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Copper(II) and Silver(I) Complexes with Chiral N-Substituted Aminoacetic Acid Derivatives Containing the Natural Monoterpenes (+)-3-Carene and (–)-α-Pinene: Synthesis and Structures

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Abstract—The complexes [Cu(HL)Cl] · $0.25H_2O$ (**I**), [Cu(HL^1)Cl] · $0.5H_2O$ (**II**), [Ag(HL)] · $0.5H_2O$ (**III**) were obtained; HL^- and $(HL^1)^-$ are the anions of the chiral N-substituted aminoacetic acid derivatives containing the natural monoterpenes (+)-3-carene and (–)-α-pinene, respectively. The μ_{eff} values for paramagnetic complexes **I** and **II** are 1.92 and 1.81 μ_B, respectively. Single crystals of the coordination 1D polymer $[Cu(HL)Cl \cdot 2H_2O]_n$ (**IV**) were grown. According to the X-ray diffraction data (CIF file CCDC No. 1035219), the crystal structure of complex **IV** is made up of the zigzag chains $[Cu(HL)Cl]_n$ and water molecules. The coordination polyhedron of the Cu atom is a distorted square pyramid (ClN_2O_2). The anion HL^- acts as a chelating and bridging tetradentate ligand coordinated through the N atoms of the NH and NOH groups as well as through the O atoms of the COO^- groups linking two adjacent Cu atoms together. The IR spectra of complexes **I** and **II** also provide evidence for the coordination of the functional COO^-, NOH, and NH groups. In complex **III**, the coordination sphere of the Ag atom is made up of the O atoms of the COO^- group. Complex **III** shows weak green photoluminescence (λ_{max} = 530 nm).

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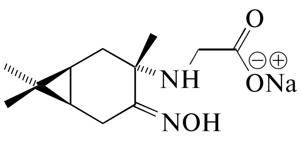
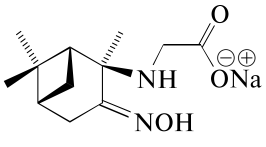
Metal complexes with natural amino acids and their derivatives are subjects of many investigations [1–4]. Much attention is also given to metal complexes with optically active derivatives of natural terpenoids [5–9]. Terpenoids are extracted from plant-based raw materials; many of them possess medicinal properties [10, 11]. For this reason, "hybrid" organic compounds combining the fragments of N-substituted amino acid derivatives and chiral terpenoids are of great interest as ligands. Such a type of ligands is promising for the synthesis of a variety of metal complexes exhibiting valuable functional properties. Recently, we have obtained the Zn(II) and Cd(II) complexes with the anions of the (+)-3-carene derivative, *N*-[(1*S*,3*S*,6*R*)-4-hydroxyimino-3,7,7-trimethylbicyclo[4.1.0]hept-3-yl]aminoacetic acid (**HL^-**), and the (–)-α-pinene derivative, *N*-[(1*R*,2*R*,5*R*)-3-hydroxyimino-2,6,6-trimethylbicyclo[3.1.1]hept-3-yl]aminoacetic acid (**HL^1**$)^-$ [12]. The salts HLNa and HL^1Na were used as the starting materials. The Zn(II) and Cd(II) complexes obtained show blue photoluminescence.



HLNa



HL^1Na

We found it interesting to study the possibility of obtaining complexes of Group 11 metals with the above ligands. Since copper is a "metal of life", Cu(II) complexes with such chiral hybrid ligands could be expected to possess potential biological activity. Silver(I) complexes are known for their bactericidal properties.

The goal of this work was to obtain Cu(II) and Ag(I) complexes with chiral derivatives of (+)-3-carene (HL^-) and (–)-α-pinene $(HL^1)^-$ and study the structures and properties of these complexes.

EXPERIMENTAL

The salts $CuCl_2 \cdot 2H_2O$ and $AgNO_3$ were analytical grade chemicals. Rectified ethanol and distilled water were used. The salts HLNa ($[\alpha]^{23}_{589}$ +150 (*c* 0.61, H_2O))

and $HL^{(1)}Na$ ($[\alpha]^{23}_{589}$ −57 (c 0.74, H_2O) were prepared as described in [12].

Synthesis of $[Cu(HL)Cl] \cdot 0.25H_2O$ (I)). A solution of $CuCl_2 \cdot 2H_2O$ (0.051 g, 0.3 mmol) in EtOH (2 mL) was added to a solution of HLNa (0.050 g, 0.2 mmol) in EtOH–H_2O (2 : 1 v/v, 3 mL). The resulting solution was stirred and concentrated to a minimum possible volume of ~2 mL. The azure precipitate was filtered off by suction, washed with cold EtOH, and dried in a vacuum desiccator. The yield of complex **I** was 0.030 g (42%).

For $C_{12}H_{19}N_2O_{3.25}ClCu$

anal. calcd. (%):	C, 42.0;	H, 5.7;	N, 8.2.
Found (%):	C, 40.8;	H, 5.6;	N, 7.8.

Synthesis of $[Cu(HL^1)Cl] \cdot 0.5H_2O$ (II). A solution of EtOH–H_2O (0.051 g, 0.3 mmol) in EtOH (2 mL) was added to a solution of HL^1Na (0.050 g, 0.2 mmol) in $CuCl_2 \cdot 2H_2O$ (2 : 1 v/v, 3 mL). Further workup followed the protocol described above for complex **I**. The yield of complex **II** was 0.030 g (42%).

For $C_{12}H_{20}N_2O_{3.5}ClCu$

anal. calcd. (%):	C, 41.5;	H, 5.8;	N, 8.1.
Found (%):	C, 40.6;	H, 5.7;	N, 7.6.

Synthesis of $[Ag(HL)] \cdot 0.5H_2O$ (III). A solution of HLNa (0.065 g, 0.25 mmol) in water (2 mL) was added to a solution of $AgNO_3$ (0.034 g, 0.2 mmol) in water (2 mL). The reaction mixture containing a white precipitate was kept in a refrigerator at ~3°C for 1 h. The precipitate was filtered off by suction, washed with cold water, and dried in a vacuum desiccator. The yield of complex **III** was 0.033 g (46%).

For $C_{12}H_{20}N_2O_{3.5}Ag$

anal. calcd. (%):	C, 40.5;	H, 5.7;	N, 7.9.
Found (%):	C, 39.9;	H, 5.5;	N, 7.7.

Microanalyses for C, H, and N were performed on a Euro EA 3000 analyzer. The thermal behavior of complexes **I–III** (sample weight ~10 mg, alumina crucibles, heating rate 10°C/min) in a helium atmosphere was studied on a TG 209 F1 Iris® thermobalance (NETZSCH). IR spectra (Nujol, KBr pellets) were recorded on Scimitar FTS 2000 and Vertex 80 FTIR spectrometers in the 4000–100 cm^{-1} range. Excitation ($V = 600$ V, slit width 5 nm) and emission spectra ($V = 600$ V, slit width 5 nm, $\lambda_{ex} = 350$ nm) were acquired on a Cary Eclipse spectrophotometer (Varian) at room temperature under identical experimental conditions.

Synthesis of $\{[Cu(HL)Cl] \cdot 2H_2O\}_n$ (IV). A solution prepared as described for the synthesis of complex **I** was kept in a loosely closed beaker at ~3°C. The resulting single crystals suitable for X-ray diffraction were withdrawn and immediately covered with Vaseline oil because of their instability in air.

Table 1. Crystallographic parameters and the data collection and refinement statistics for the coordination polymer **IV**

Parameter	Value
Empirical formula	$C_{12}H_{23}ClCuN_2O_5$
M	374.31
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell parameters:	
a, Å	8.0185(4)
b, Å	9.6335(5)
c, Å	11.0970(6)
β, deg	108.017(1)
V, Å^3	815.17(7)
Z; r_{calcd}, g/cm^3	2; 1.525
μ, mm^{-1}	1.524
Crystal dimensions, mm	0.30 × 0.22 × 0.12
θ scan range, deg	2.67–30.55
Number of measured reflections	9162
Number of unique reflections (R_{int})	4871 (0.0245)
Number of reflections with $I > 2\sigma(I)$	4723
Number of parameters refined	206
GOOF on F^2	0.941
R factor ($I > 2\ \sigma(I)$)	$R_1 = 0.0186$, $wR_2 = 0.0476$
R factor (for all I_{hkl})	$R_1 = 0.0192$, $wR_2 = 0.0478$
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.257/–0.363

X-ray diffraction study of complex IV. The unit cell parameters and reflection intensities were measured at 150 K on a Bruker X8 APEX CCD automated diffractometer (area detector, MoK_α radiation, λ = 0.71073 Å, graphite monochromator). Structure **IV** was solved by direct methods and refined anisotropically (for non-hydrogen atoms) by the full-matrix least-squares method on F^2 with the SHELXL-97 program package [13]. The hydrogen atoms of water molecules were located in a difference electron-density map and refined isotropically together with their parent atoms. Crystallographic parameters and the data collection and refinement statistics for structure **IV** are summarized in Table 1. The refined values for selected bond lengths and bond angles are given in Table 2.

The comprehensive tables of the atomic coordinates, bond lengths, and bond angles for structure **IV** have been deposited with the Cambridge Structural Database (no. 1035219; deposit@ccdc.cam.ac.uk or

Table 2. Selected bond lengths and bond angles in structure **IV***

Bond	*d*, Å	Bond	*d*, Å
Cu(1)–O(1')**	1.974(1)	C(1)–C(12)	1.514(2)
Cu(1)–O(2)	2.068(1)	C(2)–C(3)	1.521(2)
Cu(1)–N(1)	2.007(1)	C(2)–C(11)	1.541(2)
Cu(1)–N(2)	2.010(1)	C(2)–C(7)	1.549(2)
Cu(1)–Cl(1)	2.3625(4)	C(3)–C(4)	1.495(2)
O(1)–C(1)	1.258(2)	C(4)–C(5)	1.526(2)
O(2)–C(1)	1.266(2)	C(5)–C(8)	1.509(2)
O(3)–N(2)	1.390(1)	C(5)–C(6)	1.518(2)
O(3)–H(3)	0.820	C(6)–C(7)	1.518(2)
N(1)–C(12)	1.478(2)	C(6)–C(8)	1.517(2)
N(1)–C(2)	1.504(2)	C(8)–C(10)	1.506(2)
N(1)–H(1)	0.910	C(8)–C(9)	1.519(2)
N(2)–C(3)	1.283(2)	O(2*w*)–H(21*w*)	0.958(5)
O(1w)–H(11*w*)	0.962(5)	O(2*w*)–H(22*w*)	0.959(5)
O(1*w*)–H(12*w*)	0.960(5)		
Angle	**ω, deg**	**Angle**	**ω, deg**
O(1')**Cu(1)O(2)	88.47(3)	O(2)Cu(1)N(2)	140.03(4)
O(1')**Cu(1)N(2)	97.93(4)	O(1')**Cu(1)N(1)	163.01(4)
O(2)Cu(1)N(1)	82.65(4)	O(1')**Cu(1)Cl(1)	97.66(3)
N(1)Cu(1)N(2)	80.10(4)	O(2)Cu(1)Cl(1)	106.86(3)
N(1)Cu(1)Cl(1)	98.77(3)	N(2)Cu(1)Cl(1)	111.23(3)
H(11*w*)O(1*w*)H(12*w*)	110(1)	H(21*w*)O(2*w*)H(22*w*)	112(1)

* The symmetry operation code is ** 2 – *x*, *y* + 1/2, 1 – *z*.

http://www.ccdc.cam.ac.uk) or can be made available from the authors upon request.

RESULTS AND DISCUSSION

Complexes **I** and **II** were obtained in aqueous ethanol; a small molar excess of the metal (Cu^{2+} : ligand = 1.5 : 1) was used. Complexes **I** and **II** are paramagnetic (μ_{eff} = 1.92 and 1.81 μ_B, respectively), which suggests their electronic configuration d^9. Complex **III** was synthesized in water; the starting reagents were used in a stoichiometric ratio. We failed to isolate the complex [Ag(HL^1)]: as with complex **III**, a reaction of Ag^+ with $(HL^1)^-$ produces a white amorphous precipitate. However, the precipitate turns dark brown when dried even in a dark desiccator. Apparently, Ag^+ is reduced to Ag^0. All the complexes obtained contain a small amount of crystal water (0.25 to 0.5 water molecules). Slow crystallization of complex **I** afforded single crystals of coordination polymer **IV** containing two water molecules. Complexes **I** and **II** are soluble in polar solvents (water, EtOH, *iso*-PrOH, and acetone) but are poorly soluble in nonpolar solvents. Complex **III** is soluble in EtOH.

The crystal water content of complexes **I–III** was estimated by thermogravimetry. The TG curves of complexes **I** and **II** show gradual weight losses (1.4 and 2.9%, respectively) by dehydration over a range from 40 to 190°C. These values agree with the calculated water content (1.3 and 2.6%, respectively). The thermal degradation of complexes **I** and **II** occurs exothermically at 190–220°C. For complex **III**, the first weight loss (2.5%) is observed in a range from 40 to 170°C, which corresponds to the complete removal of its crystal water (the calculated water content is 2.5%). Further degradation occurs exothermically above 170°C. Complexes **I–III** are thermally less stable than similar Zn(II) complexes [12].

According to the X-ray diffraction data, the crystal structure of complex **IV** is made up of zigzag chains $[Cu(HL)Cl]_n$ of the 1D coordination polymer (aligned with the *y* axis) and crystal water molecules (Fig. 1). The complex contains two water molecules per each Cu(HL)Cl fragment. The anion HL^- acts as a chelating and bridging tetradentate ligand. The coordination polyhedron of the Cu atom is a distorted square pyramid. The base of the pyramid is made up of two O atoms of the bidentate COO^- groups (bridging two Cu

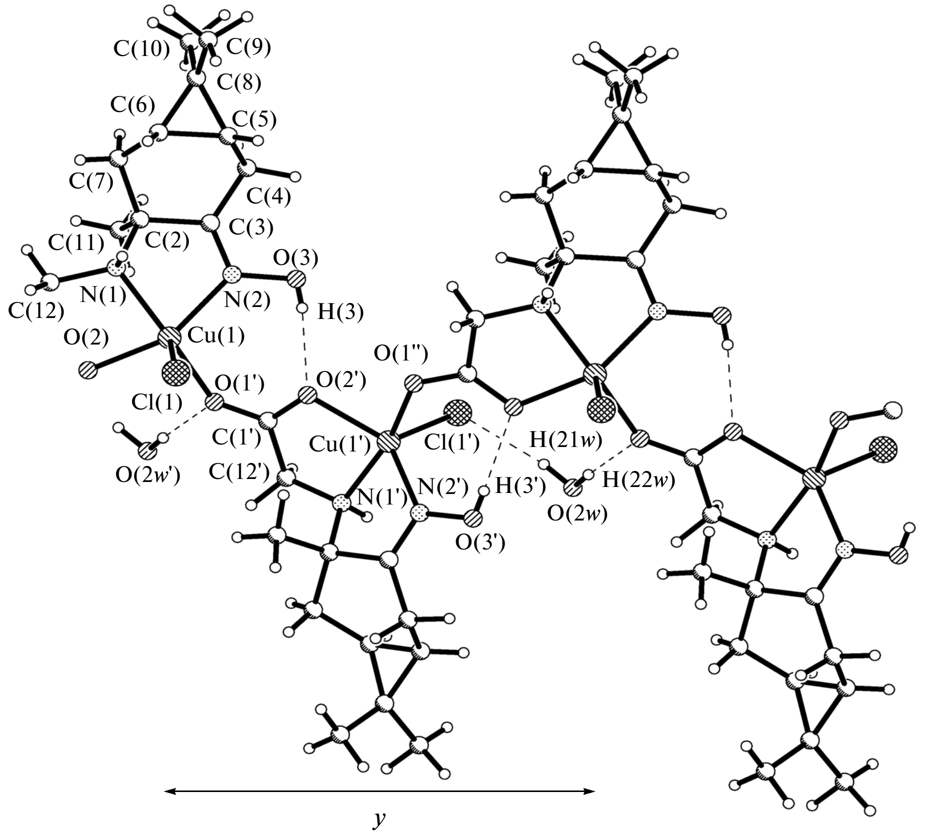


Fig. 1. Fragment of the structure made up of the polymer chains $[Cu(HL)Cl]_n$ and the crystal water molecules along the *y* axis.

atoms) and two N atoms of the oxime and imine groups. The apical position is occupied by the Cl atom. The bond lengths and bond angles in structure **IV** (Table 2) agree with those found in the isostructural coordination polymer $\{[Zn(HL)Cl] \cdot 2H_2O\}_n$ (**V**) [12]; i.e., the variation in the metal atom has no appreciable effect on the bond lengths. Yet some distinctions are worth noting. In the copper(II) complex **IV**, the Cu–O bonds differ by 0.1 Å, the Cu–N bonds being almost equal to each other. In the zinc(II) complex **V**, the Zn–O and Zn–N bonds differ by 0.04 Å.

Coordination of HL^- in structure **IV** gives rise to two five-membered chelate rings CuN_2C_2 (**A**) (N(1)Cu(1)N(2), 80.10(4)°) and $CuNOC_2$ (**B**) (N(1)Cu(1)O(2), 82.65(4)°). Both rings adopt an envelope conformation; the C(2) atom in Å is 0.570(2) Å apart and the N(1) atom in **B** is 0.297(2) Å apart from the planes of the four other atoms. The rings **A** and **B** are planar to within 0.080(5) and 0.022(5) Å, respectively. In addition, the strong intramolecular hydrogen bond O(2')···H–O(3) (O(2')···O(3) 2.710(1) Å serves to form the chelate ring Cu(1)O(1')C(1')O(2')H(3)O(3)N(2). The six-membered carbocycle adopts a distorted boat conformation: the C(4) and C(7) atoms deviate from the plane of the four other atoms by 0.672(2) and 0.495(2) Å, respectively. The dimethylcyclopropane fragment shares the C(5)–C(6) edge with the six-membered carbocycle C(2)C(3)C(4)C(5)C(6)C(7) and makes a dihedral angle of 139.5° with the plane C(2)C(3)(4)C(5)C(6).

In the chain $[Cu(HL)Cl]_n$, the distance between the adjacent Cu atoms is 5.308(1) Å. These translationally identical zigzag chains are hydrogen-bonded to crystal water molecules (O(2*w*')···O(1') 2.875(1), O(2*w*)···Cl(1') 3.207(1) Å) (Fig. 1).

The IR spectra of HLNa and HL^1Na have been examined in [12]. In the IR spectra of complexes **I** and **II**, the bands $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ are shifted to the higher frequencies against their positions in the spectra of the starting salts. The nearly equal frequency difference $\Delta\nu(COO^-) = \nu_{as}(COO^-) - \nu_s(COO^-)$ for both complexes **I** and **II** and the salts HLNa and HL^1Na (Table 3) suggests that the COO^- group functions as a bridging bidentate ligand in the coordination with Cu^{2+} ions [14, 15]. The electron-donating functional NOH and NH groups are also coordinated: the band ν(NO) and the bands ν(NH) and ν(OH) in the spectra of complexes **I** and **II** are shifted against their positions in the spectra of HLNa

Table 3. Selected vibrational frequencies (cm^{-1}) in the IR spectra of compounds HLNa and HL^{1}Na and complexes **I–III**

Assignment	HLNa*	I	III	HL^{1}Na*	II
ν(NO)	945	974	943	972	996
ν_{as}(COO$^-$)	1599	1656	1578	1584	1667
ν_s(COO$^-$)	1411	1460	1459	1389	1482
$\Delta\nu$	188	195	119	195	185
ν(NH), ν(OH)	3073, 3210, 3295	3102, 3137, 3339, 3449	3066, 3244	3041, 3173, 3400, 3480	3176, 3459

* The data from [12].

and HL^{1}Na (Table 3). The presence of crystal water in complexes **I** and **II** complicates the spectral pattern in the 3500–3050 cm^{-1} range. The IR spectroscopic data for complexes **I** and **II** are consistent with the X-ray diffraction data for the coordination polymer [Cu(HL)Cl]$_n$ in structure **IV**. According to X-ray diffraction data, HL$^-$ acts as a chelating and bridging tetradentate ligand. Apparently, HL$^-$ and (HL^1)$^-$ in complexes **I** and **II** perform the same functions. The IR spectra of complexes **I** and **II** show new bands in the 500–100 cm^{-1} range, which are absent from the spectra of HLNa and HL^{1}Na. This provides evidence for the formation of Cu–ligand bonds. The two-component bands at 314 and 286 (**I**) and 300 and 286 cm^{-1} (**II**) are due to the ν(Cu–Cl) vibrations. The splitting of ν(Cu–Cl) band can be attributed to the participation of the Cl atom in hydrogen bonding. The assignment of this band was based on the X-ray diffraction data for complex **IV** as well as on the IR spectra of structurally characterized Cu(II) complexes with α-amino oximes derived from monoterpenoids [16, 17]. The coordination of the N atoms of the NH and NOH groups and the O atoms of the COO$^-$ group to the Cu atom was confirmed by the presence of new intense bands at 502–351 cm^{-1} in the spectra of complexes **I** and **II**. However, the ν(Cu–O) and ν(Cu–N) bands are difficult to identify for these complexes.

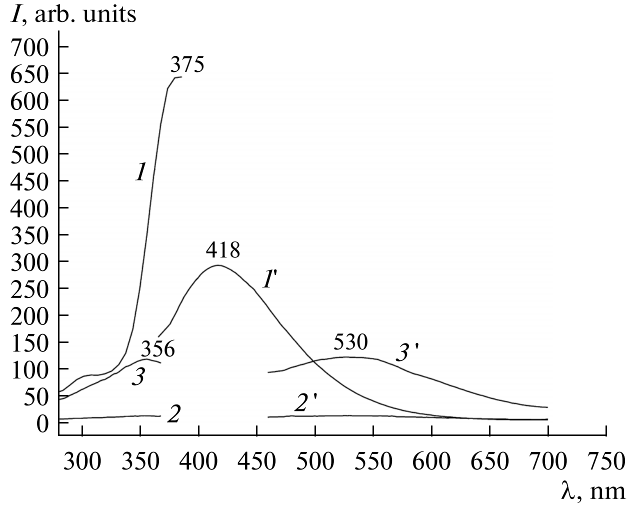


Fig. 2. Excitation (*1–3*) and emission spectra (*1'–3'*) of HLNa (*1, 1'*) and complex **III**: *V* = (*2, 2'*) 600 and (*3, 3'*) 800 V.

The IR spectrum of complex **III** shows the ν(NO) band at 943 cm^{-1} against its position at 945 cm^{-1} for HLNa. The frequency of this band remains virtually constant (Table 3), suggesting that the oxime N atom is not coordinated by the Ag$^+$ ion. The slight shifts of the ν(NH) and ν(OH) bands in the spectrum of complex **III** against their positions in the spectrum of HLNa (Table 3) may be due to the changed hydrogen bonding pattern as well as to the presence of crystal water in this complex. The bands ν_{as}(COO$^-$) and ν_s(COO$^-$) are shifted in the spectrum of complex **III** in opposite directions against their positions in the spectrum of HLNa. Apparently, this is the case of the chelating bidentate coordination of the COO$^-$ group to the Ag$^+$ ions [14]. This assumption is confirmed by the smallest $\Delta\nu$(COO$^-$) value for complex **III** among the other complexes listed in Table 3 [14, 15]. The low-frequency regions in the spectra of compounds **III** and HLNa are similar, which suggests the predominantly ionic Ag–O bonding.

Based on the data from IR spectroscopy, we can assume that complexes **I** and **II** are polynuclear structures like the coordination polymer **IV**. In complex **III**, the first coordination sphere of the Ag atom is made up of the O atoms of the chelating bidentate COO$^-$ group.

The excitation spectrum of HLNa in the range from 250 to 400 nm shows a band with an intense narrow maximum (λ_{max} = 375 nm). Complex **III** can be excited by UV radiation in the same range, though its spectrum features a wider and weak band ($\lambda_{max} \approx$ 356 nm). For this reason, we recorded emission spectra using λ_{ex} = 350 nm (Fig. 2). The salt HLNa produces blue photoluminescence in the range from 370 to 520 nm with λ_{max} = 418 nm. Complex III luminesces from 470 to 580 nm with λ_{max} = 530 nm. Clearly, the complexation causes a strong bathochromic shift of the emission peak, thus changing the initial blue luminescence of HLNa to the green luminescence of complex **III**. At the same time, the largely quenched emission of the latter suggests that the coordination of the ligands to the Ag$^+$ ions affects the photoluminescence process.

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