

Dedicated to I. L. Eremenko on the occasion of his 70th birthday

## Cu(II) and Co(II) Complexes with (4Z)-4-[(2-Diethylaminoethylamino)methylene]-5-Methyl-2-Phenylpyrazol-3-One: Synthesis, Magnetic Properties, and Crystal Structures

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**Abstract**—(4Z)-4-[(2-Diethylaminoethylamino)methylene]-5-methyl-2-phenylpyrazol-3-one (HL, product of the condensation of 5-hydroxy-3-methyl-1-phenylpyrazole-4-carbaldehyde with *N,N*-diethylethylenediamine) and its complexes CuL<sub>2</sub> (**I**) and Co(L)CH<sub>3</sub>COO · CH<sub>3</sub>OH · H<sub>2</sub>O (**II**) are synthesized. The structures and compositions of the synthesized compounds are determined by elemental analysis, <sup>1</sup>H NMR and IR spectroscopy, and X-ray diffraction analysis (CIF files CCDC nos. 1971770 (**I**) and 1971772 (**II**)). Bis(chelate) pentacoordinated complex **I** has a distorted tetragonal pyramidal structure, and a distorted octahedral structure with the monodentate acetate anion and methanol and water molecules is observed for monochelate complex **II**.

**Keywords:** metal complexes, magnetic properties, X-ray diffraction analysis, IR and <sup>1</sup>H NMR spectroscopy

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### INTRODUCTION

Metal complexes of azomethine compounds containing pyrazolone fragments attract permanent attention of researchers due to a wide range of their practically useful properties [1–3]. These compounds are characterized by diverse biological activity. In particular, the pyrazole-containing metal complexes have antioxidant activity [4, 5] and antibacterial [4, 6–11], antitubercular [12], antidiabetic [13, 14], anticancer [11, 15–20], and antimicrobial properties [6–8, 21]. Pyrazolone fragments are also observed in a series of biologically active natural alkaloids and are applied in some pharmaceuticals. The pyrazole-containing azo compounds are used as dyes for tissue and leather [22–26]. Some coordination compounds of zinc with the pyrazole-containing ligands exhibit the photoluminescence properties and are promising blue light

sources in the production of electroluminescent devices [27–31]. The copper, nickel, and cobalt compounds have interesting magnetic properties [32–37].

The copper(II) and iron(III) complexes of the aminomethylene and azo derivatives of pyrazoles were widely studied in the respect of modeling active sites of the T1 type of the copper-containing proteins and nitrile hydratase [38–43].

In this work, we present the results of the synthesis and study of the properties of new compounds: (4Z)-4-[(2-diethylaminoethylamino)methylene]-5-methyl-2-phenylpyrazol-3-one (HL, product of the condensation of 5-hydroxy-3-methyl-1-phenylpyrazole-4-carbaldehyde with *N,N*-diethylethylenediamine) and its complexes CuL<sub>2</sub> (**I**) and Co(L)CH<sub>3</sub>COO · CH<sub>3</sub>OH · H<sub>2</sub>O (**II**).

## EXPERIMENTAL

Commercially available *N,N*-diethylethylenediamine, copper acetate monohydrate, and cobalt acetate tetrahydrate (Alfa Aesar) were used. 5-Hydroxy-3-methyl-1-phenylpyrazole-4-carbaldehyde was synthesized using known procedures [44, 45].

**Synthesis of ligand HL.** A solution of *N,N*-diethylethylenediamine (1.16 g, 10 mmol) in methanol (10 mL) was added to a solution of 5-hydroxy-3-methyl-1-phenylpyrazole-4-carbaldehyde (2.02 g, 10 mmol) in methanol (20 mL). The mixture was refluxed for 2 h, and methanol was distilled off on a rotary evaporator. The oily residue was dissolved in dichloromethane (20 mL) and passed through a silica gel layer. The solvent was distilled off on a rotary evaporator to obtain the yellow-brown oil. The yield was 2.64 g (88%).

For  $C_{17}H_{23}N_4O$

Anal. calcd., %	C, 68.20	H, 7.74	N, 18.71
Found, %	C, 68.32	H, 7.80	N, 18.61

IR ( $\nu$ ,  $cm^{-1}$ ): 3243 (NH), 2970 ( $CH_2$ ), 1973, 1660 (C=O).  $^1H$  NMR (DMSO- $d_6$ ),  $\delta$ , ppm: 0.96 (t,  $J = 7.0$  Hz, 6H,  $2CH_2CH_3$ ), 2.15 (s, 3H,  $CH_3$ ), 2.47–2.48 (m, 2H,  $CH_2$ ), 2.50–2.54 (m, 2H,  $CH_2$ ), 2.59 (t,  $J = 6.0$  Hz, 2H,  $CH_2$ ), 3.46 (q,  $J = 6.0$  Hz, 2H,  $CH_2$ ), 7.04–7.09 (m, 1H,  $C_{Ar}-H$ ), 7.31–7.38 (m, 2H,  $C_{Ar}-H$ ), 7.92–7.96 (m, 2H,  $C_{Ar}-H$ ), 7.98–7.99 (m, 1H,  $CH-NH$ ), 9.54–9.59 (m, 1H, NH),

**Synthesis of complexes I and II.** A solution of copper acetate monohydrate (0.10 g, 0.5 mmol) or cobalt acetate tetrahydrate (0.245 g, 0.5 mmol) in methanol (10 mL) and triethylamine (0.1 g, 1 mmol) were added to a solution of ligand HL (0.3 g, 1 mmol) in methanol (10 mL). The mixture was refluxed for 2 h. Precipitates of complexes **I** and **II** formed on cooling were filtered off, washed with methanol (two times by 5 mL), recrystallized from a chloroform–methanol (1 : 2) mixture, and dried in a vacuum drying box at 150°C.

Bis{4-[(*E*)-2-diethylaminoethyliminomethyl]-5-methyl-2-phenylpyrazol-3-olato}copper(II) (**I**): brown crystals, yield 0.27 g (81%), mp = 192–193°C.

For  $C_{34}H_{46}N_8O_2Cu$

Anal. calcd., %	C, 61.65	H, 7.00	N, 16.92	Cu, 9.59
Found, %	C, 61.71	H, 7.10	N, 17.01	Cu, 9.70

IR ( $\nu$ ,  $cm^{-1}$ ): 2972, 2919 ( $CH_2$ ), 1623 (CH=N).  $\mu_{eff} = 2.10 \mu_B$  (294 K).

Acetato(aqua,methanol){4-[(*E*)-2-diethylaminoethyliminomethyl]-5-methyl-2-phenylpyrazol-3-ola-

to}cobalt(II) (**II**): red-brown crystals, yield 0.30 g (82%), mp = 170–171°C.

For  $C_{20}H_{32}N_4O_5Co$

Anal. calcd., %	C, 51.39	H, 6.90	N, 11.99	Co, 12.61
Found, %	C, 51.42	H, 7.04	N, 12.10	Co, 12.73

IR ( $\nu$ ,  $cm^{-1}$ ): 2971, 2916 ( $CH_2$ ), 1638 (C=O), 1619 (CH=N).  $\mu_{eff} = 4.72 \mu_B$  (294 K).

Elemental analyses to C, H, and N were carried out on a Carlo Erba Instruments TCM 480 instrument. Analysis to a metal was conducted by the gravimetric method. Melting points were measured on a Koffler bench.

$^1H$  NMR spectra were recorded on a Varian Unity-300 instrument (300 MHz) in DMSO- $d_6$ .  $^1H$  NMR chemical shifts are presented relative to residual signals of the deuterated solvent. The IR spectra of the samples were recorded on a Varian 3100-FTIR Excalibur instrument in a range of 4000–400  $cm^{-1}$  using the attenuated total internal reflection mode.

Magnetic measurements were carried out on a Lakeshore VSM 7404 magnetometer. Magnetization curves were measured at room temperature in the field range from –19 to 19 kOe. At least 210 points fall onto each measurement with an exposure of 10 s per point.

**X-ray diffraction analysis (XRD).** The unit cell parameters and reflection intensities for complexes **I** and **II** were measured at the XRD station “Kurchatov Institute” using a Rayonix Sx 165CCD two-coordinate detector ( $\lambda = 0.96990 \text{ \AA}$ ,  $T = 100 \text{ K}$ ,  $\phi$  scan mode with an increment of  $1.0^\circ$ ) [46]. Experimental data were processed using the iMOSFLM program implemented into the CCP4 program package [47]. An X-ray radiation absorption correction was applied to the obtained data using the Scala program [48]. Selected crystallographic data and structure refinement parameters are presented in Table 1. The structures were determined by direct methods and refined by full-matrix least squares for  $F^2$  in the anisotropic approximation for non-hydrogen atoms. The positions of the hydrogen atoms of the hydroxyl group of the methanol and water molecules in complex **II** were objectively revealed in the difference Fourier syntheses and included into refinement with fixed positional parameters (riding model) and isotropic displacement parameters  $U_{iso}(H) = 1.5U_{equiv}(O)$ . The positions of other hydrogen atoms in compounds **I** and **II** were calculated geometrically and included into refinement with fixed positional parameters (riding model) and isotropic displacement parameters ( $U_{iso}(H) = 1.5U_{equiv}(C)$  for  $CH_3$  groups and  $U_{iso}(H) = 1.2U_{equiv}(C)$  for other groups). All calculations were performed using the SHELXTL program package [49].

The tables of the atomic coordinates, bond lengths, bond and torsion angles, and anisotropic displace-

**Table 1.** Crystallographic data and structure refinement parameters for complexes **I** and **II**

Parameter	Value	
	<b>I</b>	<b>II</b>
<i>FW</i>	662.34	467.43
<i>T</i> , K	100(2)	100(2)
Single crystal sizes, mm	0.03 × 0.03 × 0.12	0.20 × 0.20 × 0.20
Crystal system	Monoclinic	Orthorhombic
Space group	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>Pbca</i>
<i>a</i> , Å	9.6938(19)	14.236(3)
<i>b</i> , Å	24.876(5)	17.581(4)
<i>c</i> , Å	13.726(3)	18.126(4)
β, deg	95.26(3)	90
<i>V</i> , Å <sup>3</sup>	3296.0(12)	4536.6(17)
<i>Z</i>	4	8
ρ <sub>calc</sub> , g cm <sup>-3</sup>	1.335	1.369
<i>F</i> (000)	1404	1976
μ, mm <sup>-1</sup>	1.617	1.828
θ <sub>max</sub> , deg	38.44	38.43
Number of measured reflections	32008	46599
Number of independent reflections ( <i>R</i> <sub>int</sub> )	6824 (0.084)	4706 (0.052)
Number of reflections with <i>I</i> > 2σ( <i>I</i> )	5166	4180
Number of refined parameters	413	277
<i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> ( <i>I</i> > 2σ( <i>I</i> ))	0.053, 0.126	0.044, 0.112
<i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> (all data)	0.075, 0.145	0.051, 0.122
GOOF for <i>F</i> <sup>2</sup>	1.031	1.009
Extinction coefficient	0.0039(3)	0.0012(1)
<i>T</i> <sub>min</sub> / <i>T</i> <sub>max</sub>	0.818/0.939	0.700/0.700
Δρ <sub>max</sub> /Δρ <sub>min</sub> , e Å <sup>-3</sup>	0.757/−1.020	1.724/−0.531

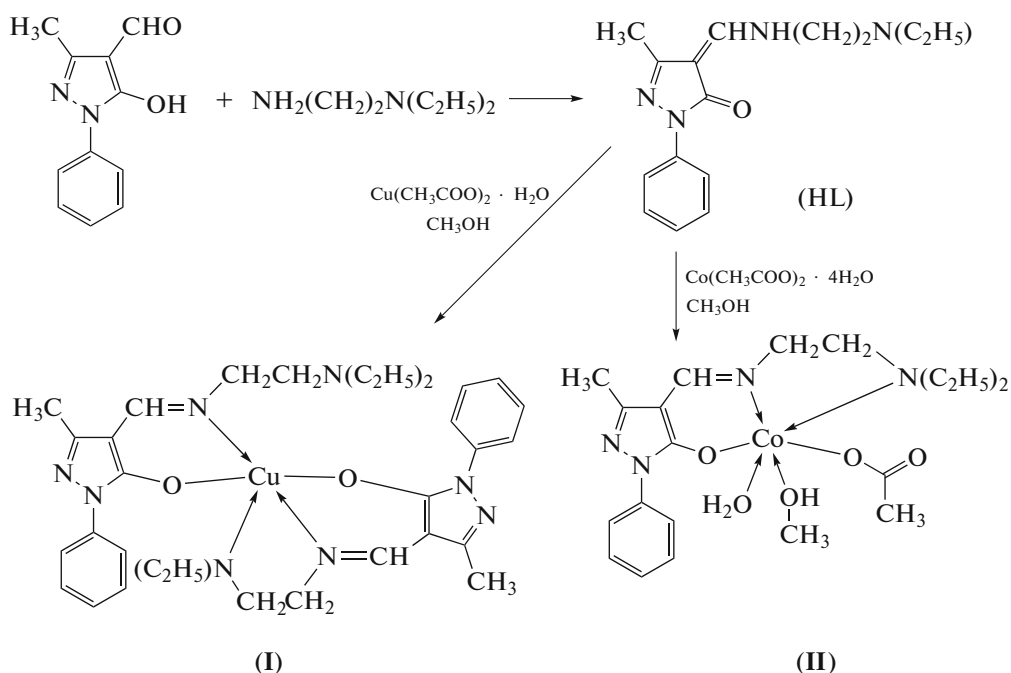
ment parameters for compounds **I** and **II** were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1971770 and 1971772, respectively; deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>)

## RESULTS AND DISCUSSION

Continuing the previous works [27, 28, 30, 50, 51] on studying the 3*d*-metal complexes with the aminomethylene derivatives of 5-hydroxy-3-methyl-1-

phenylpyrazole-4-carbaldehyde, we synthesized the new ligand HL and related Cu(II) (**I**) and Co(II) (**II**) complexes. The presence of the diethylaminoethyl fragment in the ligand molecule made it possible to assume the possibly tridentate character and the involvement of the nitrogen atom of the diethylaminoethyl group in the coordination with the metal when the chelate complexes were formed.

Ligand HL and metal complexes **I** and **II** were synthesized via the following scheme:



The structure of ligand HL was determined by IR spectroscopy,  $^1\text{H}$  NMR spectroscopy, and elemental analysis. As known from the spectral (IR,  $^1\text{H}$  NMR) data and quantum-chemical calculations, at least four tautomeric forms are possible for the pyrazole-containing ligands [1, 50–54]. However, the ketoenamine form is preferable. The conclusion about the ketoenamine structure of HL follows from the IR and  $^1\text{H}$  NMR spectroscopic data. The IR spectrum of HL in this tautomeric form exhibits a  $\nu(\text{NH})$  absorption band at  $3243\text{ cm}^{-1}$  and intense absorption bands at  $1673$  and  $1660\text{ cm}^{-1}$  corresponding to vibrations of the vinylogic amide group  $\text{C}(=\text{O})\text{--CH}=\text{NR}$  [55]. The  $^1\text{H}$  NMR spectrum of HL contains signals of the protons of the  $\text{CHNH}$  groups at  $7.98\text{--}7.99$  ppm and  $\text{NH}$  at  $9.54\text{--}9.59$  ppm.

According to the elemental analysis data, complexes **I** and **II** have different compositions:  $\text{CuL}_2$  and  $\text{Co(L)CH}_3\text{COO} \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$ , respectively. The absorption bands corresponding to the  $\nu(\text{NH})$  vibrations of the ligand disappear from the IR spectra of complexes **I** and **II**, and intense  $\nu(\text{CH}=\text{N})$  absorption bands at  $1619\text{--}1623\text{ cm}^{-1}$  appear. The  $\nu(\text{COO}^-)$  absorption band at  $1638\text{ cm}^{-1}$  is observed for complex **II**. This spectral behavior in the IR absorption range indicates that HL acts in the deprotonated enamine form in the complex formation with metal salts as found earlier for similar metal complexes [1–3, 36, 50, 51, 56].

Complexes **I** and **II** are paramagnetic. The values of  $\mu_{\text{eff}} = 2.10\ \mu_{\text{B}}$  ( $\text{M} = \text{Cu}$ ) and  $4.72\ \mu_{\text{B}}$  ( $\text{M} = \text{Co}$ ) do not change with a temperature decrease to  $77.7\text{ K}$ , indicating their mononuclear structure and the

absence of appreciable intermolecular exchange interactions in these copper(II) ( $d^9$ ,  $S = 1/2$ ) and high-spin cobalt(II) ( $d^7$ ,  $S = 3/2$ ) compounds in the crystalline state at the temperatures higher than  $77.7\text{ K}$ .

The crystal structures of complexes **I** and **II** (Fig. 1) were determined by the XRD method. The copper(II) cation forms complex **I** with two deprotonated ligands HL:  $\text{CuL}_2$  in which one of the organic ligands is tridentate (the oxygen atom and both nitrogen atoms of the azomethine fragment are bound to the metal atom). The second ligand is bidentate: the oxygen atom and imino group of the azomethine fragment are bound to the metal atom (Fig. 1a). Thus, the copper atom in complex **I** exists in the pentacoordinated environment: the coordination polyhedron is better described as a distorted tetragonal pyramid with the amino group of the tridentate ligand arranged at the vertex. The 5-methyl-2-phenylpyrazol-3-olate fragment of both ligands has a flattened structure (root-mean-square deviations of atoms  $0.043$  and  $0.068\ \text{\AA}$ , respectively), which is stabilized by both the system of conjugated bonds and intramolecular hydrogen bonds of the  $\text{C}\text{--}\text{H}\cdots\text{O}$  type (Table 2). The interplanar angle between these fragments is  $59.06(4)^\circ$ .

The copper atom shifts from the plane of the tetragonal pyramid base by  $0.512(1)\ \text{\AA}$  and lies almost in the planes of the 5-methyl-2-phenylpyrazol-3-olate fragments of both ligands. Therefore, the six-membered chelate metalocycles adopt a planar conformation. The five-membered chelate cycle has a typical envelope conformation.

The  $\text{Cu}\text{--}\text{O}$  ( $2.009(2)$  and  $2.144(2)\ \text{\AA}$ ) and  $\text{Cu}\text{--}\text{N}_{\text{imine}}$  ( $1.971(2)$  and  $2.006(2)\ \text{\AA}$ ) distances in the base

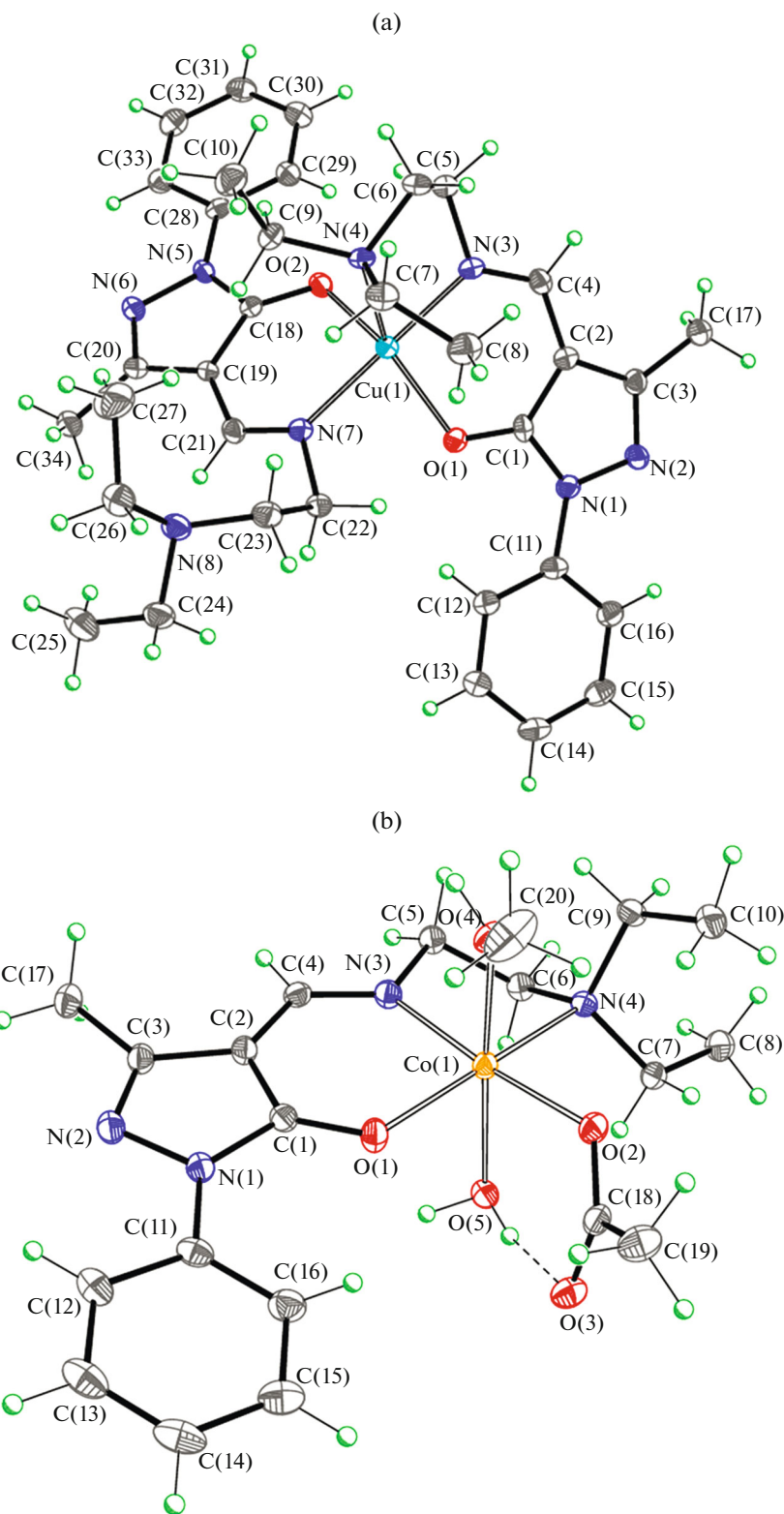


Fig. 1. Molecular structures of complexes (a) I and (b) II.

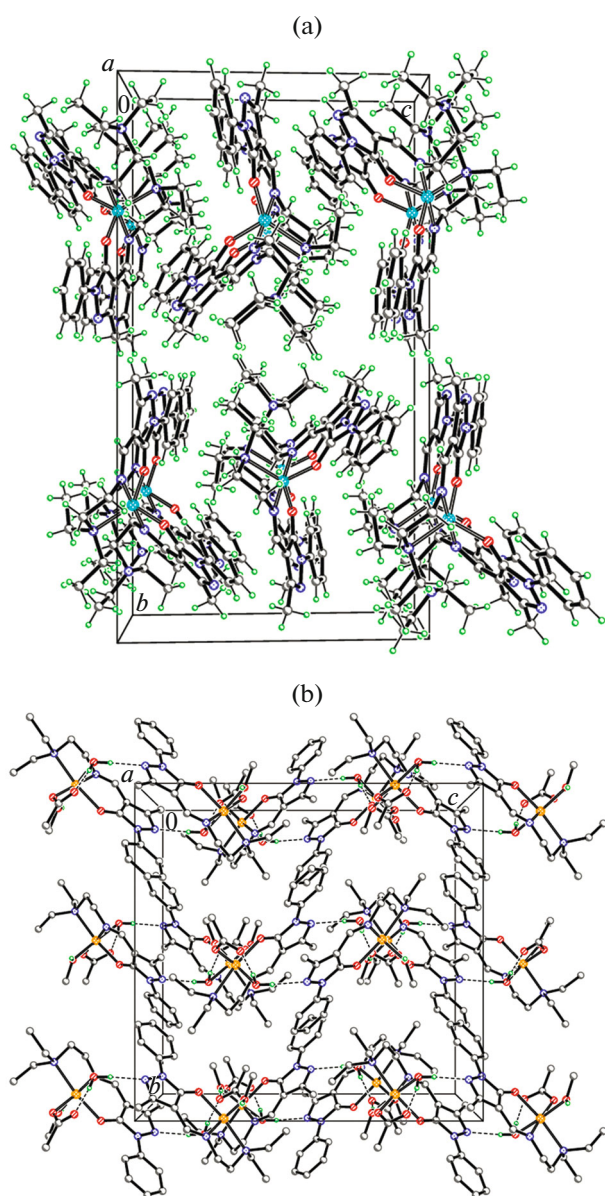
of the coordination pyramid are close, whereas the Cu–N<sub>amine</sub> bond (2.246(2) Å) with the amino group at the vertex is longer.

In the crystal, the molecules of complex I form piles along the crystallographic axis *a* (Fig. 2a). For this packing, the shortest distance between the carbon

**Table 2.** Geometric parameters of hydrogen bonds in the structures of complexes **I** and **II**\*

D–H···A	Distance, Å			Angle D–H···A, deg
	D–H	H···A	D···A	
<b>I</b>				
C(12)–H(12)···O(1)	0.95	2.24	2.896(3)	125
C(29)–H(29)···O(2)	0.95	2.27	2.915(4)	125
<b>II</b>				
O(4)–H(4A)···O(3) <sup>a</sup>	0.91	1.73	2.633(2)	175
O(5)–H(5C)···N(2) <sup>b</sup>	0.91	1.96	2.863(2)	168
O(5)–H(5D)···O(3)	0.92	1.84	2.734(2)	163
C(7)–H(7A)···O(5)	0.99	2.40	3.045(3)	122
C(16)–H(16)···O(1)	0.95	2.24	2.885(3)	124

\* Symmetry procedures for the generation of crystallographically equivalent atoms: <sup>a</sup> $x + 1/2, y, -z + 11/2$ ; <sup>b</sup> $-x + 1, -y + 1, -z + 1$ .

**Fig. 2.** Crystal structures of complexes (a) **I** and (b) **II**.

atoms of the adjacent molecules of complex **I** is C(25)···C(28) ( $1 + x, y, z$ ) 3.509(4) Å (sum of the van der Waals radii of carbon atoms 3.500 Å).

In complex **II**, the cobalt(II) cation coordinates only one deprotonated organic ligand HL in the tridentate form and the coordination environment is supplemented to a distorted octahedral environment by the monodentate acetate anion and methanol and water molecules (Fig. 1b). As in complex **I**, the tridentate ligand HL is characterized by a flattened structure of the 5-methyl-2-phenylpyrazol-3-olate fragment (root-mean-square deviation of atoms 0.091 Å), which is stabilized by both the system of conjugated bonds and intramolecular hydrogen bond C–H···O (Table 2).

The distances Co–O(2)<sub>CH<sub>3</sub>COO</sub> (2.0693(16) Å), Co–O(1)<sub>L</sub> (2.0818(16) Å), and Co–N<sub>imine</sub> (2.066(2) Å) are nearly equalized, whereas other bonds (Co–O(4)<sub>CH<sub>3</sub>OH</sub> (2.1437(17) Å), Co–O(5)<sub>H<sub>2</sub>O</sub> (2.1880(16) Å), and Co–N<sub>amine</sub> (2.2358(19) Å)) are noticeably longer.

In the crystal, the molecules of complex **II** form layers parallel to the (010) plane via hydrogen bonds of the O–H···O and O–H···N types (Table 2, Fig. 2b).

To conclude, depending on the metal nature and under the same synthesis conditions, the tridentate ligand (4Z)-4-[(2-diethylaminoethylamino)methylene]-5-methyl-2-phenylpyrazol-3-one forms the paramagnetic complexes of various compositions and structures: pentacoordinated bis(chelate) complex CuL<sub>2</sub> with the distorted tetragonal pyramidal structure and monochelate complex Co(L)CH<sub>3</sub>COO · CH<sub>3</sub>OH · H<sub>2</sub>O with the distorted octahedral structure containing the monodentate acetate anion and methanol and water molecules.

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IR and  $^1\text{H}$  NMR spectra were recorded using the equipment of the Center for Collective Use Molecular Spectroscopy.

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## CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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