

Nickel(II) and Copper(II) Dicyanoargentate Complexes with Ethylenediamine and 4,4'-Bipyridyl Ligands

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Abstract—The reactions of an aqueous solution of potassium dicyanoargentate with a mixture of nickel(II) or copper(II) chloride and ethylenediamine or 4,4'-bipyridyl in ethanol afford coordination polymers [Ni(En)₂(Ag(CN)₂)]_n [Ag(CN)₂] (I), [Cu(En)₂(Ag(CN)₂)]_n [Ag(CN)₂] (II), and [Cu(4,4'-Bipy)₂(Ag(CN)₂)₂] (III) characterized by XRD (CIF files CCDC nos. 2225984 (I), 2214320 (II), and 2229270 (III)) and IR spectroscopy. According to the XRD data, the crystals of complexes I and II are formed by 1D chains {·NC—Ag—CN—M(En)₂·}_n (M = Ni (I), Cu (II)) linked with each other by the dicyanoargentate anions via argentophilic contacts (Ag···Ag 3.288(8) Å (I), 3.1616(14) Å (II)). The crystal of compound III consists of independent interpenetrating 3D networks built of polymer layers {Cu[Ag(CN)₂]₂}_n bound to each other by the 4,4'-bipyridyl molecules. The bipyridyl linkers connect the Cu centers with the Ag centers of the [Ag(CN)₂][−] anions thus providing the tridentate coordination of the silver atoms. No Ag···Ag interactions are observed in the crystal of complex III.

Keywords: coordination polymers, dicyanoargentates, nickel(II), copper(II), ethylenediamine, 4,4'-bipyridyl

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INTRODUCTION

The design and synthesis of coordination polymers of various architecture are of interest due to their potential use in the very diverse areas (electroconducting and magnetically and optically active materials, catalysis, energy engineering, etc.) [1–3].

The main factor controlling the supramolecular topology and dimensionality of the structure in the design of coordination polymers is the choice of a ligand/metal/linker combination. In addition, various noncovalent interactions, for instance, hydrogen bonds, π – π stacking, metallophilicity, and others, also exert a significant effect on the crystal structures of these complexes.

Cyanometallate linkers based on silver, gold, platinum, and other transition metals play an important role among building blocks for coordination polymers. In particular, the dicyanoargentate derivatives have diverse potentially useful properties such as luminescence [4–9], spin crossover [10–15], and catalytic [16] and biological activity [17–19]. The strategic choice of counterions and auxiliary ligands makes it possible to modify these properties.

Continuing a number of studies on the dicyanoargentate complexes [20–22], in this work we describe the synthesis and specific structural features of the coordination polymers [Ni(En)₂(Ag(CN)₂)]_n [Ag(CN)₂] (I), [Cu(En)₂(Ag(CN)₂)]_n [Ag(CN)₂] (II),

and [Cu(4,4'-Bipy)₂(Ag(CN)₂)₂] (III), where En is ethylenediamine and 4,4'-Bipy is 4,4'-bipyridyl.

EXPERIMENTAL

Commercially available potassium dicyanoargentate (analytical grade, DTsM-Analitika), nickel(II) chloride (reagent grade, Profsnab), copper(II) chloride (98%, ABCR), copper(II) sulfate pentahydrate (analytical grade, Khimreaktivsnab), ethylenediamine (high-purity grade, Khimreaktivsnab), and 4,4'-bipyridyl (high-purity grade, Khimreaktivsnab) were used.

Synthesis of complex [Ni(En)₂(Ag(CN)₂)]_n [Ag(CN)₂] (I). A solution of anhydrous nickel(II) chloride (32 mg, 0.25 mmol) and ethylenediamine (30 mg, 0.50 mmol) in ethanol was added with stirring to a solution of potassium dicyanoargentate (100 mg, 0.50 mmol) in water (5 mL). A transparent violet solution was formed. The solvent was evaporated to isolate violet crystals of compound I with $T_{\text{decomp}} = 199^\circ\text{C}$ in a yield of 85 mg (69%).

IR (ν , cm^{-1}): 3343 s, 3302 w, 3273 s, 3154 w, 2976 m, 2963 m, 2941 s, 2884 m, 2156 s, 2137 s, 1630 w, 1597 s, 1452 m, 1396 w, 1368 w, 1321 m,

1277 m, 1136 m, 1084 m, 1074 w, 1005 s, 970 s, 872 w, 662 s, 581 w, 513 s, 420 m.

For $C_8H_8N_8NiAg_2$

Anal. calcd., %	C, 19.58	H, 1.65
Found, %	C, 19.48	H, 1.70

Complex II was synthesized according to a procedure similar to that of complex **I** using $CuCl_2$ instead of $NiCl_2$.

Complex II: blue-violet crystals, 77% yield, $T_m = 204^\circ C$.

IR (v, cm^{-1}): 3335 s, 3256 s, 3130 w, 2984 w, 2972 m, 2955 w, 2893 w, 2137 s, 1580 s, 1456 m, 1392 w, 1363 w, 1317 w, 1281 w, 1267 m, 1153 w, 1084 m, 1024 s, 976 m, 881 w, 696 s, 604 w, 530 m, 453 w.

For $C_8H_8N_8CuAg_2$

Anal. calcd., %	C, 19.39	H, 1.63
Found, %	C, 19.30	H, 1.69

Synthesis of complex $[Cu(4,4'-Bipy)_2(Ag(CN)_2)_2]$ (III). A solution of copper(II) sulfate pentahydrate (62 mg, 0.25 mmol) in ethanol was added with stirring to a solution of potassium dicyanoargentate (100 mg, 0.50 mmol) and 4,4'-bipyridyl (78 mg, 0.50 mmol) in water (5 mL). A brown-green precipitate was formed and dissolved by the addition of several droplets of a concentrated aqueous solution of ammonia. The evaporation of the solvent gave blue crystals of complex **III** with $T_{decomp} = 184^\circ C$ in a yield of 50 mg (29%).

IR (v, cm^{-1}): 3044 w, 2168 m, 2126 m, 1612 m, 1599 s, 1531 m, 1516 w, 1487 m, 1458 w, 1427 w, 1410 m, 1342 w, 1323 w, 1217 m, 1139 w, 1125 w, 1109 w, 1072 m, 1043 w, 1018 w, 1003 m, 976 w, 957 w, 866 w, 851 w, 839 w, 810 s, 745 w, 729 m, 629 m, 569 m, 559 w, 492 m, 455 m, 420 w.

For $C_{24}H_{16}N_8CuAg_2$

Anal. calcd., %	C, 41.43	H, 2.32
Found, %	C, 41.31	H, 2.37

The IR spectra of compounds **I–III** were recorded on a Shimadzu IRAffinity-1S FT-IR spectrometer, and the samples were prepared by pelleting with KBr (absorption range 4000–400 cm^{-1}).

XRD of the crystals of complexes **I–III** was carried out on a D8 QUEST Bruker diffractometer (Mo K_α radiation, $\lambda = 0.71073 \text{ \AA}$, graphite monochromator). Data were collected and edited, unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [23]. All calculations on structure determination and refinement were performed using the SHELXL/PC [24] and OLEX2 [25] programs. The structures were

determined by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms.

The full tables of the atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2225984, 2214320, and 2229270 for complexes **I–III**, respectively; deposit@ccdc.cam.ac.uk; <http://www.ccdc.cam.ac.uk>).

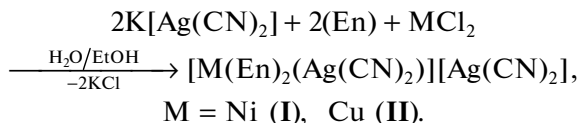
Compound I: violet crystals, triclinic, space group $P\bar{1}$, $a = 6.576(17)$, $b = 8.354(18)$, $c = 8.44(2) \text{ \AA}$, $\alpha = 102.13(13)^\circ$, $\beta = 109.60(14)^\circ$, $\gamma = 108.67(9)^\circ$, $V = 386.7(17) \text{ \AA}^3$, $Z = 1$, $\rho_{calc} = 2.107 \text{ g/cm}^3$; $\mu = 3.709 \text{ mm}^{-1}$, $F(000) = 234.0$. Total number of measured reflections 20 117, independent reflections 4402 ($R_{int} = 0.0521$), refinement parameters 91: $R_1 = 0.0457$, $wR_2 = 0.1245$.

Compound II: blue-violet crystals, orthorhombic, space group $Immm$, $a = 6.323(3)$, $b = 9.033(4)$, $c = 13.217(6) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$, $V = 754.9(6) \text{ \AA}^3$, $Z = 2$, $\rho_{calc} = 2.215 \text{ g/cm}^3$; $\mu = 3.963 \text{ mm}^{-1}$, $F(000) = 486.0$. Total number of measured reflections 6097, independent reflections 557 ($R_{int} = 0.0280$), refinement parameters 46: $R_1 = 0.0182$, $wR_2 = 0.0414$.

Compound III: blue crystals, monoclinic, space group $P2_1/n$, $a = 8.952(5)$, $b = 11.505(4)$, $c = 12.608(5) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 106.42(2)^\circ$, $\gamma = 90^\circ$, $V = 1245.4(9) \text{ \AA}^3$, $Z = 2$, $\rho_{calc} = 1.855 \text{ g/cm}^3$; $\mu = 2.432 \text{ mm}^{-1}$, $F(000) = 678.0$. Total number of measured reflections 62964, independent reflections 9831 ($R_{int} = 0.0396$), refinement parameters 160: $R_1 = 0.0401$, $wR_2 = 0.0900$.

RESULTS AND DISCUSSION

Polymer complexes **I** and **II** in the single-crystal form were prepared by the reaction of an aqueous solution of potassium dicyanoargentate with a mixture of ethylenediamine and nickel(II) chloride (**I**) or copper(II) chloride (**II**) (2 : 2 : 1 mol/mol/mol) in ethanol



Note that the structure of complex **II** has already been determined previously but in other space group ($Pnmm$) [26].

Attempts to isolate crystals of the copper(II) complex with 4,4'-bipyridyl using a similar procedure were unsuccessful because of the very fast formation of a fine precipitate of the product insoluble in water and available organic solvents (acetone, acetonitrile, chloroform, THF, DMSO, and DMF) for subsequent crystallization. The addition of several droplets of a concentrated aqueous solution of ammonia to the

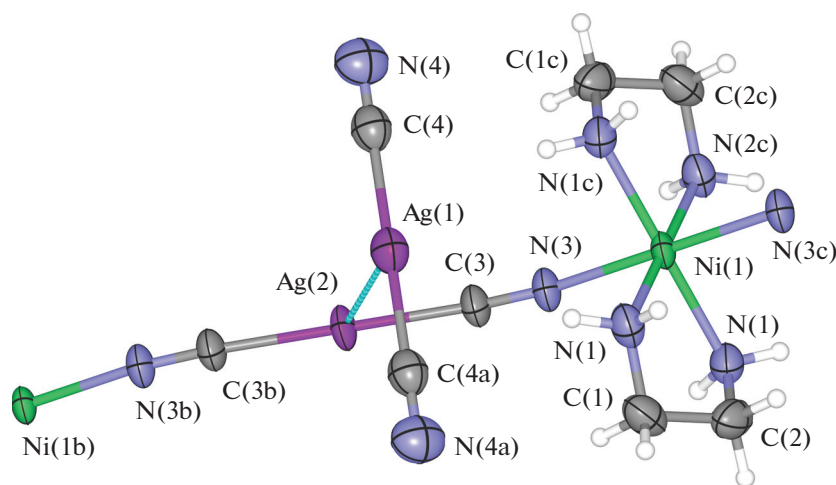
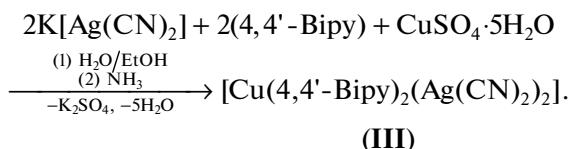


Fig. 1. Structure of the fragment of complex I.

reaction mixture until the precipitate was dissolved completely followed by solvent evaporation gave blue single crystals of product **III** suitable for XRD.



A similar approach has previously been described [27] for the polymer dicyanoaurate complexes $[\text{M}(\text{Bpym})(\text{NH}_3)_2(\text{Au}(\text{CN})_2)_2]$ ($\text{M} = \text{Co}, \text{Ni}, \text{Cu}$; Bpym is 2,2'-bipyrimidine). However, thus synthesized complex **III** contains no ammonia molecules in the coordination sphere of the Cu^{2+} cations, most likely, due to a higher affinity to the 4,4'-Bipy ligand.

The presence of cyano groups in organic and inorganic compounds can rather easily be found using IR spectroscopy: the corresponding absorption bands lie in a sufficiently narrow frequency range from 2200 to 2000 cm^{-1} [28]. Cyanide ligands in the dicyanoargentate (as well dicyanoaurate) complexes are divided into terminal and bridging ligands, and rather certain frequency range in the IR range corresponds to each type. The absorption bands lying lower than 2150 cm^{-1} indicate the presence of the terminal $\text{Ag}-\text{C}\equiv\text{N}$ fragment, and the values higher than 2150 cm^{-1} indicate the presence of the $\text{Ag}-\text{C}\equiv\text{N}-\text{M}$ bridges and polymer character of the complex [29]. Thus, the IR spectra are important for understanding the structures of the dicyanoargentate derivatives and often allow one to propose their structures before performing XRD.

For instance, for complexes **I–III**, the absorption bands of stretching vibrations of the $\text{C}\equiv\text{N}$ bonds in the $[\text{Ag}(\text{CN})_2]^-$ anion are highly intense and lie at 2156 and 2137 (**I**), 2137 (**II**), and 2168 and 2126 cm^{-1} (**III**), which assumes that complexes **I** and **III**, unlike prod-

uct **II**, contain both the terminal and bridging $\text{C}\equiv\text{N}$ groups.

The IR spectra of ethylenediamine-containing compounds **I** and **II** also distinctly exhibit absorption bands of NH_2 group vibrations: at 3343, 3273 cm^{-1} (**I**) and 3335, 3256 cm^{-1} (**II**) for stretching vibrations and at 1597 cm^{-1} (**I**) and 1580 cm^{-1} (**II**) for bending vibrations [28].

According to the XRD data, nickel- and copper-containing complexes **I** and **II** have polymer structures similar to each other and are presented in the crystal by weakly bent 1D chains built of the μ_2 -bridging dicyanoargentate anions and octahedrally coordinated M^{2+} cations ($\text{M} = \text{Ni}$ (**I**), Cu (**II**)). The axial positions of the latter are occupied by the nitrogen atoms of the $[\text{Ag}(\text{CN})_2]^-$ ions. The equatorial plane around the Ni and Cu centers is formed by four nitrogen atoms from two chelate ethylenediamine molecules. Charge compensation occurs due to the uncoordinated dicyanoargentate anions that join the $\{\cdots\text{NC}-\text{Ag}-\text{CN}-\text{M}(\text{En})_2\cdots\}_n$ chains into *pseudo*-2D massifs using argentophilic interactions $\text{Ag}\cdots\text{Ag}$ (3.288(8) Å (**I**) and 3.1616(14) Å (**II**)). The doubled van der Waals radius of the silver(I) atom is 3.44 Å [30] and, hence, these contacts in products **I** and **II** can be considered significant. The structure of complex **I** and the fragment of its crystal organization are shown in Figs. 1 and 2, respectively.

Both bridging and free $[\text{Ag}(\text{CN})_2]^-$ ions in complexes **I** and **II** have the typical linear geometry. The $\text{Ag}-\text{C}$ and $\text{C}\equiv\text{N}$ bond lengths in the free anions are 2.042(5), 1.106(5) Å (**I**) and 2.054(3), 1.125(5) Å (**II**), respectively. Those in the bridging anions are 2.080(6), 1.154(4) Å (**I**) and 2.064(4), 1.124(6) Å (**II**), respectively.

The $\text{M}-\text{N}$ distances in products **I** and **II** vary in different ranges. For complex **I**, the range is relatively

product, all $[\text{Ag}(\text{CN})_2]^-$ anions play the role of μ_2 -bridges and join the Cu^{2+} cations into wavy 2D polymer layers $\{\text{Cu}[\text{Ag}(\text{CN})_2]_2\}_n$. The 4,4'-bipyridyl molecules together with dicyanoargentate anions act as linear μ_2 -bridging ligands: they coordinate to the copper cores of one $\{\text{Cu}[\text{Ag}(\text{CN})_2]_2\}_n$ layer and thus penetrate the adjacent polymer layer and bind to the silver cores of the next polymer layer. Thus, the final crystal structure of complex **III** is presented by two independent interpenetrating 3D polymer networks. In this case, no $\text{Ag}\cdots\text{Ag}$ contacts are observed, since the dicyanoargentate anions are rather remote from each other. The structure of complex **III** and the fragment of independent interpenetrating 3D polymer networks are shown in Figs. 3 and 4, respectively.

The geometry of the $[\text{Ag}(\text{CN})_2]^-$ ions in this complex significantly differs from the linear one due to bringing together the silver and nitrogen atoms of the 4,4'-bipyridyl ligands. Thus, the Ag centers are tricoordinated with $\text{Ag}(1)-\text{N}(2a)$, $\text{Ag}(1)-\text{C}(11)$, and $\text{Ag}(1)-\text{C}(12)$ distances of 2.4841(18), 2.0955(16), and 2.0881(16) Å, respectively, and the $\text{C}(11)\text{Ag}(1)\text{C}(12)$ angle decreases to $157.32(6)^\circ$. The lengths of the $\text{C}(11)-\text{N}(3)$ and $\text{C}(12)-\text{N}(4)$ triple bonds in the anions are not equivalent: 1.1453(19) and 1.137(2) Å. Nonuniformity of the $\text{Cu}-\text{N}$ distances is also observed inside each $\{\text{Cu}[\text{Ag}(\text{CN})_2]_2\}_n$ layer: the $\text{Cu}(1)-\text{N}(3)$ bond length is 1.9615(14) Å, whereas the $\text{Cu}(1)-\text{N}(4)$ distance is significantly longer and equal to 2.568 Å. The differences in the $\text{Ag}-\text{C}$, $\text{C}\equiv\text{N}$, and $\text{Cu}-\text{N}$ bond lengths can explain the appearance of two absorption bands of cyano groups in the IR spectrum of the product, although the crystal of complex **III** formally contains dicyanoargentate anions of only one type. Two remained sites in the octahedral environment of the Cu^{2+} cations are occupied by the nitrogen atoms of two 4,4'-bipyridyl ligands: the $\text{Cu}(1)-\text{N}(1)$ bond length is 2.0636(15) Å.

Thus, the reactions of potassium dicyanoargentate with nickel(II) and copper(II) chlorides in the presence of ethylenediamine or 4,4'-bipyridyl afforded the coordination polymers $[\text{Ni}(\text{En})_2(\text{Ag}(\text{CN})_2)]-[\text{Ag}(\text{CN})_2]$, $[\text{Cu}(\text{En})_2(\text{Ag}(\text{CN})_2)][\text{Ag}(\text{CN})_2]$, and $[\text{Cu}(4,4'\text{-Bipy})_2(\text{Ag}(\text{CN})_2)_2]$. The ethylenediamine derivatives contain dicyanoargentate anions of two types: bridging and free. The latter link the $\{\cdots\text{NC}-\text{Ag}-\text{CN}-\text{M}(\text{En})_2\}_n$ chains into the *pseudo*-2D massifs using the $\text{Ag}\cdots\text{Ag}$ contacts with the bridging anions of the chains. The $[\text{Cu}(4,4'\text{-Bipy})_2(\text{Ag}(\text{CN})_2)_2]$ complex contains only bridging anions linking the Cu^{2+} centers into the $\{\text{Cu}[\text{Ag}(\text{CN})_2]_2\}_n$ layers. The 4,4'-Bipy ligands coordinate with the copper and silver

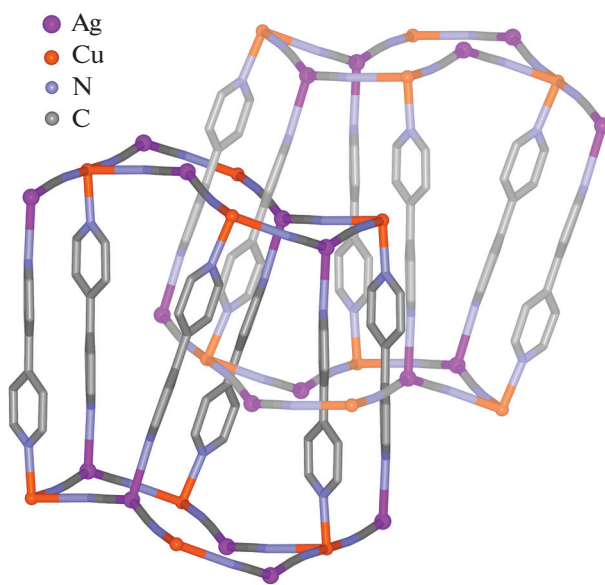


Fig. 4. Independent interpenetrating 3D polymer networks in the structure of complex **III**.

cores to join the layers into the independent interpenetrating 3D networks without $\text{Ag}\cdots\text{Ag}$ interactions.

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CONFLICT OF INTEREST

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

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