

## SYNTHESIS AND STRUCTURE OF THE Co(III) COMPLEX WITH 2-[(E)-(2-(ETHYLAMINO)-5- NITROPHENYLIMINO)METHYL)-4- (PHENYLDIAZENYL)]-PHENOL

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A new anionic Co(III) complex with 2-[(E)-(2-(ethylamino)-5-nitrophenylimino)methyl)-4-(phenyl diazenyl)]-phenol is prepared and characterized by <sup>1</sup>H NMR, IR spectroscopy, and elemental analysis. The crystal structure of the complex is studied by single crystal X-ray diffraction (XRD). From the single crystal XRD data it is found that in the complex, two structurally non-equivalent molecules of the doubly deprotonated ligand are tridentate-chelated by the central Co atom. The cobalt(III) ion is in a slightly distorted octahedral environment of four N and two O atoms.

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### INTRODUCTION

Azo compounds of aromatic and heterocyclic series are important and widely studied compounds [1-6]. Based on them, metal chelates with five- and six-membered metallacycles were obtained [7, 8]. Of particular interest are metallocomplexes of azomethine ligands in which the azo group is not at the coordination site [9, 10]. These metallocomplexes are capable of light-induced *E/Z* isomerization with the properties of molecular switches and are promising objects for studying the crossover effect [11, 12].

As opposed to a huge number of neutral azomethine coordination compounds (chelates of the  $ML_n$  composition) and numerous cationic complexes ( $ML_n^+A^-$ ), anionic coordination structures in the series of metallocomplexes of Schiff's bases are few [13-15].

In continuation of the research reported in [11, 16], in this work, a new anionic Co(III) complex with 2-[(E)-(2-(ethylamino)-5-nitrophenylimino)methyl)-4-(phenyldiazenyl)]-phenol was prepared, and its structure was studied by spectroscopic methods and single crystal X-ray diffraction (XRD).

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## EXPERIMENTAL

The compounds were synthesized using commercially available solvents, 98% cobalt(II) triflate hexahydrate CAS No. 73475-71-5, triethylamine CAS No. 121-44-8, 2-ethylamino-5-nitroaniline (1-N-ethyl-4-nitrobenzene-1,2-diamine) CAS:66668-41-5; 2-hydroxy-5-phenylazobenzaldehyde was obtained following the procedure [17].

The elemental analysis was carried out on an automated EuroEA-3000 C,H,N analyzer (EuroVektor). The IR spectrum was recorded on a Varian 3100-FTIR Excalibur by the frustrated total internal reflection method. The  $^1\text{H}$  NMR spectrum was measured on a Varian Unity-300 (300 MHz) instrument in the internal stabilization mode of the  $^2\text{H}$  polar resonance line in DMSO- $d_6$ .

**2-[(E)-(2-(ethylamino)-5-nitrophenylimino)methyl]-4-(phenyldiazenyl)-phenol ( $\text{H}_2\text{L}$ )** was obtained from 2-hydroxy-5-phenylazobenzaldehyde and 2-ethylamino-5-nitroaniline following the procedure [16].

**$[\text{Co}^{\text{III}}\text{L}_2]^- \cdot \text{HNet}_3^+ \cdot 1.5\text{H}_2\text{O} \cdot 3\text{MeCN}$  complex (**1**)**. A solution of azomethine  $\text{H}_2\text{L}$  (0.078 g, 0.0002 mol) in 15 mL of a 1:1 methylene chloride - acetonitrile mixture was added with triethylamine (0.020 g, 0.0002 mol) and a solution of cobalt triflate hexahydrate ( $\text{Co}(\text{CF}_3\text{SO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) (0.093 g, 0.0002 mol) in 7 mL of ethanol. The resulting mixture was boiled for 2 h with vigorous stirring and then cooled to room temperature. The polycrystalline precipitate formed was filtered off and washed with hot alcohol. Yield: 86%. Brown crystals.  $T_{\text{melt}} > 250$  °C.

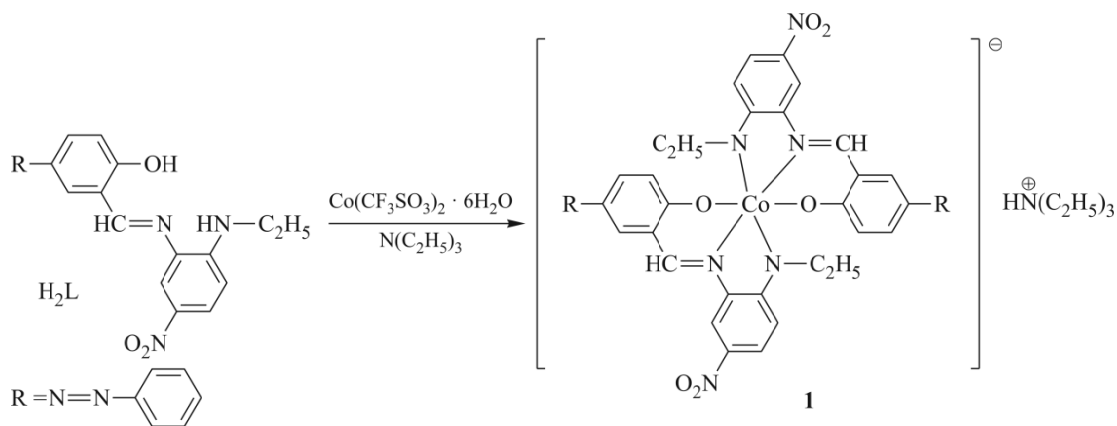
Found (%): C 60.01, H 5.82, N 18.04. For  $\text{C}_{54}\text{H}_{61.5}\text{CoN}_{14}\text{O}_{7.25}$  calculated (%): C 59.96, H 5.74, N 18.13. IR spectrum, ( $\nu$ ,  $\text{cm}^{-1}$ ): 1598 s (C = N), 1328 w (Ph-O).  $^1\text{H}$  NMR (DMSO- $d_6$ ),  $\delta$  (ppm): 0.52 (6H, t,  $J = 6.6$  Hz,  $\text{NCH}_2\text{CH}_3$ ), 1.62 (9H, t,  $J = 7.2$  Hz,  $\text{CH}_3$ -triethylamine), 2.48-2.51 (4H, m,  $\text{NCH}_2\text{CH}_3$ ), 3.08 (6H, m,  $\text{CH}_2$ -triethylamine), 6.48 (2H, d,  $J = 9.3$  Hz,  $\text{C}_{\text{Ar-H}}$ ), 6.60 (2H, d,  $J = 9.3$  Hz,  $\text{C}_{\text{Ar-H}}$ ), 7.43-7.75 (8H, m,  $\text{C}_{\text{Ar-H}}$ ), 7.76-7.80 (6H, m,  $\text{C}_{\text{Ar-H}}$ ), 8.47 (2H, d,  $J = 2.7$  Hz,  $\text{C}_{\text{Ar-H}}$ ), 9.01 (2H, d,  $J = 2.4$  Hz,  $\text{C}_{\text{Ar-H}}$ ), 9.48 (2H, s, CH = N).

Single crystals precipitated from the mother liquor are suitable for XRD.

**Single crystal XRD** of complex **1** was performed on a Bruker APEX II diffractometer (CCD detector,  $\text{MoK}\alpha$ ,  $\lambda = 0.71073$  Å, graphite monochromator) [18]. A semi-empirical absorption correction was applied [19]. The structure was solved by direct and Fourier methods and refined in the full-matrix anisotropic approximation for all non-hydrogen atoms. Hydrogen atoms at carbon atoms of organic ligands were generated geometrically and refined with a riding model. The calculations were performed with the SHELXL 2018/3 software [20] using the Olex2 program package [21]. The structure was refined using the standard ISOR, DFIX, DELU, RIGU restrictions. The crystallographic parameters and details of the structure refinement of complex **1** are as follows:  $\text{C}_{54}\text{H}_{61.5}\text{CoN}_{14}\text{O}_{7.25}$ ,  $M = 1081.60$ , crystal size  $0.35 \times 0.35 \times 0.20$  mm,  $T = 100(2)$  K, triclinic crystal system with the space group  $P\bar{1}$ ,  $a = 12.039(2)$  Å,  $b = 13.613(3)$  Å,  $c = 18.198(4)$  Å,  $\alpha = 86.998(3)^\circ$ ,  $\beta = 71.632(3)^\circ$ ,  $\gamma = 80.093(3)^\circ$ ,  $V = 2788.3(10)$  Å $^3$ ,  $Z = 2$ ,  $\rho = 1.288$  g/cm $^3$ ,  $\mu = 0.372$  mm $^{-1}$ ,  $\theta = 1.52$ - $31.39^\circ$ ,  $-17 \leq h \leq 17$ ,  $-19 \leq k \leq 19$ ,  $-26 \leq l \leq 26$ ; 34562 measured reflections, 17376 independent reflections, 9389 reflections with  $I \geq 2\sigma(I)$ ,  $R_{\text{int}} = 0.0338$ ,  $T_{\text{min}} / T_{\text{max}} = 0.8808 / 0.9293$ ,  $\text{GOOF} = 1.048$ ,  $R_1 = 0.0796$ ,  $wR_2 = 0.2343$  (for  $I \geq 2\sigma(I)$ ),  $R_1 = 0.1207$ ,  $wR_2 = 0.2684$  (for all data),  $\Delta\rho_{\text{min}} / \Delta\rho_{\text{max}} = -1.211 / -2.093$  e/Å $^3$ . CCDC 2283608 contains additional crystallographic parameters for the structure of complex **1**.

## RESULTS AND DISCUSSION

By the interaction of Schiff's base  $\text{H}_2\text{L}$  with  $\text{Co}^{\text{II}}(\text{CF}_3\text{SO}_3)_2 \cdot 6\text{H}_2\text{O}$  and triethylamine in a  $\text{CH}_2\text{Cl}_2$ - $\text{CH}_3\text{CN}$ - $\text{C}_2\text{H}_5\text{OH}$  solvent mixture, rare-type anionic complex **1** was obtained which had the composition  $[\text{Co}^{\text{III}}\text{L}_2]^- \cdot \text{HNet}_3^+ \cdot 1.5\text{H}_2\text{O} \cdot 3\text{MeCN}$  (Scheme 1). The reaction carried out in air was accompanied by Co(II) oxidation to Co(III). Compound **1** was preliminary characterized by  $^1\text{H}$  NMR spectroscopy (DMSO- $d_6$ ). In the spectrum of **1**, the bands of the NH (5.06 ppm) and OH protons (12.78 ppm) disappear compared to the spectrum of the initial compound  $\text{H}_2\text{L}$ . A successful measurement of a well-resolved

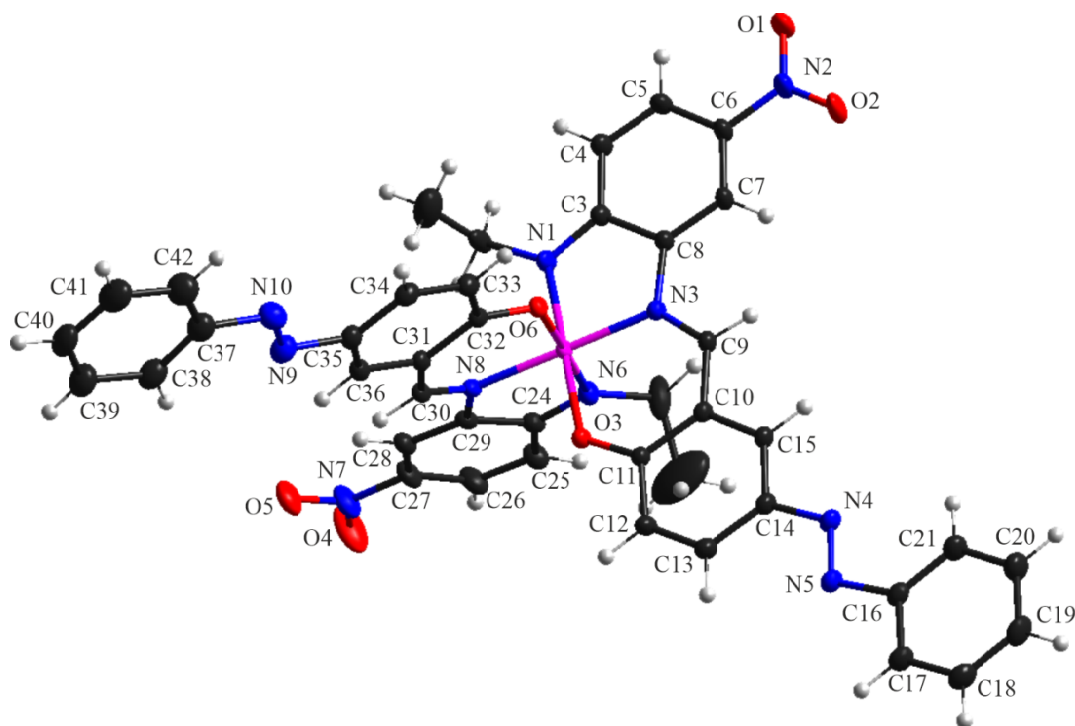


**Scheme 1.** Synthesis of anionic Co(III) complex **1**.

NMR spectrum is fully consistent with diamagnetism of the resulting complex and confirms the oxidation state of the central Co(III) atom.

The synthesis of high-quality single crystals made it possible to unambiguously determine the structure of complex **1** by single crystal XRD (Fig. 1). It is significant that despite the equimolar reagent ratio ( $\text{Co}:\text{H}_2\text{L}:\text{N}(\text{C}_2\text{H}_5)_3 = 1:1:1$ ) the reaction product is a mononuclear bischelatate complex with a ratio  $\text{Co}:\text{L} = 1:2$ . This result can be partially attributed to the introduction of triethylamine in an amount sufficient to deprotonate only half of the  $\text{H}_2\text{L}$  molecules. We have not further investigated the influence of reactant ratios on the composition of the reaction product.

Compound **1** crystallizes in the triclinic crystal system, space group  $P\bar{1}$ . In the complex, two structurally non-equivalent molecules of the doubly deprotonated ligand are tridentate-chelated by the central Co atom. The Co1 atom is in a slightly distorted octahedral environment of four N and two O atoms. The Co–O and Co–N bond lengths are close to 1.91 Å (Table 1), which confirms the trivalent state of the cobalt atom. The N3–Co–N8, N1–Co–O3, N6–Co–O6 bond angles are



**Fig. 1.** Structure of the anionic unit of complex **1** (thermal ellipsoids are shown at 30% probability). Solvate molecules of acetonitrile, triethylamine, and water are omitted.

**TABLE 1.** Main Bond Lengths and Angles in the Structure of **1**

Bond, Å		Angle, deg	
N4–N5	1.254(3)	N3–Co1–N8	179.30(10)
N9–N10	1.210(4)	N3–Co1–N1	84.25(10)
C9–N3	1.302(3)	N8–Co1–N1	95.07(10)
C30–N8	1.289(4)	N3–Co1–O3	95.88(9)
Co1–N3	1.902(2)	N8–Co1–O3	84.80(9)
Co1–N8	1.905(2)	N1–Co1–O3	179.34(11)
Co1–N1	1.908(2)	N3–Co1–N6	95.39(10)
Co1–N6	1.911(3)	N8–Co1–N6	84.42(10)
Co1–O3	1.9097(19)	N1–Co1–N6	90.71(12)
Co1–O6	1.935(2)	O3–Co1–N6	89.93(10)
C3–N1	1.332(4)	N3–Co1–O6	85.61(9)
C8(Et)–N3	1.412(3)	N8–Co1–O6	94.59(9)
C9(Ph)–N3	1.302(3)	N1–Co1–O6	89.88(10)
C11–O3	1.293(3)	O3–Co1–O6	89.48(9)
C24(Ph)–N6	1.330(4)	N6–Co1–O6	178.89(9)
C22(Et)–N6	1.461(4)		
C29–N8	1.418(3)		
C32–O6	1.307(3)		

**TABLE 2.** Hydrogen Bond Parameters in the Crystal

Hydrogen bond	<i>D</i> –H, Å	H··· <i>A</i> , Å	<i>D</i> ··· <i>A</i> , Å	<i>D</i> –H··· <i>A</i> , deg
O1W–H1WA...O6	0.91(4)	1.90(4)	2.777(3)	161(4)
O1W–H1WB...N1S	0.89(2)	2.00(2)	2.872(4)	168(4)
N4S–H4S...O1W (–1+ <i>x</i> , <i>y</i> , <i>z</i> )	1.00	1.71	2.707(5)	174
C7–H7...O2 (– <i>x</i> , 2– <i>y</i> , – <i>z</i> )	0.95	2.53	3.474(4)	171
C9–H9...O2 (– <i>x</i> , 2– <i>y</i> , – <i>z</i> )	0.95	2.45	3.381(4)	165
C15–H15...O1 (– <i>x</i> , 2– <i>y</i> , – <i>z</i> )	0.95	2.49	3.412(4)	165
C9S–H9SA...N3S (–1– <i>x</i> , 2– <i>y</i> , –1– <i>z</i> )	0.95	2.37	3.356(15)	173
C30–H30...O5 (–1– <i>x</i> , 2– <i>y</i> , –1– <i>z</i> )	0.95	2.44	3.375(4)	168
C36–H36...O4 (–1– <i>x</i> , 2– <i>y</i> , –1– <i>z</i> )	0.95	2.43	3.352(6)	164

**TABLE 3.** Parameters of  $\pi$ ··· $\pi$  Interactions in the Crystal Packing of **1** (*Cg* is the phenyl ring centroid; *Cg*–Perp is the shortest distance from *Cg* to the adjacent ring plane;  $\alpha$  is the interplanar angle between *Cg*, *Cg*2–*C*10 → *C*15, *Cg*3–*C*16 → *C*21)

Interaction	<i>Cg</i> ··· <i>Cg</i> , Å	<i>Cg</i> ···Perp, Å	$\alpha$ , deg
<i>Cg</i> 2··· <i>Cg</i> 3 (1– <i>x</i> , 3– <i>y</i> , – <i>z</i> )	3.832(2)	3.3626(12)	5.04(16)

**TABLE 4.** *X*–*Y*··· $\pi$  Interactions in the Crystal Packing of **1** (*Cg* is the aromatic ring centroid; *Y*–Perp is the shortest distance from the *Y* atom to the ring plane; *Cg*5–*C*3 → *C*8, *Cg*6–*C*10 → *C*15, *Cg*8–*C*24 → *C*29, *Cg*9–*C*31 → *C*36, *Cg*10–*C*37 → *C*42)

Interaction	<i>X</i> ··· <i>Cg</i> , Å	<i>X</i> –Perp, Å	$\gamma$ , deg	$\angle Y$ – <i>X</i> ··· <i>Cg</i> , deg	<i>Y</i> ··· <i>Cg</i> , Å
C2–H2B... <i>Cg</i> 5 (– <i>x</i> , 2– <i>y</i> , –1– <i>z</i> )	2.61	2.61	2.30	144	3.451(6)
C4S–H4SA... <i>Cg</i> 6 (– <i>x</i> , 2– <i>y</i> , –1– <i>z</i> )	2.74	2.63	16.55	164	3.694(11)
C4S–H4SC... <i>Cg</i> 1	2.84	2.77	12.93	161	3.781(11)
C11S–H11A... <i>Cg</i> 4	2.85	2.78	12.15	132	3.589(9)
C19–H19... <i>Cg</i> 5 (–1– <i>x</i> , 3– <i>y</i> , – <i>z</i> )	2.89	2.88	6.47	140	3.670(4)
C23–H23A... <i>Cg</i> 2 (–1– <i>x</i> , 2– <i>y</i> , – <i>z</i> )	2.98	2.64	27.76	149	3.854(7)

close to 180°; the N1–Co–N8, O6–Co–O3, O3–Co–N3, O6–Co–N8 bond angles are close to 90° (Table 1). The azomethine C=N bond lengths are 1.289(4) Å and 1.302(3) Å. The structural moieties of the molecule of **1** are in *trans*-position relative

to the azo group; the respective N9–N10–C37, C35–N9–N10, C14–N4–N5, N4–N5–C16 bond angles are 112.1(3)-114.8(3)°. The main structural parameters of complex **1** are given in Table 1.

The mutual arrangement of the aromatic moieties and the nitro group is nearly coplanar; the angle between the planes of the phenyl moieties divided by the C=N group ranges from 1.2(2)° to 15.5(2)°; that of the moieties divided by the N=N group ranges from –5.0(2)° to 10.6(2)°; the angle between the phenyl moiety and the NO<sub>2</sub> group ranges from –2.0(8)° to 2.6(6)°.

The outer-sphere HNEt<sub>3</sub> cation forms an H-bond with the solvate water molecule (Table 2). The crystal packing of the molecules of the complex is explained by the presence of water and acetonitrile solvate molecules, which form intermolecular H-bonds, C–H...O/N/π contacts, and weak π–π interactions between the phenyl rings of coordinated ligands (Tables 2-4).

## CONCLUSIONS

A new anionic cobalt(III) complex with 2-[(E)-(2-(ethylamino)-5-nitrophenylimino)methyl]-4-(phenyldiazenyl)]-phenol was synthesized and structurally characterized. It was shown that the complex crystallized in the triclinic crystal system, space group  $P\bar{1}$ . To the central cobalt ion two ligands are tridentate-chelated by four nitrogen and two oxygen atoms, forming a slightly distorted octahedral environment. Intermolecular hydrogen bonds with water and acetonitrile solvate molecules form the molecular crystal packing of the complex.

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## CONFLICT OF INTERESTS

The authors of this work declare that they have no conflicts of interests.

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