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Nitrosoruthenium Hydroxo and Aqua Complexes of the *trans*-Dipyridine Series: Synthesis, Structures, and Characterization

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Abstract—A procedure for the synthesis of *trans*-$Ru(NO)(Py)_2Cl_2(OH)$ (**I**) from $K_2[Ru(NO)Cl_5]$ was proposed. Treatment of hydroxo complex **I** with HCl or H_2SO_4 at room temperature gave the corresponding salts *trans*-$[Ru(NO)(Py)_2Cl_2(H_2O)]Cl \cdot 2H_2O$ (**II**) and *trans*-$[Ru(NO)(Py)_2Cl_2(H_2O)]HSO_4$ (**III**). All the complexes obtained were characterized by ^1H and ^{13}C NMR and IR spectroscopy and elemental analysis; their structures were determined by X-ray diffraction. The structures are stabilized by π-stacking between the pyridine ligands of adjacent complex species.

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INTRODUCTION

In the chemistry of nitrosoruthenium complexes, much research is currently devoted to long-lived photoinduced metastable isomers with different ways of coordinating the nitroso group to the ruthenium atom [1–3]. In a stable state, the NO group is coordinated to the transition metal through the N atom. Exposure of the starting nitroso complex to a laser radiation produces isomers in which NO is coordinated through either the O atom (MS1 state) or simultaneously the O and N atoms (MS2). Reversible photoisomerization offers scope for the synthesis of hybrid materials combining, in the same crystal lattice, photochromism, conductance, magnetism, special optical properties, etc. [4, 5].

Among a variety of currently known nitroso complexes of ruthenium, the highest population of the metastable states has been noted for the pyridine complex $[Ru(NO)(Py)_4Cl](PF_6)_2 \cdot 0.5H_2O$ [6, 7]; its crystal structure has been determined. Photoisomerization of the nitrosoruthenium complexes containing fewer than four inner-sphere pyridine molecules has not been studied, probably because of very scarce data on the structures of, and synthetic routes to, these complexes.

The first example of nitroso complexes of ruthenium of the dipyridine series, $[Ru(NO)(Py)_2Cl(C_2O_4)]$, has been obtained in low yield by heating $K_2[Ru(NO)Cl(C_2O_4)_2]$ with a double amount of pyridine [8]. Reflux of this dipyridine complex with concentrated HCl gives $[Ru(NO)Py_2Cl_2(H_2O)]Cl \cdot 2H_2O$ as red needle-like crystals [8]. The synthesis of the trichlorodipyridine complex $[Ru(NO)(Py)_2Cl_3]$ in ≤30% yield has also been reported [9, 10]. The molecular formulas of these three complexes were determined by elemental analysis and IR spectroscopy but their crystal structures remain unclear.

The goal of this study was to obtain the nitrosoruthenium hydroxo and aqua complexes of the dipyridine series *trans*-$[Ru(NO)(Py)_2Cl_2(OH)]$ (**I**), *trans*-$[Ru(NO)Py_2Cl_2(H_2O)]Cl \cdot 2H_2O$ (**II**), and *trans*-$[Ru(NO)Py_2Cl_2(H_2O)]HSO_4$ (**III**), characterize them by spectroscopic methods, and determine their crystal structures by X-ray diffraction.

EXPERIMENTAL

The starting reagent $K_2[Ru(NO)Cl_5]$ was synthesized in ~97% yield from commercial ruthenium trichloride hydrate as described in [11]. Other reagents and solvents (reagent grade or higher) were used without further purification.

IR spectra (KBr pellets) were recorded on a Scimitar FTS 2000 FTIR spectrometer in the 4000–375 cm^{-1} range. NMR spectra were recorded on a Bruker Avance III spectrometer (500.0 (^1H) and 125.7 MHz (^{13}C)) in DMSO. Analysis for C, H, N, and S was carried out on a EURO EA3000 analyzer; the Cl content was determined using the Schöniger flask test [12].

X-ray diffraction studies of complexes **I–III** were carried out on a DRON-3M diffractometer (R = 192 mm, CuK_α radiation, Ni filter, scintillation detec-

tor with amplitude discrimination, $2\theta = 5°–50°$) at room temperature. Samples were applied in thin films to the smooth side of a standard quartz cell. Complete indexing of the single-crystal X-ray diffraction patterns of the complexes obtained confirms their single-phase structures.

The unit cell parameters and experimental reflection intensities for solving crystal structures were measured at 150 K on a Bruker X8 Apex CCD automated diffractometer (MoK_α radiation). The structures were solved by the heavy atom method and refined anisotropically by the full-matrix least-squares method. The hydrogen atoms were located geometrically in idealized positions and refined isotropically. All calculations were performed with the SHELX-97 program package [13].

Selected crystallographic parameters and the data collection and refinement statistics for structures **I**–**III** are given in Table 1. The comprehensive crystallographic data for structures **I**, **II**, and **III** have been deposited with the Cambridge Crystallographic Data Centre (nos. 841640, 924080, and 924081, respectively; http://www.ccdc.cam.ac.uk/data_request/cif).

Synthesis of complex I. The complex $K_2[Ru(NO)Cl_5]$ (0.5 g, 1.3 mmol) was placed in a beaker. Then a water–ethanol mixture (1 : 1 v/v, ~10 mL), pyridine (~0.25 mL, 3.1 mmol), and $KHCO_3$ (~0.15 g, 1.5 mmol) were added. The beaker was covered with a watch glass, and the reaction mixture was stirred with a magnetic stirring bar at 65–70°C for ~30 min. Then the watch glass was removed, and the resulting solution was concentrated with stirring to a minimum volume (~3 mL) and cooled to room temperature. The orange precipitate that formed was filtered off on a porous glass filter and washed with water (~5 mL), ethanol, and acetone. The yield was ~50%.

Single crystals of complex **I** suitable for X-ray diffraction were obtained by slow evaporation of its saturated solution in DMF.

For $C_{10}H_{11}N_3O_2Cl_2Ru$

anal. calcd., %:	C, 31.84;	H, 2.94;	N, 11.14;	Cl, 18.80.
Found, %:	C, 31.83;	H, 2.98;	N, 11.11;	Cl, 18.72.

IR (ν, cm^{-1}): 3540 ν(OH); 3100–3000 ν(CH); 1817 ν(NO); 1606, 1570, 1482, 1451, 1348 ν(C_{arom}–C_{arom}), ν(C_{arom}–N_{arom}); 1242, 1208, 1067, 1018 δ($CH_{in\text{-}plane}$); 913 δ(RuOH); 763, 690, 647 δ($CH_{out\text{-}of\text{-}plane}$); 605 ν(Ru–$N_{NO}$), δ(Ru–NO); 574 ν(Ru–$O_{OH}$); 465 ν(Ru–$N_{Py}$).

^{1}H NMR (δ, ppm): 9.21 (d, 4H, H(2), H(6)); 8.45 (t, 2H, H(4)); 8.02 (t, 4H, H(3), H(5)).

^{13}C NMR (δ, ppm): 153.23 (C(2), C(6)); 140.35 (C(4)); 125.65 (C(3), C(5)).

Synthesis of complex II. An orange powder of hydroxo complex **I** (~0.25 g, 0.66 mmol) was treated on a porous glass filter with concentrated HCl (~1 mL) at room temperature. The resulting red-wine precipitate was washed with ethanol (~5 mL) and diethyl ether (~5 mL). The yield was nearly quantitative.

Single crystals of complex **II** suitable for X-ray diffraction were obtained by slow evaporation of its saturated solution in 6 M HCl.

For $C_{10}H_{16}N_3O_4Cl_3Ru$

anal. calcd., %:	C, 27.61;	H, 3.59;	N, 9.34;	Cl, 23.65.
Found, %:	C, 27.11;	H, 3.65;	N, 9.32;	Cl, 23.61.

IR (ν, cm^{-1}): 3330 ν(H_2O); 3150–3030 ν(CH); 1897 ν(NO); 1605, 1570, 1484, 1450, 1356 ν(C_{arom}–C_{arom}), ν(C_{arom}–N_{arom}); 1242, 1211, 1159, 1067, 1051, 1017, 981 δ($CH_{in\text{-}plane}$); 869, 816 δ(H_2O_{coord}); 762, 689, 650, 634 δ($CH_{out\text{-}of\text{-}plane}$); 615 ν(Ru–$N_{NO}$), δ(Ru–NO); 484 ν(Ru–O); 455 ν(Ru–N_{Py}).

^{1}H NMR (δ, ppm): 9.38 (d, 4H, H(2), H(6)); 8.73 (t, 2H, H(4)); 8.29 (t, 4H, H(3), H(5)).

^{13}C NMR (δ, ppm): 152.47 (C(2), C(6)); 140.83 (C(4)); 125.84 (C(3), C(5)).

Synthesis of complex III was carried out as described for complex **II**. A small amount (3–4 drops) of concentrated H_2SO_4 was added to an orange powder of hydroxo complex **I** (~0.25 g, 0.66 mmol) on a porous glass filter. The resulting red-wine precipitate was washed with diethyl ether (2 ×5 mL). The yield was nearly quantitative.

For $C_{10}H_{13}N_3O_6SCl_2Ru$

anal. calcd., %:	C, 25.27;	H, 2.76;	N, 8.84;	S, 6.75.
Found, %:	C, 25.21;	H, 2.45;	N, 8.32;	S, 6.73.

IR (ν, cm^{-1}): 3340 ν(H_2O); 3150–3030 ν(CH); 1904 ν(NO); 1607, 1488, 1452, 1362 ν(C_{arom}–C_{arom}), ν(C_{arom}–N_{arom}); 1313, 1187 ν(HSO_4); 1229, 1157, 1070, 1038, 1015, 976 δ($CH_{in\text{-}plane}$); 881, 859 δ(H_2O_{coord}); 762, 690, 650 δ($CH_{out\text{-}of\text{-}plane}$); 605 ν(Ru–$N_{NO}$), δ(Ru–NO); 594, 572 δ($HSO_4$); 484 ν(Ru–O); 455, 435 ν(Ru–N_{Py}).

RESULTS AND DISCUSSION

Potassium (pentachloro)(nitroso)ruthenate was used as a ruthenium source because this complex salt can easily be prepared in high yield from commercially available $RuCl_3 \cdot xH_2O$ [11]. When heated in aqueous ethanol, the anion $[Ru(NO)Cl_5]^{2-}$ undergoes aquation to give a weakly acidic aquachloro complex ($pK_a \approx 6.0$) [14, 15]:

$$[Ru(NO)Cl_5]^{2-} + H_2O \rightleftharpoons [Ru(NO)Cl_4(H_2O)]^- + Cl^-,$$

$$[Ru(NO)Cl_4(H_2O)]^- \rightleftharpoons [Ru(NO)Cl_4(OH)]^{2-} + H^+.$$

To scavenge the resulting H^+ species, we added $KHCO_3$ to the reaction mixture. The use of excess pyridine, as well as experiments without addition of $KHCO_3$, fails. The reaction mixture becomes a viscous

Table 1. Crystallographic parameters and the data collection and refinement statistics for structures **I–III**

Parameter	Value		
	I	**II**	**III**
M	377.19	449.68	475.26
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$	$P2_1/c$
a, Å	7.0976(4)	13.5861(10)	10.1383(5)
b, Å	7.9259(5)	14.3235(10)	20.1069(10)
c, Å	12.6906(6)	8.5786(5)	8.2298(4)
α, deg	80.912(2)	90	90
β, deg	76.504(2)	90.163(4)	96.5360(10)
γ, deg	66.977(2)	90	90
V, Å^3	637.12(6)	1669.4(2)	1666.74(14)
Z	2	4	4
ρ_{calcd}, g/cm^3	1.966	1.789	1.894
μ, mm^{-1}	1.645	1.435	1.417
F(000)	372	896	944
Crystal dimensions, mm	0.20 × 0.10 × 0.05	0.45 × 0.15 × 0.12	0.40 × 0.28 × 0.26
θ scan range, deg	2.80–30.51	2.84–30.59	2.02–31.72
Ranges of h, k, and l indices	$-10 \le h \le 10$, $-11 \le k \le 11$, $-17 \le l \le 16$	$-19 \le h \le 19$, $-20 \le k \le 20$, $-7 \le l \le 12$	$-14 \le h \le 14$, $-29 \le k \le 29$, $-12 \le l \le 11$
Number of measured reflections	6243	12390	18711
Number of unique reflections	3779	2563	5533
R_{int}	0.0132	0.0302	0.0113
Completeness of data collection, %	98.6 (θ = 25.00)	99.9 (θ = 25.00)	99.2 (θ = 25.25)
Number of parameters refined	164	108	217
GOOF on F^2	1.075	1.091	1.172
R factor ($I > 2\sigma(I)$)	$R_1 = 0.0274$ $wR_2 = 0.0673$	$R_1 = 0.0202$, $wR_2 = 0.0482$	$R_1 = 0.0265$, $wR_2 = 0.0622$
R factor for all reflections	$R_1 = 0.0349$ $wR_2 = 0.0694$	$R_1 = 0.0240$, $wR_2 = 0.0497$	$R_1 = 0.0293$, $wR_2 = 0.0635$
$\Delta\rho_{max}$, $\Delta\rho_{min}$, e Å^{-3}	1.037, −1.082	0.509, −0.469	1.308, −1.063

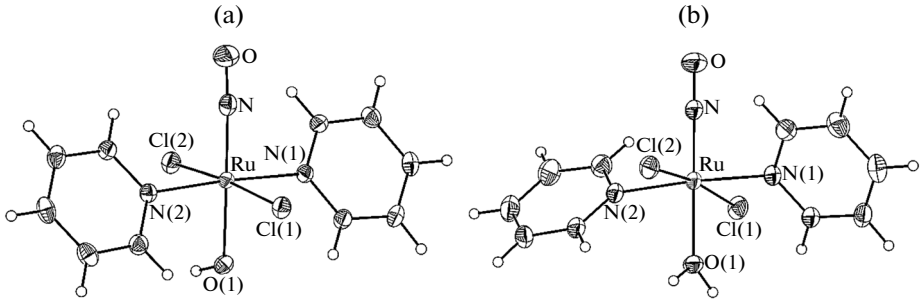

Fig. 1. Structures of the complex species: (a) *trans*-$[Ru(NO)(Py)_2Cl_2(OH)]$ (in **I**) and (b) *trans*-$[Ru(NO)(Py)_2Cl_2(H_2O)]^+$ (in **II** and **III**).

brown solution probably containing polymeric forms of nitrosoruthenium complexes with bridging hydroxide ions. The formation of such polymeric hydroxo complexes has already been observed in the synthesis of ammine complexes [16, 17].

The net equation for the formation of hydroxo complex **I** can be written as follows:

$$[Ru(NO)Cl_5]^{2-} + 2Py + HCO_3^- \longrightarrow [Ru(NO)Py_2Cl_2(OH)]\downarrow + 3Cl^- + CO_2\uparrow.$$

Room-temperature reactions of hydroxo complex **I** with concentrated HCl and H_2SO_4 results in protonation of the coordinated hydroxide ion, yielding the corresponding salts of aqua complexes **II** and **III**.

The IR spectra of the complexes obtained show intense bands due to the ν(NO) stretching vibrations: 1817 (**I**), 1897 (**II**), and 1904 cm^{-1} (**III**). These values fall within a range characteristic of most nitroso complexes of ruthenium that combine the diamagnetic metal center Ru(II) and the linearly coordinated species NO^+ [18–20]. Note that the ν(NO) frequency depends on the ligand that is *trans* to the nitroso group and is shifted to the shorter wavelengths when moving from the neutral hydroxo complex **I** to the cationic aqua complexes **II** and **III**.

Apart from the ν(NO) bands, the IR spectra of complexes **I–III** contain bands characteristic of coordinated pyridine molecules [21, 22]: narrow bands of medium and low intensities at 3100–3000 cm^{-1} (ν(CH)), narrow bands of medium and high intensities at 1607–1348 cm^{-1} (ν(C_{arom}–C_{arom}), ν(C_{arom}–N_{arom})), and medium- and high-intensity bands at 1242–976 and 763–634 cm^{-1} (δ($CH_{in\text{-}plane}$) and δ($CH_{out\text{-}of\text{-}plane}$), respectively).

In addition, the IR spectra of these complexes show medium- and low-intensity bands due to the inner-sphere hydroxide ion (**I**) or the inner-sphere water molecules (**II**). Note that these bands are broadened in the spectra of the aqua complexes, which can be explained by the presence of larger networks of hydrogen bonds in their structures.

The ^1H and ^{13}C NMR spectra of complexes **I–III** contain three groups of signals for the ^1H and ^{13}C nuclei in the *ortho*-, *meta*-, and *para*-positions of the coordinated pyridine molecules. The chemical shifts of these signals strongly depend on both the substituent in the *trans*-position and the solvent. For nitroso complexes of ruthenium, the δ(^1H) values are in the 7–10 ppm range [9, 23, 24] and the δ(^{13}C) values are in the 160–120 ppm range [25, 26].

The crystal structure of complex **I** is built from neutral complex species *trans*-$[Ru(NO)(Py)_2Cl_2(OH)]$; structures **II** and **III** are made up of complex cations *trans*$[Ru(NO)Py_2Cl_2(H_2O)]^+$ and outer-sphere Cl^- (in **II**) and HSO_4^- anions (in **III**). Complex **II** also contains molecules of crystallization water. The structures of the complex species in **I–III** with atomic numbering and thermal displacement ellipsoids are shown in Fig. 1. Selected bond lengths and bond angles are listed in Table 2.

In all the three structures studied, the coordination polyhedra of the Ru atoms are slightly distorted octahedra (RuN_3Cl_2O). In structure **II**, the complex cation is on the axis 2. The bond angles at the Ru atoms deviate from 90° by at most 4.5°.

The pyridine molecules in complexes **I–III** are always *trans* to each other; the square environment in the equatorial plane is completed with two chloride ions. The Ru–N(Py) bond lengths are ~2.10 Å. The Ru–Cl bonds in neutral complex **I** (2.379 Å) are longer by ~0.030 Å than those in aqua complexes **II** and **III**. These values fall within a bond length range characteristic of nitrosoruthenium aminochloro complexes [9, 10, 27].

The axial positions in complex **I** are occupied by the nitroso group and hydroxide ion; in complexes **II** and **III**, it is the water molecules that are *trans* to the nitroso group. The Ru–O(1) bond in hydroxo complex **I** (1.971 Å) is shorter than those in aqua complexes **II** and **III** (on average, 2.031 Å). The geometrical parameters of the fragment $(RuNO)^{3+}$ in complexes **I–III** agree well with the literature data [10, 28, 29]: the N–O and Ru–N(NO) bond lengths are nearly 1.14 and 1.73 Å, respectively; the ONRu angles

Table 2. Selected bond lengths and bond angles in structures **I–III**

Bond	*d*, Å		
	I	**II**	**III**
N–O	1.125(3)	1.145(2)	1.141(2)
Ru–N	1.755(2)	1.7190(17)	1.7283(16)
Ru–O(1)	1.9707(16)	2.0247(13)	2.0368(14)
Ru–N(1)	2.1061(19)	2.0994(13)	2.0991(16)
Ru–N(2)	2.0959(19)	2.0994(13)	2.1042(16)
Ru–Cl(1)	2.3790(6)	2.3507(4)	2.3495(5)
Ru–Cl(2)	2.3790(6)	2.3507(4)	2.3484(5)
Angle	ω, deg		
	I	**II**	**III**
ONRu	175.4(2)	180	179.25(18)
NRuO(1)	179.27(8)	180	179.36(7)
NRuN(1)	91.88(8)	92.19(3)	91.41(7)
NRuN(2)	91.96(8)	92.19(3)	91.73(7)
O(1)RuN(1)	88.50(7)	87.81(3)	89.22(6)
O(1)RuN(2)	87.66(7)	87.81(3)	87.65(6)
N(1)RuN(2)	176.15(7)	175.63(6)	176.47(6)
NRuCl(1)	93.64(7)	93.993(9)	94.38(6)
NRuCl(2)	91.95(7)	93.993(9)	93.85(6)
O(1)RuCl(1)	86.98(5)	86.007(9)	85.47(4)
O(1)RuCl(2)	87.42(5)	86.007(9)	86.30(4)
N(1)RuCl(1)	89.82(5)	90.56(3)	92.05(5)
N(1)RuCl(2)	89.83(5)	89.13(3)	87.56(5)
N(2)RuCl(1)	89.64(5)	89.13(3)	89.32(4)
N(2)RuCl(2)	90.34(5)	90.56(3)	90.62(4)
Cl(2)RuCl(1)	174.40(2)	172.014(19)	171.766(19)

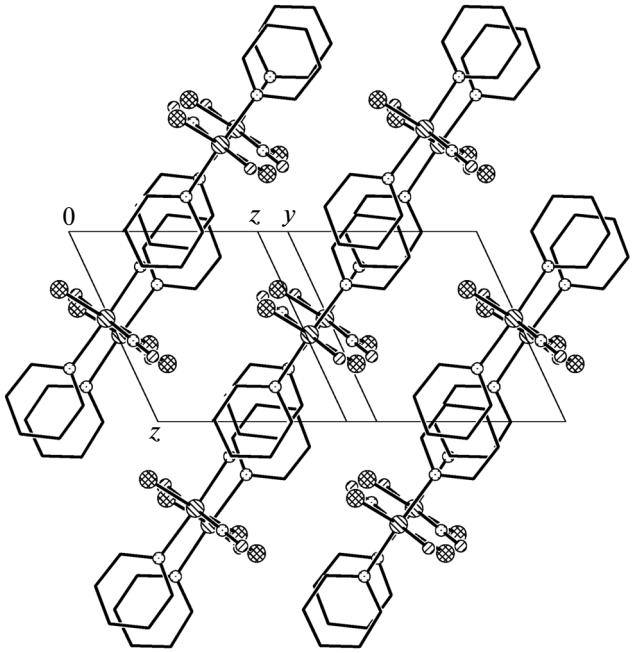

Fig. 2. Fragment of the crystal structure of complex **I**.

approximate to 180°. The central Ru atom in all the complex species deviates from the equatorial plane by ~0.1 Å toward the nitroso group.

In the coordinated pyridine molecules, the average N–C and C–C bond lengths have standard values (~1.35 and 1.38 Å, respectively). In neutral complex **I**, the planes of the pyridine rings are nearly parallel. The angle between the normals to their planes is 10°, and the angles between the planes of the pyridine rings and the equatorial plane are 61° and 57°. In aqua complexes **II** and **III**, the planes of the pyridine rings make with each other angles of 103° and 98°, respectively. The angles between their planes and the equatorial plane are 52° and 49°.

In the hydrosulfate ion of complex **III**, the S–OH bond length is 1.561 Å; the other three S–O bonds are close in length with each other (on average, ~1.45 Å). The OSO angles vary from 103° to 114°. The anions are united through the hydrogen bonds O–H···O (2.58 Å) into infinite chains along the axis *z*; the S···S distances are 4.42 Å.

The general views of the packing patterns in the crystals of complexes **I–III** are shown in Figs. 2 and 3. In structure **I**, the complex molecules form layers perpendicular to the direction [011]. Within these layers, the pyridine molecules of adjacent complexes are linked by π-stacking ($d \approx 3.3–3.5$ Å). Structure **I** also shows short contacts C–H···Cl (~2.9 Å) and O···H–C (~2.5 Å). The shortest four Ru···Ru distances are 6.787–7.092 Å.

In complex **II**, the complex cations and outer-sphere chloride ions are linked by molecules of crystallization water. Each of its molecules is bound to two chloride anions (O(*w*)···Cl 3.10 and 3.13 Å) and to the coordinated water molecule (O(*w*)···O(*w*) 2.59 Å). In the crystal of complex **III**, the complex cations are linked to the hydrosulfate ions by hydrogen bonds involving the coordinated water molecule and the O atoms of the HSO_4^- (O(*w*)···O 2.60 and 2.65 Å). The hydrogen bonding patterns in structures **II** and **III** are shown in Fig. 3. The complex cations in these structures are aligned with the axis *z*; the shortest Ru···Ru distances are 5.812 Å (**II**) and 5.651Å (**III**). Channels

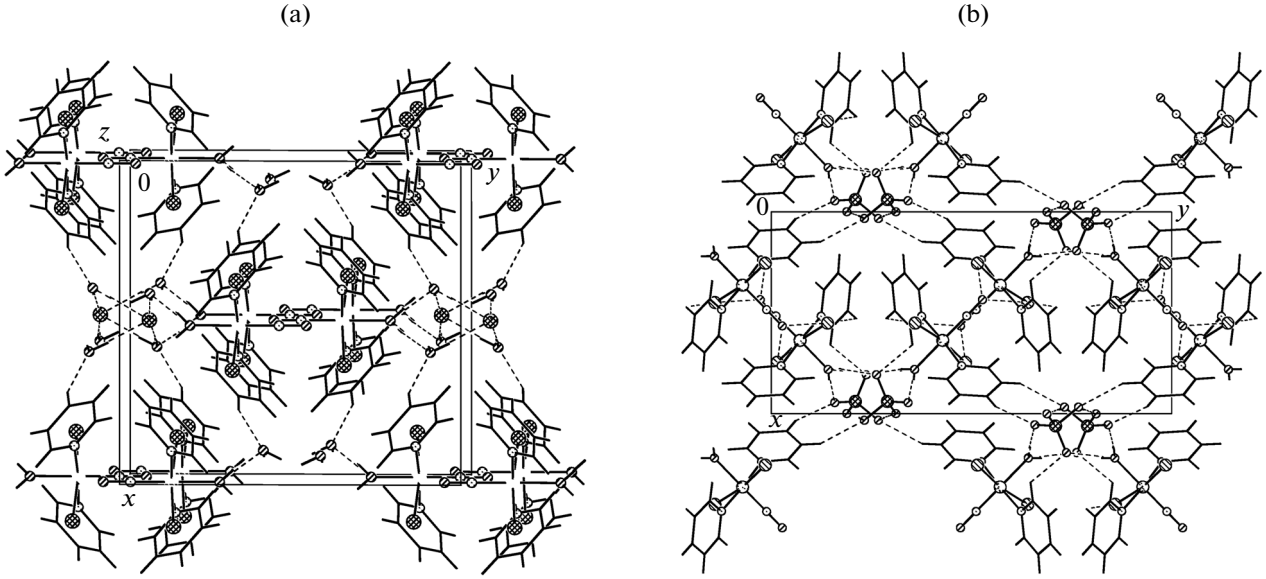

Fig. 3. Fragments of the crystal structures of complexes **II** (a) and **III** (b).

between the complex cations accommodate outer-sphere anions and crystallization water molecules.

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