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Novel Cobalt Bis-*o*-semiquinonato Complexes with Bidentate N-Donor Ligands

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Abstract—Two new cobalt bis-*o*-semiquinonato complexes, (Pyz-Phen)Co(3,6-DBSQ)$_2$ (**I**) and (Bpyz)Co(3,6-DBSQ)$_2$ (**II**) (Pyz-Phen = pyrazino[2,3-f][1,10]phenanthroline, Bpyz = bipyrazine, 3,6-DBSQ = 3,6-di-*tert*-butyl-*o*-benzoquinone radical anion), were synthesized. According to X-ray diffraction data, both complexes have a trigonal-prismatic geometry of the inner coordination sphere. The distribution of C–O and Co–O bond lengths, which reflects the valence state of the metal and the ligands, indicates that the complexes are formed by cobalt(II) surrounded by two semiquinone radical anions. The results of magnetochemical measurements show that the pyrazino[2,3-f][1,10]phenanthroline complex is a derivative of low-spin divalent cobalt, whereas its bipyrazine structural analogue is a high-spin cobalt(II) derivative.

Keywords: cobalt complexes, *o*-semiquinones, trigonal prism, magnetic properties
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INTRODUCTION

Six-coordinate cobalt complexes based on redox-active ligands (in particular, sterically hindered *o*-quinones) attract considerable research interest because many of these compounds demonstrate redox isomerism [1–3]. In the general case, redox isomerism implies the reversible intramolecular electron transfer between the transition metal ion and the redox-active ligand induced by an external stimulus. In the case of cobalt bis-dioxolene derivatives, this process is accompanied by a change in the spin multiplicity of the metal ion. Redox isomerization takes place both in solutions [4] and in the crystalline state [5] under the action of factors such as temperature, pressure, magnetic field, and electromagnetic radiation of various ranges [6–9]. Since the change in the electronic molecular structure upon redox isomerization induces switching of macroscopic properties of complexes (magnetization, spectral characteristics, electrical conductivity), these compounds possess a considerable potential for application as components of molecular switches, sensors, memory storage devices, and other new-generation functional materials [10].

Characteristics of the redox isomerization in the crystalline phase are determined by the structural features of the packing of the complex [11, 12], the presence of guest solvent molecules in the lattice [13], and, most of all, by the electron-withdrawing properties of dioxolene and N-donor ligands contained in the complex [14, 15]. It is noteworthy that all currently known cobalt bis-dioxolene derivatives capable of redox isomerism have a distorted octahedral geometry. Complexes of analogous composition with a trigonal-prismatic geometry do not undergo redox-isomeric transformations, but exist only as bis-*o*-semiquinone derivatives of divalent high-spin cobalt [15–17]. On the other hand, high-spin Co^{2+} compounds with the indicated geometry behave as single-molecule magnets (SMMs) [18, 19].

In this study, we synthesized cobalt bis-dioxolene complexes based on 3,6-di-*tert*-butyl-*o*-benzoquinone (3,6-DBQ) containing pyrazino[2,3-f][1,10]phenanthroline (Pyz-Phen) and bipyrazine (Bpyz) as N-donor ligands and studied their molecular structure and magnetic properties.

EXPERIMENTAL

The complexes were synthesized in evacuated tubes in the absence of air or water traces. The solvents used in the study were purified according to recommendations [20]. Pyrazino[2,3-f][1,10]phenanthroline, bipyrazine, and dicobalt octacarbonyl were commercial chemicals (Aldrich). Elemental analysis was carried out on a Elementar Vario EL cube analyzer. IR spectra

(7000–400 cm^{-1} range) were recorded on an FSM-1201 Fourier Transform spectrometer in mineral oil using KBr cells. The temperature dependence of the magnetic susceptibility was measured on a Quantum Design MPMSXL SQUID magnetometer in the temperature range of 2–350 K in a 5000 Oe magnetic field. The paramagnetic components of the magnetic susceptibility (χ) were determined with allowance for the diamagnetic contribution, which was estimated from the Pascal constants. The effective magnetic moment (μ_{eff}) was calculated from the formula $\mu_{eff} = [3k\chi T/(N_A\mu_B^2)]^{1/2}$, where N_A, μ_B, and k are the Avogadro number, Bohr magneton, and Boltzmann constant, respectively. The computer simulation of the magnetic properties of the complexes and determination of the parameters of exchange interaction between the paramagnetic centers in the complexes was carried out using the Mjollnir software [21].

Synthesis of the complex (Pyz-Phen)Co(3,6-DBSQ)$_2$·2C$_7$H$_8$ (I). A solution of $Co_2(CO)_8$ (0.093 g, 0.272 mmol) in THF (10 mL) was added to a solution of 3,6-DBQ (0.239 g, 1.088 mmol) and pyrazino[2,3-f][1,10]phenanthroline (0.126 g, 0.544 mmol) in THF (10 mL). The dark brown reaction mixture was heated on a water bath until the evolution of CO ceased. The solvent was evaporated to dryness, the solid residue was dissolved in toluene, and the solution was filtered and left overnight in a hot water bath. The precipitated dark plates with a violet tint were separated on a filter, washed with cold *n*-hexane, and dried in vacuum. During drying, the crystals started to degrade; therefore, drying was not completed. Toluene was added to the isolated crystals for better preservation. The yield was ~0.085 g (21%).

For $C_{56}H_{64}N_4O_4Co$

Anal calcd., %	C, 73.42	H, 7.04	N, 6.12
Found, %	C, 73.68	H, 7.02	N, 6.08

IR (ν, cm^{-1}): 1578 w, 1545 m, 1506 w, 1456 s, 1446 s, 1406 m, 1352 m, 1344 m, 1277 w, 1211 w, 1121 w, 1081 w, 1027 w, 955 m, 820 w, 812 m, 735 m, 723 w, 653 w, 531 w, 488 w.

Synthesis of the complex (Bpyz)Co(3,6-DBSQ)$_2$·2C$_7$H$_8$ (II). A solution of 3,6-di-*tert*-butyl-*o*-benzoquinone (0.219 g, 0.994 mmol) in toluene (10 mL) was added to a solution of $Co_2(CO)_8$ (0.085 g, 0.248 mmol) in toluene (10 mL). The solution was heated on a water bath for 40 min, and a hot solution of bipyrazine (0.0785 g, 0.497 mmol) in toluene (5 mL) was added. The dark blue solution was left for slow cooling at room temperature. The small black-violet crystals were separated on a filter, washed with *n*-hexane, and dried in vacuum. The yield was 0.117 g (28%).

For $C_{50}H_{62}N_4O_4Co$

Anal. calcd., %	C, 71.32	H, 7.42	N, 6.65
Found, %	C, 71.67	H, 7.76	N, 6.89

IR (ν, cm^{-1}): 1602 w, 1543 m, 1467 s, 1445 s, 1407 m, 1354 m, 1343 m, 1308 w, 1274 w, 1201 w, 1155 m, 1106 w, 1060 w, 1034 m, 958 m, 934 w, 841 m, 829 w, 814 w, 736 m, 696 w, 654 m, 536 w, 511 w, 493 w, 455 m.

X-ray diffraction study of complexes **I** and **II** was carried out on Oxford Xcalibur Eos (complex **I**) and Bruker D8 Quest (complex **II**) automated diffractometers (graphite monochromator, MoK_α radiation, ω-scan mode, λ = 0.71073 Å). The experimental sets of reflection intensities were integrated using CrysAlisPro software [22] for complex **I** and SAINT software [23] for **II**. The SCALE3 ABSPACK algorithm (**I**) and the SADABS program [24] (**II**) were used to apply the absorption corrections. All structures were solved by the dual-space method using the SHELXT program package [25]. All non-hydrogen atoms of complexes **I** and **II** were refined by the full-matrix least-squares method on F_{hkl}^2 in the anisotropic approximation using the SHELXTL program package [26]. The hydrogen atoms were placed into the geometrically calculated positions and refined in the riding model ($U_{iso}(H) = 1.5U_{equiv}(C)$ for CH_3 groups and $U_{iso}(H) = 1.2U_{equiv}(C)$ for other groups). Compound **II** was refined as a two-component twin with a weight ratio of 0.59:0.41. The crystals of both **I** and **II** contain two toluene solvate molecules per molecule of the target complex. The toluene molecules in **I** are disordered over two positions and were refined using the AFIX 66, DFIX, and ISOR restraints. The main crystallographic data and X-ray experiment details for complexes **I** and **II** are summarized in Table 1.

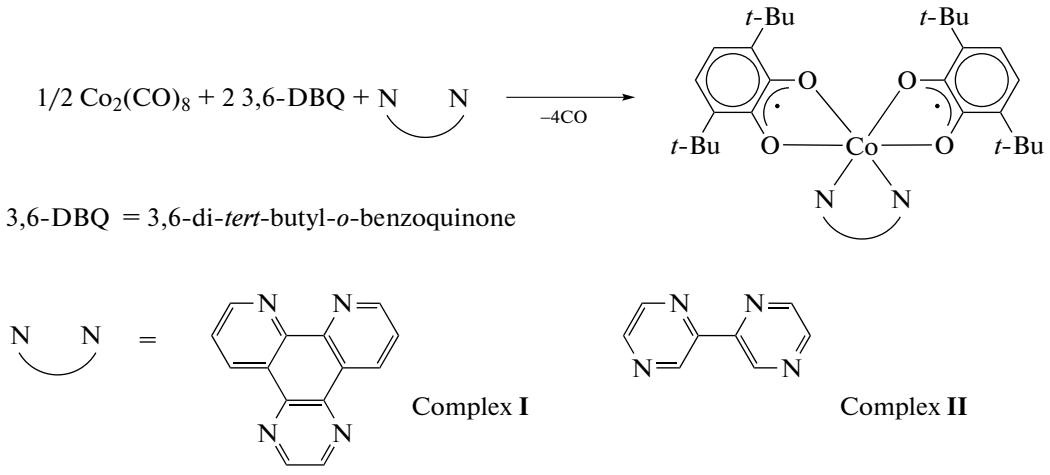
The structures are deposited with the Cambridge Crystallographic Data Centre (2165092 (**I**) and 2165093 (**II**); ccdc.cam.ac.uk/structures/).

RESULTS AND DISCUSSION

Complexes **I** and **II** were obtained by the reaction of dicobalt octacarbonyl with two equivalents of *o*-quinone and one equivalent of a neutral N-donor ligand (Scheme 1).

Table 1. Main crystallographic data and X-ray experiment details for complexes **I** and **II**

Parameter	Value	
	I	**II**
Molecular formula	$C_{56}H_{64}N_4O_4Co$	$C_{50}H_{62}N_4O_4Co$
M	916.04	841.96
Temperature, K	100(2)	100(2)
System	Orthorhombic	Orthorhombic
Space group	*Cmcm*	$P2_12_12_1$
Unit cell parameters:		
a, Å	22.628(3)	10.7481(6)
b, Å	11.4129(13)	18.8946(10)
c, Å	18.896(2)	22.6404(11)
V, Å^3	4879.9(11)	4597.8(4)
Z	4	4
ρ (calcd.), mg/m^3	1.247	1.216
μ, mm^{-1}	0.402	0.420
Crystal size, mm	0.61 × 0.23 × 0.18	0.63 × 0.49 × 0.29
F(000)	1948	1796
θ, deg	2.940–27.997	2.097–27.999
Number of reflections collected/unique	21561/3079	40605/11079
R_{int}	0.1118	0.0311
R_1, wR_2 ($I > 2\sigma(I)$)	0.0783 0.1688	0.0341 0.0752
R_1, wR_2 (for all data)	0.0870 0.1725	0.0430 0.0781
S	1.073	1.039
Absolute structural parameter		0.414(11)
Residual electron density (max/min), e/Å^3	0.741/–0.910	0.244/–0.392



Scheme 1.

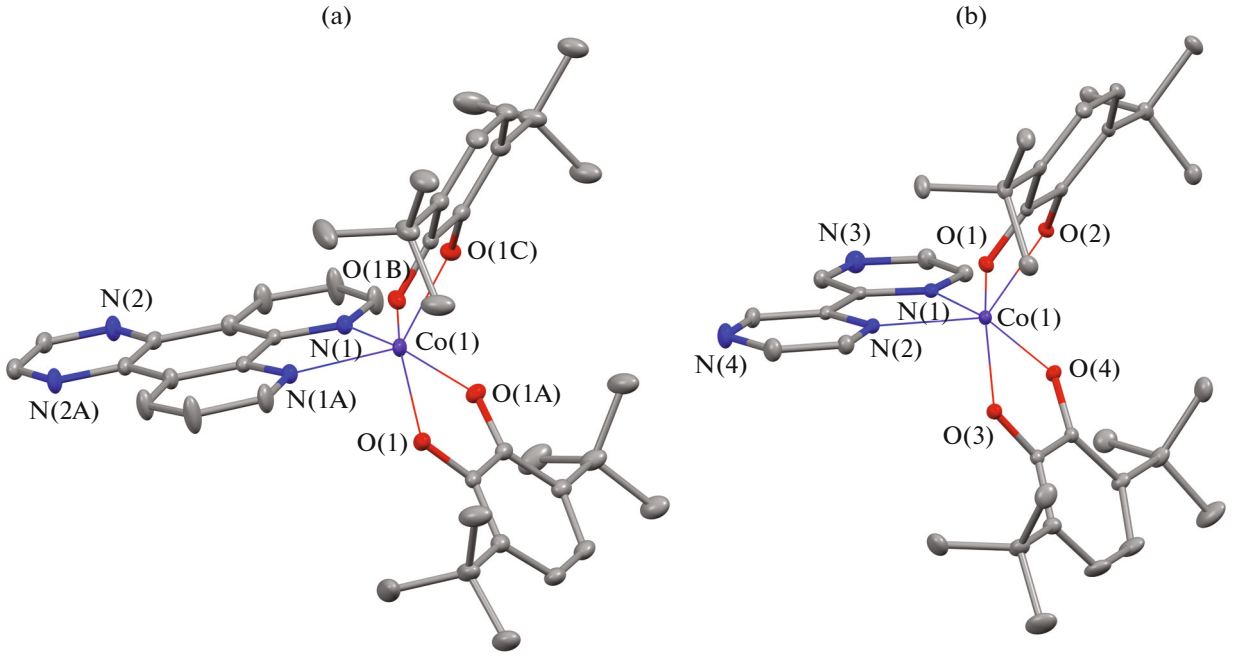


Fig. 1. Molecular structure of complexes (a) **I** and (b) **II**. Thermal ellipsoids are shown at 30% probability level. The hydrogen atoms are omitted for clarity. Symmetry codes used to generate equivalent atoms in complex **I**: (A) $x, y, -z + 1/2$; (B) $-x + 1, y, z$; (C) $-x + 1, y, -z + 1/2$.

The complexes were isolated in an individual state and characterized by single-crystal X-ray diffraction analysis, IR spectroscopy, and magnetochemistry.

According to X-ray diffraction data, compounds **I** and **II** are six-coordinate cobalt complexes. The independent part of the unit cell of **I** contains only 0.25 molecules of the complex. The Co^{2+} cations in both molecules are linked to two dioxolene ligands and one neutral chelating nitrogen ligand (Fig. 1). The coordination environment of cobalt in both complexes is a distorted trigonal prism. The key geometric characteristics of complexes **I** and **II** are close to one another (Table 2). The distributions of distances in the dioxolene ligands of **I** and **II** correspond to the radical anion form [27, 28] (the calculated charges of the ligands are $-1.03e$ (**I**) and -0.99 and $-0.89e$ (**II**)). The Co–O and Co–N distances in **I** and **II** (Table 2) are in good agreement with those in the previously reported six-coordinate Co(II) complexes [16, 29, 30].

The crystal structures of the complexes have common features. The molecules in the crystal form layers (Fig. 2).

The molecules of complex **I** are arranged in the crystal in such a way that four types of intermolecular O...H van der Waals contacts are formed (Fig. 3). The distances between the hydrogen and oxygen atoms are 2.605 Å. Also, each molecule of the complex additionally forms two intermolecular π...π contacts with toluene molecules. The dihedral angle between the planes of toluene and the pyrazino[2,3-f][1,10]phenanthroline ligand is 5.02°, and the distance between the centers of aromatic systems is 3.491 Å. These geometric characteristics attest to the presence of intermolecular π...π interactions [31]. Unlike complex **I**, complex **II** has no intermolecular O...H interactions in the crystal. However, the toluene planes in **II** are virtually parallel to the plane of the bipyrazine ligand. The corresponding dihedral angles are 1.19° and 4.44°, and the distances between the centroids composed of the aromatic systems of toluenes and the midpoint of the bipyrazine C(32)–C(33) bond are 3.464 and 3.566 Å. Thus, like in the case of complex **I**, each molecule of complex **II** forms two intermolecular π...π contacts with toluene molecules (Fig. 4).

The IR spectra of complexes reflect their ligand composition with allowance for the oxidation state. Thus, the presence of neutral N-donor ligands in the complexes can be derived from the IR spectra (Figs. 5, 6).

In addition, the structural similarity of **I** and **II** is reflected in the IR spectra, which are very similar. The presence of the semiquinone ligand in the complexes is evidenced by a characteristic set of bands in the 700–1600 cm^{-1} range. There are bands at 1545 (**I**) and 1542 cm^{-1} (**II**) corresponding to the C=C (arom.) stretching modes; the band at 1406 cm^{-1} corresponds to the bending (*sym* and *asym*) modes of the methyl groups (CH_3 group of *tert*-butyl); the bands at 1277 (**I**) and 1274 (**II**) cm^{-1} are due to the skeletal vibrations of the *tert*-butyl group; the bands at 955 and 958 cm^{-1} refer to the out-of-plane C–H bending modes of iso-

Table 2. Selected bond lengths (Å) and bond angles (deg) in complexes **I** and **II**

Bond	**I**, Å	**II**, Å
Co(1)–O(1)	2.056(2)	2.0414(16)
Co(1)–O(2)		2.0408(16)
Co(1)–O(3)		2.0325(16)
Co(1)–O(4)		2.0360(16)
Co(1)–N(1)	2.150(4)	2.1403(19)
Co(1)–N(2)		2.1536(19)
O(1)–C(1)	1.282(4)	1.283(3)
O(2)–C(2)		1.282(3)
C(1)C(1A*)/C(1)C(2)	1.475(7)	1.465(3)
O(3)–C(15)		1.283(3)
O(4)–C(16)		1.278(3)
		1.469(3)
Angle	**I**, deg	**II**, deg
OCo(1)O	77.35(13), 87.00(14), 136.78(13)	78.03(6), 78.35(6), 86.22(6), 89.18(6), 138.84(7), 139.18(7)
OCo(1)N	84.94(10), 132.27(8)	82.54(7)–85.42(7), 129.07(7)–133.83(7)
NCo(1)N	75.0(2)	73.97(7)

* Symmetry codes used to generate equivalent atoms in complex **I**: (A) $x, y, -z + 1/2$; (B) $-x + 1, y, z$; (C) $-x + 1, y, -z + 1/2$.

lated aromatic hydrogen atoms. For identification of the single-charged *o*-benzosemiquinone ligand, the intense stretching bands for the sesquialteral C–O bond, located at 1450 cm^{-1} (1456, 1446 cm^{-1} for **I** and 1458, 1446 cm^{-1} for **II**) are most important [15, 32]. Previously, it was shown that vibrations of the C=O double bond inherent in neutral *o*-benzoquinone occur at ~1650 cm^{-1}; and vibrations of the single C–O bonds belonging to the catecholate dianion are at ~1250 cm^{-1}. Some bands characteristic of neutral N-donor ligands are observed in the spectra of the complexes (Figs. 5, 6). Thus, the IR spectroscopy data confirm that complexes **I** and **II** are cobalt(II) compounds with *o*-benzosemiquinone ligands.

According to magnetochemical measurements, the effective magnetic moments of complexes virtually do not change with temperature and amount to 3.32–3.41 μ_B for **I** and 4.37–4.50 μ_B for **II** (Fig. 7). Only in

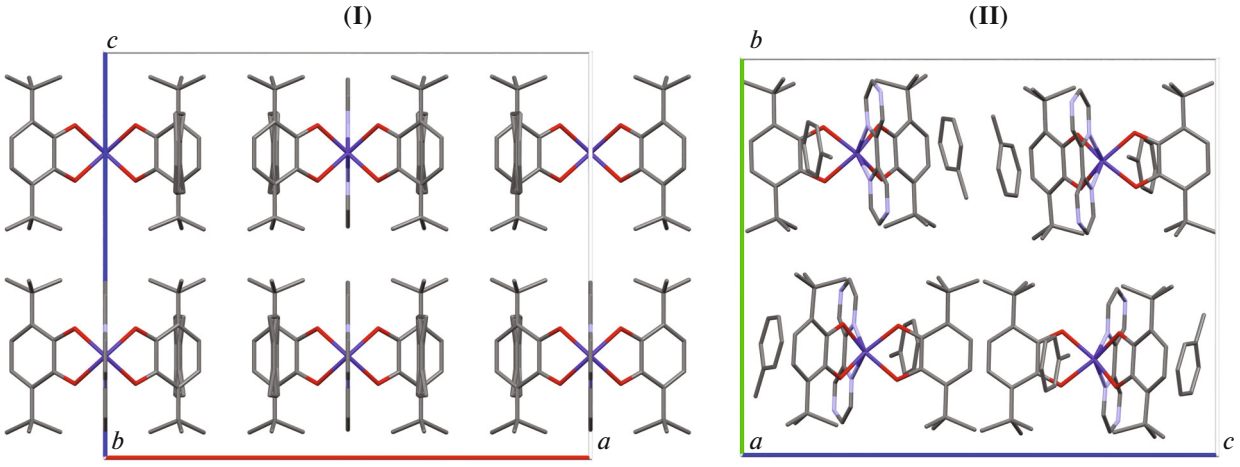


Fig. 2. Side view of the unit cell of complexes **I** and **II**.

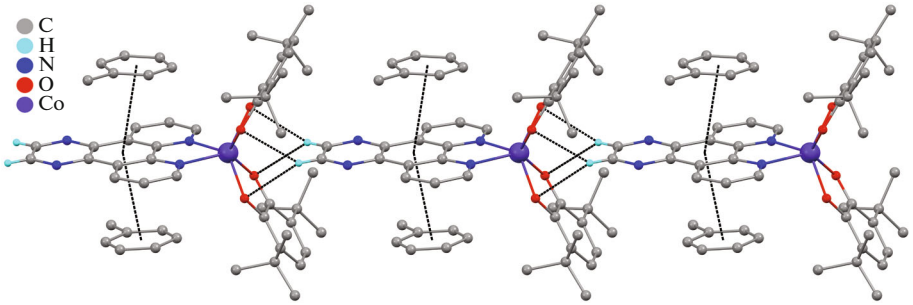


Fig. 3. Fragment of the crystal packing of complex **I**.

the low-temperature region at about 50 K for **I** and 20 K for **II**, the magnetic moment sharply decreases, apparently due to weak intermolecular antiferromagnetic interactions or zero-field splitting [33]. Furthermore, the decrease in μ_{eff} occurs at higher temperature for complex **I**, which is apparently attributable to the presence of more defined intermolecular contacts (see above). The magnetic moments observed for complex **II** in the temperature range of 20–300 K are close to the spin-only value for a system comprising a high-spin cobalt(II) ion (d^7, $S = 3/2$) and two radical anion ligands ($S = 1/2$) (4.58 μ_B). However, the μ_{eff} value for complex **I** is markedly lower. It is close to the value for a similar system containing a low-spin divalent cobalt ion (d^7, $S = 1/2$), which is 3.00 μ_B. Generally, the low-spin state of the metal is untypical of *o*-semiquinone cobalt(II) derivatives. Only one such example has been reported so far [34]. Unexpected is the fact that two isostructural complexes are characterized by different spin multiplicities of the central metal ion. This may be due to different π-acceptor abilities of pyrazino[2,3-f][1,10]phenanthroline and bipyrazine [35, 36].

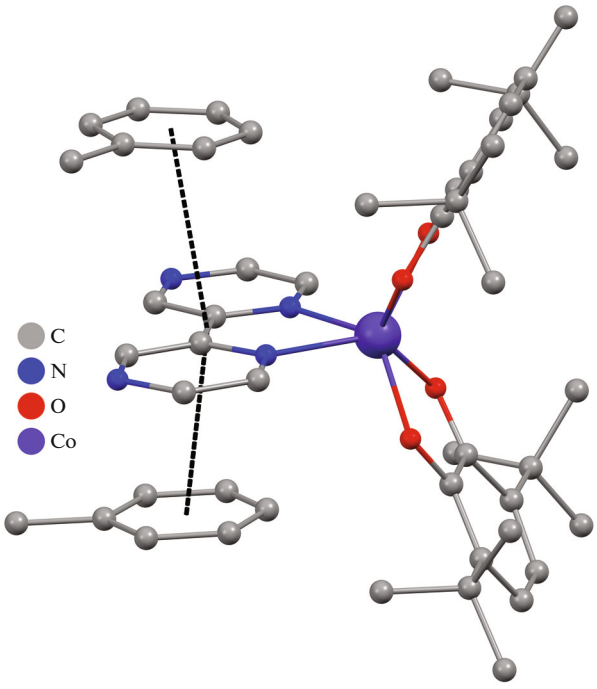


Fig. 4. Fragment of the crystal packing of complex **II**.

The energy parameters of the exchange interactions in complexes **I** and **II** were calculated using a model system of three exchange-coupled centers with the Hamiltonian:

$$H = -2J_{Co\text{-}SQ}(S_1S_2 + S_2S_3) - 2J_{SQ\text{-}SQ}S_1S_3 + DS_2^2 + g_2\mu_B S_2 H,$$

where $S_1 = S_3 = 1/2$ are the spins of *o*-semiquinone ligands, S_2 is the spin of the cobalt(II) ion ($S = 1/2$ (**I**) and $S = 3/2$ (**II**)), D is the axial zero-field splitting parameter, taking into account the intermolecular exchange interaction (zJ') and the temperature-independent paramagnetism (TIP) parameter. The experimental $\mu_{eff}(T)$ values were best reproduced at the following parameters: for complex **I**, $J_{Co\text{-}SQ} = -0.2$ cm^{-1}, $g_{\parallel Co} = 2.74$, $g_{\perp Co} = 2.91$, $D = 15.0$ cm^{-1}, $J_{SQ\text{-}SQ} = -9.0$ cm^{-1}, $zJ' = -1.3$ cm^{-1}, TIP = 0.0001; for complex **II**, $J_{Co\text{-}SQ} = -0.1$ cm^{-1}, $g_{\parallel Co} = 1.93$, $g_{\perp Co} = 2.00$, $D = 1.0$ cm^{-1}, $J_{SQ\text{-}SQ} = -2.1$ cm^{-1}, $zJ' = -0.1$ cm^{-1}, TIP = 0.0002. According to the calculated data, both complexes are characterized by very weak antiferromagnetic exchange between the metal and paramagnetic ligands (<–0.5 cm^{-1}), which is correlated with the data for analogous cobalt derivatives [15, 33]. The ligand–ligand interaction is also antiferromagnetic and has a relatively low energy. Similar exchange energies for the complexes are quite consistent with the similarity of their geometric structures. It should be noted that the energy of intermolecular exchange is higher in **I** than in **II**, which correlates with the X-ray diffraction data.

The trigonal-prismatic geometry of the coordination unit is unusual for complexes of this composition [37]. The complexes with this geometry do not exhibit redox isomerism, as they are high-spin Co(II) com-

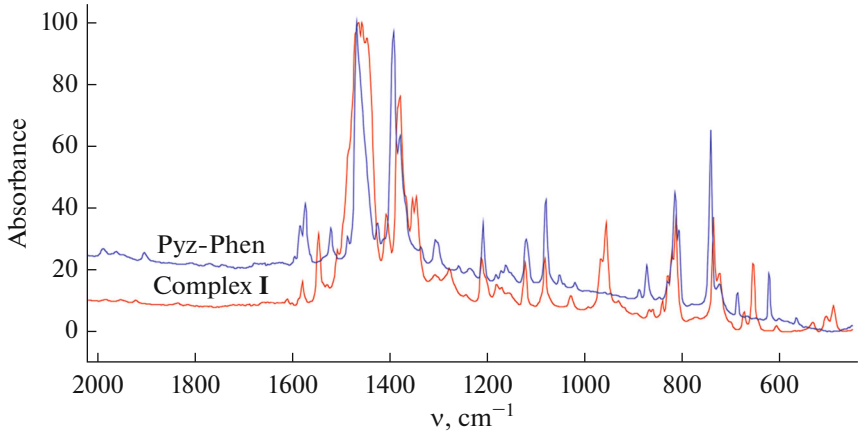


Fig. 5. IR spectra of pyrazino-phenanthroline and its complex (**I**) (mineral oil).

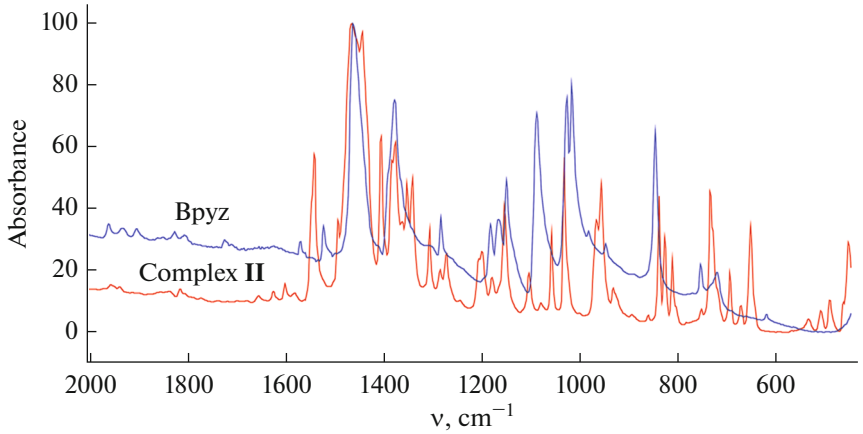


Fig. 6. IR spectra of bipyrazine and its complex (**II**) (mineral oil).

pounds. In particular, a complex containing 5-nitro-1,10-phenanthroline as the N-donor ligand was reported [38]. The magnetic and spectral properties of this compound indicated that it was a bis-*o*-semiquinone complex of divalent high-spin cobalt. The effective magnetic moment for a crystalline sample of this complex regularly decreased from 4.6 μ_B at 300 K to ~4.0 μ_B at 50 K. In addition, a complex with pyridyl-substituted benzoxazole in which the Co(II) ion in the trigonal-prismatic environment occurred in the high-spin state was described [16]. The magnetic properties of both above complexes were generally similar to those of complex **II**.

Thus, in this study, we synthesized new cobalt bis-*o*-semiquinonato complexes containing pyrazino[2,3-f][1,10]phenanthroline and bipyrazine as neutral ligands. According to X-ray diffraction studies of the molecular structures, the complexes have a trigonal-prismatic geometry of the coordination sphere. Magnetochemical measurements showed that the complex

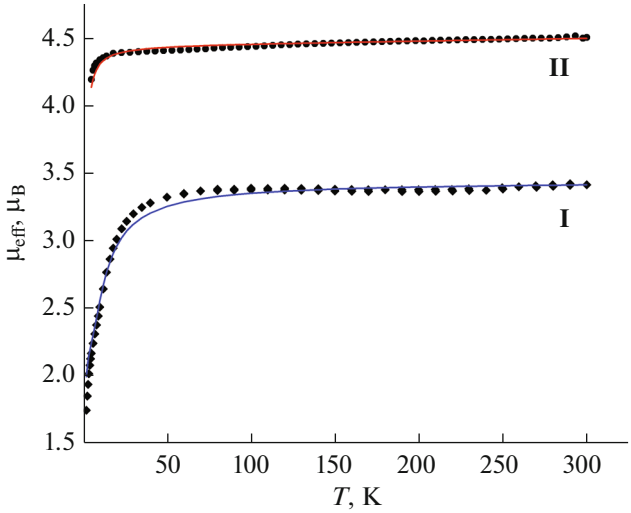


Fig. 7. Temperature dependences of the effective magnetic moment of complexes **I** and **II**.

with pyrazino[2,3-f][1,10]phenanthroline is a derivative of low-spin divalent cobalt, whereas the bipyrazine structural analogue is a high-spin cobalt(II) compound.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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