# Synthesis and Structure of Polycrystalline Adducts of Co(II) Azomethine Complexes with Redox-Active 2,4,6,8-Tetrakis-(*tert*-Butyl)Phenoxazin-1-one

E. P. Ivakhnenko<sup>*a*, \*</sup>, Yu. V. Koshchienko<sup>*a*</sup>, P. A. Knyazev<sup>*a*</sup>, V. B. Nalbandyan<sup>*b*</sup>, K. A. Lyssenko<sup>*c*</sup>, I. V. Ananyev<sup>*c*</sup>, A. S. Bogomyakov<sup>*d*</sup>, and V. I. Minkin<sup>*a*</sup>

<sup>a</sup>Research Institute of Physical and Organic Chemistry, Southern Federal University, Rostov-on-Don, Russia <sup>b</sup>Southern Federal University, Rostov-on-Don, Russia

<sup>c</sup>Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, ul. Vavilova 28, Moscow, 119991 Russia

<sup>d</sup>International Tomography Center, Siberian Branch, Russian Academy of Sciences, Novosibirsk, Russia

\*e-mail: e.ivakhnenko@ipoc.sfedu.ru

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Abstract—The six-coordinate cobalt complexes,  $C_{57}H_{63,50}N_{4,50}O_4Co$  (IIa),  $C_{60}H_{69}N_5O_4Co$  (IIb),  $C_{58}H_{67}N_3O_8Co$  (IIc),  $C_{56}H_{61}N_5O_{10}Co$  (IId),  $C_{56}H_{63}N_3O_6Co$  (IIe),  $C_{58}H_{66}N_4O_6Co$  (IIf), and  $C_{58}H_{63}N_7O_8Co$  (IIg), adducts of high-spin tetrahedral Co(II) bis(salicylaldiminates) ( $C_{29}H_{24,50}N_{3,50}O_2Co$  (Ia),  $C_{32}H_{30}N_4O_2Co$  (Ib),  $C_{30}H_{28}N_2O_6Co$  (Ic),  $C_{28}H_{22}N_4O_8Co$  (Id),  $C_{28}H_{24}N_2O_4Co$  (Ie),  $C_{30}H_{27}N_3O_4Co$  (If), and  $C_{30}H_{24}N_6O_6Co$  (Ig)) and redox-active 2,4,6,8-tetrakis(*tert*-butyl)phenoxazin-1-one (L), were synthesized and studied for structure and magnetic properties. Complexes IIa—IIg have octahedral structure (CIF files CCDC nos. 1403920 (IIf), 1403922 (IIg)) and exist in the ground low-spin state (*ls*-Co<sup>III</sup>-SQ), which arises upon intramolecular single-electron redox process in the ligand—metal system. The presence of substituents of different nature in the azomethine ligands of IIa—IIg does not induce any significant changes in their magnetic properties.

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## INTRODUCTION

Molecular magnetic materials that exist in the crystalline state in two or several thermodynamically stable forms are of considerable interest as molecular switches with magnetic response and high-capacity molecular memory. Among the most promising objects for the design of these materials are coordination compounds of transition metals containing electro-active (so-called non-innocent) ligands that can have nearly degenerate electronic states arising upon low-barrier thermo- or photo-stimulated electron transfer between the ligand and metal ion [1-6]. These states (valence tautomers) have different physical properties [7-10] and, therefore, compounds and dynamic materials based on them are of considerable interest for diverse applications in technology [11, 12].

Previously, we demonstrated the possibility of preparing potentially valence-tautomeric mixed-

ligand six-coordinate cobalt complexes,  $C_{57}H_{63} {}_{50}N_{4} {}_{50}O_4Co$  (IIa) and  $C_{60}H_{69}N_5O_4Co$  (IIb), by the reaction of the redox-active 2,4,6,8tetrakis(*tert*-butyl)phenoxazin-1-one ( $C_{28}H_{39}NO_2$ , L) with high-spin tetrahedral Co(II) bis(salicylaldiminates) (I) on heating in toluene [13, 14]. This work deals with the synthesis of new complexes:  $C_{58}H_{67}N_{3}O_{8}Co$ (IIc),  $C_{56}H_{61}N_5O_{10}Co$ (IId), $C_{56}H_{63}N_3O_6Co$  (IIe),  $C_{58}H_{66}N_4O_6Co$  (IIf), and  $C_{58}H_{63}N_7O_8Co$  (IIg) (Scheme 1), the spatial and electronic structures of these complexes, and the effect of substituents in the azomethine moiety of cobalt complexes on the state and redox isomerism processes. A new convenient solid-phase synthesis of complexes II is presented and the magnetic properties and the EPR spectra of the crystalline powders of the resulting adducts are studied.



Ia, b, c, d, e, f, g

L



**d**:  $R = OCH_3$ ,  $R_1 = NO_2$ 

Scheme 1.

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### **EXPERIMENTAL**

The IR spectra of the polycrystalline samples were recorded on a Varian Excalibur 3100 FRT-IR spectrometer. Powder X-ray diffraction analysis was performed on an ARL X'tra powder diffractometer (Cu $K_{\alpha}$ radiation) using a semiconductor detector to eliminate the fluorescent radiation of cobalt. The EPR spectra were recorded on an EMX Plus 10/12 Bruker X-band spectrometer. The hyperfine splitting (HFS) constants for the EPR spectra were calculated using the Win EPR SimFonia Bruker program package and the magnetic measurements for polycrystalline complexes

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were performed using a Quantum Design MPMSXL-SQUID magnetometer in the 2-300 K range.

Cobalt bis[2-(phenyliminomethyl)phenolate] **Ia** and cobalt bis[2-(*p*-tolyliminomethyl)phenolate] **Ib** were prepared by a reported procedure [15].

General procedure for the preparation of complexes IIa–IIg. A solution of phenoxazinone L (0.3 mmol) in benzene (5 mL) was added to a solution of complex I (0.3 mmol) in benzene (5 mL), and the resulting solution was refluxed for 30 min. The solvent was evaporated, and the residue was dried in air. The complexes thus obtained were recrystallized from acetonitrile.

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Parameter	value			
Tarameter	IIf	IIg		
Molecular formula	C <sub>58</sub> H <sub>66</sub> CoN <sub>4</sub> O <sub>6</sub>	C <sub>58</sub> H <sub>63</sub> CoN <sub>7</sub> O <sub>8</sub>		
M	975.59	1045.08		
<i>Т</i> , К	100	100		
Crystal system	Triclinic	Triclinic		
Space group	$P\overline{1}$	$P\overline{1}$		
Ζ	2	2		
<i>a</i> , Å	14.3796(16)	12.8556(16)		
<i>b</i> , Å	14.7410(16)	12.9725(16)		
<i>c</i> , Å	14.9120(17)	16.013(2)		
α, deg	61.7910(18)	86.366(2)		
β deg	69.3090(19)	83.232(2)		
γ, deg	68.603(2)	80.839(2)		
$V, Å^3$	2529.7(5)	2615.3(6)		
$\rho$ (calcd.), g cm <sup>-3</sup>	1.281	1.327		
$\mu$ , cm <sup>-1</sup>	3.95	3.91		
<i>F</i> (000)	1037	1102		
$2\theta_{\text{max}}$ , deg	50	54		
Number of measured reflections	32195	24674		
Number of unique reflections	14805	11348		
Number of reflections with $I > 2\theta(I)$	10703	6278		
Number of refined parameters	656	681		
$R_1, wR_2 (I > 2\theta(I))$	0.0490, 0.1183	0.0514, 0.0993		
$R_1$ , $wR_2$ (all data)	0.0759, 0.1314	0.1211, 0.1208		
GOOF	1.015	0.914		
$\Delta \rho_{\rm max} / \rho_{\rm min}, e {\rm \AA}^{-3}$	0.937/-0.664	0.574/-0.892		

 Table 1. Selected crystallographic data and refinement details for structures IIf and IIg

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Complexes **IId** and **IIg** were synthesized in DMF. The crystals of complexes **IIf** and **IIg** suitable for X-ray diffraction were prepared by slow evaporation of acetonitrile solutions. Complexes **IIa** and **IIb** were synthesized by reported procedures [13, 14].

Complex IIc ( $R = R^1 = OCH_3$ ): crystals, brown needles. Yield 94%. mp = 165–166°C.

For	$C_{58}$	H <sub>67</sub> N	N <sub>3</sub> C	<sub>8</sub> Co
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anal. calcd., %:	C, 70.15;	H, 6.80;	N, 4.23.
Found, %:	C, 70.08;	Н, 6.94;	N 4.28.

Complex IId ( $R = OCH_3$ ,  $R^1 = NO_2$ ): crystals, brown needles. Yield 68%. mp = 190–191°C.

For C<sub>56</sub>H<sub>61</sub>N<sub>5</sub>O<sub>10</sub>Co

anal. calcd., %:	C, 65.74;	H, 6.01;	N, 6.93.
Found, %:	C, 65.63;	H, 6.15;	N, 6.93.

Complex IIe (R = H,  $R^1 = OCH_3$ ): crystals, brown needles. Yield 91%. mp= 133–134°C.

For C<sub>56</sub>H<sub>63</sub>N<sub>3</sub>O<sub>6</sub>Co

anal. calcd., %:	C, 72.09;	H, 6.81;	N, 4.50.
Found, %:	C, 72.01;	H, 6.89;	N, 4.58.

Complex IIf ( $R = OCH_3$ ,  $R^1 = H$ ): crystals, brown needles. Yield 86%. mp = 133–134°C.

For	C55	H	6N	0	Co
		<u> </u>	0 - 4	(	)

anal. calcd., %:	C, 71.51;	H, 6.83;	N, 5.75.
Found, %:	C, 71.98;	Н, 6.90;	N, 5.67.

Complex IIg ( $R = NO_2$ ,  $R^1 = H$ ): crystals, brown needles. Yield 95%. mp = 138–139°C.

For C58H63N7O8Co

anal. calcd., %:	C, 66.66;	Н, 6.08;	N, 9.38.
Found, %:	C, 66.70;	H, 6.05;	N, 9.37.

Synthesis of adducts IIa, IIb, and IIg by the solidphase reaction of Co(II) bis(salicylaldiminates) with 2,4,6,8-tetra(*tert*-butyl)phenoxazin-1-one. Compound L (84 mg, 0.2 mmol) and compound Ia (0.2 mmol) were placed in agate mortar and thoroughly ground with an agate pestle to give a uniform mixture. Complex IIa was a dark brown crystalline powder (mp = 139–141°C; yield 98%). Complexes IIb and IIg were prepared by a similar procedure (mp = 134-136°C and 138-139°C, respectively). The completeness of the reaction was checked by IR spectroscopy.

**X-ray diffraction study** of **IIf** and **IIg** was carried out on a Bruker APEX II Duo diffractometer (Mo $K_{\alpha}$  radiation, graphite monochromator,  $\omega$ -scan mode). The structures were solved by the direct method and refined by the least squares method in the anisotropic approximation on  $F_{hkl}^2$ . The hydrogen atoms were located from the Fourier difference maps and refined in the isotropic approximation in the riding model. The structures were solved and refined using the SHELXTL PLUS program package [16]. Selected crystallographic data and refinement parameters for **IIf** and **IIg** are summarized in Table 1 and the bond lengths and angles are in Table 2. Full X-ray diffraction data for **IIf** and **IIg** are available at the Cambridge

David	<i>d</i> , Å		
Bond	IIf	IIg	
Co(1)-O(1)	1.8780(12)	1.890(2)	
Co(1)-O(3)	1.8737(12)	1.871(2)	
Co(1)-O(6)	1.8920(12)	1.873(2)	
Co(1)-N(1)	1.9772(14)	1.967(2)	
Co(1)–N(2)	1.9410(15)	1.931(2)	
Co(1)-N(4)	1.9344(15)	1.930(2)	
Angle	ω,	deg	
O(3) Co(1) O(6)	87.83(5)	89.22(9)	
O(3) Co(1) O(1)	86.93(5)	91.53(9)	
O(1) Co(1) O(6)	171.39(5)	174.00(8)	
O(3) Co(1) N(1)	169.61(6)	173.58(10)	
O(6) Co(1) N(1)	101.80(6)	95.64(9)	
O(1) Co(1) N(1)	83.95(5)	84.07(9)	
O(3) Co(1) N(4)	86.33(6)	82.38(9)	
O(6) Co(1) N(4)	93.82(6)	94.13(9)	
O(1) Co(1) N(4)	92.66(6)	91.87(9)	
N(4) Co(1) N(1)	89.11(6)	93.06(9)	
O(3) Co(1) N(2)	94.47(6)	92.92(9)	
O(6) Co(1) N(2)	85.11(6)	88.55(9)	
O(1) Co(1) N(2)	88.49(6)	85.47(9)	
N(2) Co(1) N(1)	90.28(6)	91.40(9)	
N(4) Co(1) N(2)	178.64(6)	174.54(10)	

Table 2. Selected bond lengths (Å) and bond angles (deg) in complexes IIf and IIg

Crystallographic Data Centre (CCDC no. 1403920 and no. 1403922, respectively; www.ccdc.ac.uk).

## **RESULTS AND DISCUSSION**

All of the obtained adducts II are crystalline compounds stable in the presence of air oxygen (the crystals are dark brown-colored) and can be recrystallized from solvents of different polarity. As was shown [13, 14], mixed-ligand cobalt complexes II in their ground low-spin electronic state *ls*-Co<sup>III</sup>-SQ are formed upon barierless electron transfer, which occurs either via direct coordination of the redox-active ligand to the Co(II) complex I or with inclusion of the intermediate high-spin form of the adduct II' *hs*-Co<sup>II</sup>-Q (Scheme 1). The molecular structure of complexes IIf and IIg containing electron-donating (OCH<sub>3</sub>) and electron-withdrawing (NO<sub>2</sub>) substituents in the azomethine moiety was studied by X-ray diffraction (Fig. 1).

The X-ray diffraction study of cobalt complexes **IIf** and **IIg** demonstrated that parameters of the metal coordination polyhedron (distorted octahedron) in found previously for low-spin compounds IIa and IIb with the phenoxazinone ligand in the semiquinolate (SQ) form [14]. The Co-O and Co-N bond lengths of the phenoxazin-1-one moiety in these compounds are 1.871(2) - 1.8920(12) and 1.930(2) - 1.9772(14) Å, respectively, and almost do not differ from the Co-O and Co-N bond lengths in IIa and IIb (1.873(2)-1.893(2) and 1.950(3) - 1.976(3) Å, respectively). The Co-O and Co-N bond lengths are somewhat shorter than those expected in a coordination unit for quinoid ligands containing oxygen and nitrogen [17]. This indicates that the Co<sup>3+</sup> central ion exists in the lowspin form. In compounds **IIf** and **IIg**, the O(1)-C(1)bond lengths (1.311(2)-1.320(3) Å) differ only within the error from the corresponding values in IIa and IIb (1.314(4)-1.312(4) Å) [14] and are consistent with the average lengths of these bonds for the ligands existing in the iminosemiquinone form [18]. The most pronounced differences in the geometry of complexes IIf and IIg are found for the rotation angle of the aryl substituent. This angle is  $49^{\circ}-68^{\circ}$  in **IIf** and  $75^{\circ}-84^{\circ}$  in IIg. Despite the fact that the intramolecular C…C and C...N contacts of the aryl substituents with phenoxazin-1-one ligand are shortened in all structures, their values are considerably different, which indirectly points to their sterically forced nature. A distinctive feature of the nitro-substituted structure IIg is a substantial distortion of the phenoxazine ring, resulting in the boat conformation for the central 6-membered ring with oxygen and nitrogen atoms deviating from the plane by  $\sim 0.13$  Å and folding angle between the carbon rings and the central ring being  $\sim 16^{\circ}$ . In all other complexes, the phenoxazine ring is actually planar. The comparison of X-ray diffraction data for IIf and **IIg** with the previously described **IIa** and **IIb** [14] indicates that the substituents R in the series H,  $CH_3$ , OCH<sub>3</sub>, NO<sub>2</sub> do not influence the key structural characteristics of the coordination polyhedron and, hence, the state of the central cobalt ion.

these complexes virtually do not differ from those

It was found that the reaction of redox active phenoxazinone L with I, resulting in the formation of adducts II, can take place without a solvent in the solid phase. A nearly quantitative yield of the target product is attained by thorough grinding of equimolar amounts of the reactants I and L in an agate mortar. The described reaction is clearly detected by IR spectroscopy. Figure 2 presents the absorption bands of the reactants in the stretching regions of the C=O (phenoxazinone L) and C=N (complex I) groups responsible for the formation of the coordination unit of adducts II. The coordination accompanied by the formation of radical ion adduct II leads to disappearance of the absorption band of the phenoxazinone ring carbonyl group (1640 cm<sup>-1</sup>) and increase in the intensity of the azomethine absorption band (C=N) in mixedligand complex II. The solid-phase reaction is accompanied by color change to give the dark brown crystals





Fig. 1. Molecular structure of complexes (a) IIf and (b) IIg with atoms represented by probability ellipsoids of displacements (p = (0.5). The hydrogen atoms are omitted.

of the adduct and a sharp change in the volume of the crystalline mass, due to higher density of the resulting complex. It is noteworthy that obtaining the adducts in a quantitative yield is faced with procedural difficulties associated with the process of grinding. Since friction takes place on the crystal surface, the inner part of the



Fig. 2. Fragments of the IR spectra: (1) Ia, (2) L, (3) adduct IIa, (4) adduct IIa obtained in toluene as the solvent and recrystallized from acetonitrile.

crystals remains unaffected. In other words, the amounts of unreacted compounds I and L are related to the sizes of reacting particles. This assumption is consistent with the data of IR spectroscopy and powder X-ray diffraction. The presence of trace amount of the absorption band characteristic of the phenoxazi-



Fig. 3. Power X-ray diffraction pattern of (1) L, (2) Ia, (3) adduct IIa.

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none ring carbonyl ( $1640 \text{ cm}^{-1}$ ) can be recorded even after long-term grinding of the reactants (Fig. 2).

The X-ray powder diffraction pattern of the grinding product corresponds to a new phase and does no show reflections of the initial phases to within the instrument sensitivity (2%). However, it does not coincide with the X-ray diffraction pattern calculated from the single crystal data, because the solid-phase synthesis product does not contain solvent molecules (Fig. 3).

The ability of phenoxazinone L to undergo reversible single-electron redox processes to give stable radical ions (PhenoxSQ) is well known [19, 20]. High stability of this radical anion is ensured by unpaired electron shielding by the bulky *tert*-butyl groups and its high degree of delocalization in the tricyclic phenoxazinone system, which is reflected in the HFS constants ( $a^{N} = 6.32$ ,  $a_{1}^{H} = 3.12$ ,  $a_{2}^{H} = 3.43$ ,  $a_{3}^{H} = 3.39$  G). The HFS constants in the obtained complexes **IIc–IIg** were studied by EPR, and the radical ion structure was established for the complexes. The EPR spectra of a toluene solution of adducts **IIc–IIg** in the X-range at 300 K represent a group consisting of 10 HFS lines, which result from interaction of the unpaired electron

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Fig. 4. EPR spectra of cobalt complex IIc: (a) in toluene ((1) experimental and (2) simulated spectrum); (b) polycrystalline sample at 300 K.

with nitrogen (S = 3/2) and cobalt (S = 7/2) nuclei with approximately equal HFS constants (Fig. 4a). The simulation in terms of this model using the isotropic splitting constants  $a^{\rm N} = 9.45$ ,  $a^{\rm Co} = 13.45$  G (g = 2.001) gives a satisfactory agreement between the calculated and experimental spectra. Similar values of HFS constants found for IIa-IIg indicate that substituents in the azomethine moiety have a minor effect on the delocalization of the unpaired electron. The higher value  $a^{N}$  = 9.45 G for cobalt complexes IIa–IIg compared with similar constant in the radical ion potassium salt PhenoxSO ( $a^{N} = 6.32$  G) is attributable to the presence of strong coordination bonds in the radical ion complexes IIa-IIg. The EPR spectrum of the crystalline powders of IIc-IIg (X-range, 300 K) is the exchange-narrowed lines of  $\sim 30$  G width and g-factor of ~2.00 (Fig. 4b).

The dimeric structure of crystalline **IIb** and different contents of acetonitrile solvent molecules (the crystal of **IIb**, unlike unsubstituted **IIa**, contains two independent molecules of the cobalt complex and four acetonitrile solvent molecules [14]) were found not to induce significant differences in the magnetic properties.

Figure 5 shows the dependence of the effective magnetic moment ( $\mu_{eff}$ ) on temperature for complexes **IIa** and **IIb**. The decrease in the temperature in the 300–20 K range changes  $\mu_{eff}$  insignificantly from 2.3 to 1.8  $\mu_B$ , which implies a paramagnetic state with one unpaired electron with the spin S = 1/2, in this particular case, a low-spin radical ion cobalt complex **IIa**. The curve for  $\mu_{eff}$  vs. temperature in the range of 300–100 K for **IIb** (Fig. 5b) shows a minor hysteresis. The data of magnetic susceptibility measurements of **IIa**–**IIg** attest to a low-spin electronic state. In the solid phase, the  $\mu_{eff}$  value for such complexes is 1.85  $\mu_B$ , being almost unchanged in the range of 77–298 K.

It turned out that the presence of different substituents in complexes **IIa–IIg** does not significantly change their magnetic behavior.

The proposed approach to the construction of octahedral mixed-ligand Co(II) complexes capable of intramolecular redox isomerism (valence tautomer-



Fig. 5. Temperature dependence of  $\mu_{eff}$  for cobalt complexes: (a) IIa, (b) IIb.

ism) opens up the possibility of using a broad range of tetrahedral metal chelates with different structures of the coordination unit for the design of systems with magnetically controlled properties. The high cyclability of the redox isomerization in complexes of this type is provided by the intramolecular electron transfer mechanism requiring no significant structural rearrangement of ligands.

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