\times10^{-3}$ mol/L, $[Cu(NO_3)_2] = 1\times10^{-4}–1.0\times10^{-2}$ mol/L, $\varepsilon_{complex} = 322.47$ L cm^{-1} mol^{-1}.

Complexes of compound 1 with Co(II) and Cu(II) ions. 0.5 g (0.14 mmol) of compound **1** was dissolved in 3 mL of 1,4-dioxane, and a solution of 1.4 mmol of $Cu(NO_3)_2 \cdot 3H_2O$ or $Co(NO_3)_2 \cdot 6H_2O$ in 2 mL of ethanol was added. The solution turned dark red (in the case of cobalt) and green (copper). The reaction mixture was stirred during 9 h at 40°C. The major part of the solvent was removed under vacuum, the residue was poured into petroleum ether, the precipitate was separated off and dried during 5 h in a vacuum oven at 50°C (1 mmHg).

Complex 2. Yield 92%, dark red tar. Electronic absorption spectrum (DMSO), λ_{max}, nm (ε, L mol^{-1} cm^{-1}): 262 (2674), 269 (2371), 281 (1744), 317 (378.4), 536 (112.22), 673 (33.84). IR spectrum, ν, cm^{-1}: 3700–3000 (H_2O, O–H, N–H, C–H_{Ph}), 1729 (C=O), 1658 (C=O_{Bz});

1606, 1449 ($C=C_{Ph}$); 1506, 1301 [(C–N), δ(N–H), amide II and III]; 1405 (C=S); 1449, 1081, 817, 752 [δ($C–H_{Ph}$)]; 925, 870 [γ($C–H_{Ph}$)], 925 (N–O); 925, 711 [γ($C=C_{Ph}$)]; 817, 615 [δ(N–H), amide V and IV]; 667 [δ($C=C_{Ph}$)], 529 (Co–O). Found, %: C 32.85; H 5.03; N 5.68; S 4.56; Co 8.23. $C_{138}H_{258}Co_7N_{21}O_{138}S_7$. Calculated, %: C 32.77; H 5.11; N 5.82; S 4.43; Co 8.17.

Complex 3. Yield 86%, dark green tar. Electronic absorption spectrum (DMSO), λ_{max}, nm (ε, L mol^{-1} cm^{-1}): 255 (21153.23), 274 (25420.08), 318 (10220.79), 805 (322.47). IR spectrum, ν, cm^{-1}: 3700–3000 (H_2O, O–H, N–H, $C–H_{Ph}$), 1726 (C=O), 1584 ($C=O_{Bz}$), 1456 ($C=C_{Ph}$), 1295 [(C–N), δ(N–H), amide III]; 1456, 1295, 1008 (N–O),1407 (C=S); 1456, 1008, 720 [δ($C–H_{Ph}$)]; 921, 870, 809 [γ($C–H_{Ph}$)]; 921, 685 [γ($C=C_{Ph}$)], 613 [δ(N–H), amide IV]; 685 (Cu–O). Found, %: C 35.29; H 4.72; N 6.05; S 4.63; Cu 9.47. $C_{138}H_{216}N_{21}O_{117}S_7Cu_7$. Calculated, %: C 35.16; H 4.59; N 6.24; S 4.76; Cu 9.51.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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