

# Some Transformations of *trans*-Tetrapyridine Complexes of Nitrosoruthenium: Crystal Structures of $[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})](\text{PF}_6)_2 \cdot (\text{CH}_3)_2\text{CO}$ and $[\text{H}_5\text{O}_2]_2[\text{Ru}(\text{NO})\text{Py}_4\text{Cl}]\text{Cl}_4$

A. N. Makhinya<sup>a, b</sup>, M. A. Il'in<sup>a, b, \*</sup>, I. V. Korol'kov<sup>a, b</sup>, and I. A. Baidina<sup>a</sup>

<sup>a</sup>Nikolaev Institute of Inorganic Chemistry, Siberian Branch, Russian Academy of Sciences, Novosibirsk, 630090 Russia

<sup>b</sup>Novosibirsk State University, Novosibirsk, 630090 Russia

\*e-mail: ima@niic.nsc.ru

Received December 11, 2017

**Abstract**—The slow evaporation of an acetone solution containing *trans*- $[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})]^{2+}$  cations and hexafluorophosphate anions results in the crystallization of *trans*- $[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})](\text{PF}_6)_2 \cdot (\text{CH}_3)_2\text{CO}$  (I). The reactions of *trans*- $[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})]\text{Cl}_2 \cdot \text{H}_2\text{O}$  with solutions of chloric or hydrochloric acid followed by the evaporation of the reaction solutions at ambient temperature afford *trans*- $[\text{Ru}(\text{NO})\text{Py}_4(\text{H}_2\text{O})](\text{ClO}_4)_3$  (II) or  $[\text{H}_5\text{O}_2]_2[\text{Ru}(\text{NO})\text{Py}_4\text{Cl}]\text{Cl}_4$  (III), respectively. The obtained chloride complex III is unstable and at ambient temperature eliminates hydrogen chloride to transform into *trans*- $[\text{Ru}(\text{NO})\text{Py}_4\text{Cl}]\text{Cl}_2 \cdot 4\text{H}_2\text{O}$  (IV). The crystal structures of compounds I and III are determined by X-ray structure analysis (CIF files CCDC nos. 1421042 (I) and 1421041 (III)).

**Keywords:** nitrosoruthenium complexes, amino complexes, pyridine, chloride complexes, hydroxo complexes, X-ray structure analysis, IR spectroscopy

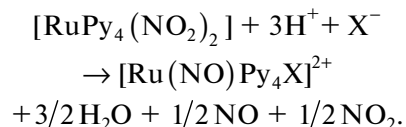
**DOI:** 10.1134/S1070328418100093

## INTRODUCTION

Interest of researchers in the nitrosoruthenium complexes is caused by two main factors. On the one hand, these compounds are biologically active and considered as low-toxicity regulators of the nitro-gen(II) oxide concentration in biological systems [1, 2]. On the other hand, the nitrosoruthenium complexes are capable of forming two photoinduced long-lived metastable states (MS1 and MS2) differed in the coordination mode of the nitroso group to the central atom [3–5]. A phenomenon of this photoisomerization provides prospects for the synthesis of hybrid materials combining the photochromic properties with electrical conductance or magnetism [6, 7]. Among the presently known nitrosoruthenium complexes, the tetrapyridine complex  $[\text{Ru}(\text{NO})\text{Py}_4\text{Cl}](\text{PF}_6)_2 \cdot 0.5\text{H}_2\text{O}$  has the record-breaking high degree of transition to the metastable states MS1 and MS2 (92 and 48%, respectively) [8, 9].

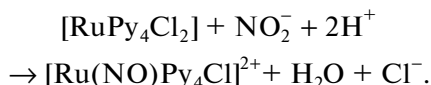
Several approaches to the synthesis of the tetrapyridinenitrosoruthenium complexes are presented in the literature. One of them [10] makes it possible to obtain the halide complexes  $[\text{Ru}(\text{NO})\text{Py}_4\text{X}]^{2+}$  (X = Cl, Br)

and consists of the treatment of the corresponding *trans*-dinitro complex with hydrogen halide acid



Only compounds with  $\text{ClO}_4^-$  or  $\text{PF}_6^-$  anions in the external sphere can be isolated to the solid phase from aqueous solutions in high yields (70–85%). The hydroxo complexes  $[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})](\text{ClO}_4)_2$  or  $[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})](\text{PF}_6)_2$  can be obtained using chloric [11] or trifluoroacetic [12] acid in the synthesis.

Somewhat modified method for the synthesis of the nitrosotetrapyridine complexes is based on the nitrosylation of the dichlorotetrapyridine complexes with sodium nitrite in a hydrochloric acid medium [12]



Another approach to the synthesis of the tetrapyridine complex was described [13]. The *trans*-

[Ru(NO)Py<sub>4</sub>(OH)]Cl<sub>2</sub> · H<sub>2</sub>O complex is obtained in a yield of ~70% by reflux of *fac*-[Ru(NO)Py<sub>2</sub>Cl<sub>3</sub>] with an excess of an aqueous solution of pyridine.

In this work, we consider the transformations of the hydroxotetrapyridine complex [Ru(NO)Py<sub>4</sub>(OH)]<sup>2+</sup> in an acidic medium, proposed the procedures for the synthesis of the chloro- and aquanitrosoruthenium complexes of the tetrapyridine series, and determined the crystal structures of *trans*-[Ru(NO)Py<sub>4</sub>(OH)](PF<sub>6</sub>)<sub>2</sub> · (CH<sub>3</sub>)<sub>2</sub>CO (**I**) and *trans*-[H<sub>5</sub>O<sub>2</sub>]<sub>2</sub>[Ru(NO)Py<sub>4</sub>Cl]Cl<sub>4</sub> (**III**).

## EXPERIMENTAL

The starting *trans*-[Ru(NO)Py<sub>4</sub>(OH)]Cl<sub>2</sub> · H<sub>2</sub>O compound was synthesized using a known procedure [13]. Other reagents and solvents were not lower than reagent grade and were used without additional purification.

**Synthesis of complex I.** A weighed sample of *trans*-[Ru(NO)Py<sub>4</sub>(OH)]Cl<sub>2</sub> · H<sub>2</sub>O (~0.5 g, 9.0 × 10<sup>-4</sup> mol) was dissolved in a minimum amount of water (~10 mL), and a 10% excess of an aqueous solution of NH<sub>4</sub>PF<sub>6</sub> was added. The precipitate formed was filtered off on a glass porous filter (40 pores), washed with ethanol and diethyl ether, and dried in an air flow. The yield of *trans*-Ru(NO)Py<sub>4</sub>(OH)(PF<sub>6</sub>)<sub>2</sub> · H<sub>2</sub>O was ~95%. The compound was highly soluble in acetone from which crystals of the solvate of compound **I** suitable for X-ray structure analysis were obtained by slow evaporation.

IR (ν, cm<sup>-1</sup>): 3580, 3395 ν(OH); 3127–2820 ν(CH); 1867 ν(NO); 1715 ν(CO); 1612, 1576, 1491, 1454, 1406, 1364 ν(C<sub>arom</sub>–C<sub>arom</sub>), ν(C<sub>arom</sub>–N<sub>arom</sub>); 1246, 1223, 1163, 1072, 1020 δ(CH<sub>pl</sub>); 974, 835 ν(Ru–OH); 756, 691, 652 δ(CH<sub>out-of-pl</sub>); 637, 556 ν(Ru–N<sub>NO</sub>), δ(Ru–NO); 459 ν(Ru–N<sub>Py</sub>).

**Synthesis of *trans*-[Ru(NO)Py<sub>4</sub>(H<sub>2</sub>O)](ClO<sub>4</sub>)<sub>3</sub> (II).** Water (~20 mL) and concentrated HClO<sub>4</sub> (~1 mL) were added to a weighed sample of *trans*-[Ru(NO)Py<sub>4</sub>(OH)]Cl<sub>2</sub> · H<sub>2</sub>O (~0.1 g, 1.8 × 10<sup>-4</sup> mol). The obtained solution was evaporated at ambient temperature to a minimum volume (~2 mL). The precipitate formed was filtered off on a glass porous filter (40 pores) and washed with cooled ethanol (~1 mL) and with the same amount of diethyl ether. The yield of compound **II** was ~50%.

IR (ν, cm<sup>-1</sup>): 3450 ν(H<sub>2</sub>O); 3120–3050 ν(CH); 2800, 2400 ν(OH···O); 1934 ν(NO); 1650 δ(H<sub>2</sub>O); 1612, 1492, 1454, 1365 ν(C<sub>arom</sub>–C<sub>arom</sub>), ν(C<sub>arom</sub>–N<sub>arom</sub>); 1227, 1018 δ(CH<sub>pl</sub>); 1097 ν<sub>3</sub>(ClO<sub>4</sub>); 928 δ(Ru–OH<sub>2</sub>); 762, 696 δ(CH<sub>out-of-pl</sub>); 623 ν<sub>4</sub>(ClO<sub>4</sub>); 452 ν(Ru–N<sub>Py</sub>).

**Synthesis of complex III.** A weighed sample of *trans*-[Ru(NO)Py<sub>4</sub>(OH)]Cl<sub>2</sub> · H<sub>2</sub>O (~0.1 g, 1.8 × 10<sup>-4</sup> mol)

was dissolved in ~1 mL of a 6 M solution of HCl, and the mixture was left to evaporate slowly at ambient temperature to form slightly wet crystals of complex **III** suitable for X-ray structure analysis.

IR (ν, cm<sup>-1</sup>): 3450, 3380, 3236, 2500, 2100 ν(H<sub>5</sub>O<sub>2</sub><sup>+</sup>); 3120–2800 ν(CH); 1921 ν(NO); 1729, 1641 ν(H<sub>5</sub>O<sub>2</sub><sup>+</sup>); 1610, 1491, 1451, 1364 ν(C<sub>arom</sub>–C<sub>arom</sub>), ν(C<sub>arom</sub>–N<sub>arom</sub>); 1274, 1242, 1221, 1159, 1125, 1071, 1017 δ(CH<sub>pl</sub>); 880 δ(H<sub>5</sub>O<sub>2</sub><sup>+</sup>); 765, 698, 651 δ(CH<sub>out-of-pl</sub>); 600 ν(Ru–N<sub>NO</sub>), δ(R–NO); 452 ν(R–N<sub>Py</sub>).

### Synthesis of *trans*-[Ru(NO)Py<sub>4</sub>Cl]Cl<sub>2</sub> · 4H<sub>2</sub>O (IV).

Hydrogen chloride was removed upon the complete drying of the crystals of compound **II** in air for several days at ambient temperature due to which compound **V** was formed in a quantitative yield.

IR (ν, cm<sup>-1</sup>): 3400 ν(H<sub>2</sub>O); 311–2800 ν(CH); 1921 ν(NO); 1641 δ(H<sub>2</sub>O); 1610, 1491, 1451, 1364 ν(C<sub>arom</sub>–C<sub>arom</sub>), ν(C<sub>arom</sub>–N<sub>arom</sub>); 1274, 1242, 1221, 1159, 1125, 1071, 1017 δ(CH<sub>pl</sub>); 765, 699, 651 δ(CH<sub>out-of-pl</sub>); 600 ν(R–N<sub>NO</sub>), δ(R–NO); 452 ν(R–N<sub>Py</sub>). The IR spectra of the samples in KBr pellets were recorded on a Scimitar FTS 2000 FTIR spectrometer in a range of 4000–375 cm<sup>-1</sup>.

The X-ray diffraction analysis of the powdered crystals was carried out on a Shimadzu XRD-7000 diffractometer (CuK<sub>α</sub> radiation, Ni filter, 2θ = 5°–60°). The samples were deposited as thin layers on the smooth side of a standard quartz cell.

The spectroscopic and diffraction data were processed using the OriginPro 7.5 program package [14].

### X-ray structure analyses of compounds I and III.

The unit cell parameters were determined and a set of experimental intensities was obtained on an X8 APEX Bruker automated diffractometer (MoK<sub>α</sub> radiation, graphite monochromator, two-coordinate CCD detector). The structures were solved by a direct method and refined in the anisotropic (isotropic for H atoms) approximation. The positions of all hydrogen atoms were localized from the difference synthesis. All calculations were performed using the SHELX-97 program package [15]. The crystallographic characteristics and the main refinement parameters for compounds **I** and **III** are presented in Table 1. Selected interatomic distances for compounds **I** and **III** are given in Tables 2 and 3, respectively. The bond angles in compound **III** are listed in Table 3.

The data on the structures of compounds **I** and **III** were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1421042 and 1421041, respectively; [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif)).

**Table 1.** Crystallographic data and experimental and structure refinement parameters for compounds **I** and **III**

Parameter	Value	
	<b>I</b>	<b>III</b>
<i>FW</i>	812.51	698.81
Crystal system	Orthorhombic	Monoclinic
Space group	<i>Pca</i> 2 <sub>1</sub>	<i>C2/c</i>
<i>a</i> , Å	18.4344(5)	23.6655(5)
<i>b</i> , Å	10.3909(3)	12.0859(3)
<i>c</i> , Å	34.1819(8)	10.7972(2)
β, deg		107.57(1)
<i>V</i> , Å <sup>3</sup>	6547.5(3)	2944.05(11)
<i>Z</i>	8	16
ρ <sub>calcd</sub> , g/cm <sup>3</sup>	1.648	1.577
μ, mm <sup>-1</sup>	0.679	1.024
<i>F</i> (000)	3248	1416
Crystal size, mm	0.25 × 0.22 × 0.18	0.22 × 0.16 × 0.12
Range of θ, deg	2.25–28.31	1.81–28.47
Ranges of reflection indices	–24 ≤ <i>h</i> ≤ 24, –13 ≤ <i>k</i> ≤ 12, –32 ≤ <i>l</i> ≤ 45	–31 ≤ <i>h</i> ≤ 31, –16 ≤ <i>k</i> ≤ 16, –7 ≤ <i>l</i> ≤ 14
Number of measured reflections	54226	10631
Number of independent reflections	15084	3581
<i>R</i> <sub>int</sub>	0.0410	0.0124
Completeness of data collection, %	99.8 (θ = 25.25)	97.8 (θ = 25.25)
Number of refined parameters	835	195
GOOF for <i>F</i> <sup>2</sup>	1.027	1.052
<i>R</i> factor ( <i>I</i> > 2σ( <i>I</i> ))	<i>R</i> <sub>1</sub> = 0.0409, <i>wR</i> <sub>2</sub> = 0.1027	<i>R</i> <sub>1</sub> = 0.0172, <i>wR</i> <sub>2</sub> = 0.0431
<i>R</i> factor (for all data)	<i>R</i> <sub>1</sub> = 0.0546, <i>wR</i> <sub>2</sub> = 0.1089	<i>R</i> <sub>1</sub> = 0.0198, <i>wR</i> <sub>2</sub> = 0.0441
Δρ <sub>max</sub> , Δρ <sub>min</sub> , e Å <sup>-3</sup>	0.598, –0.441	0.356, –0.413

**Table 2.** Selected interatomic distances in the structure of compound **I** and comparison with the data for *trans*-[Ru(NO)Py<sub>4</sub>(OH)](PF<sub>6</sub>)<sub>2</sub> · H<sub>2</sub>O [16]

Bond	<i>d</i> , Å		
	<b>IA</b>	<b>IB</b>	<i>trans</i> -[Ru(NO)Py <sub>4</sub> (OH)](PF <sub>6</sub> ) <sub>2</sub> · H <sub>2</sub> O [16]
N–O	1.138(5)	1.142(5)	1.145(4)
Ru–N <sub>NO</sub>	1.764(3)	1.760(3)	1.757(3)
Ru–N <sub>Py</sub> (av.)	2.11(2)	2.105(5)	2.103(3)
Ru–O <sub>OH</sub>	1.930(3)	1.955(3)	1.910(2)

## RESULTS AND DISCUSSION

The reaction of an aqueous solution of *trans*-[Ru(NO)Py<sub>4</sub>(OH)]Cl<sub>2</sub> · H<sub>2</sub>O with a minor excess of NH<sub>4</sub>PF<sub>6</sub> results in the precipitation of *trans*-

[Ru(NO)Py<sub>4</sub>(OH)](PF<sub>6</sub>)<sub>2</sub> · H<sub>2</sub>O in an almost quantitative yield. The structure of this compound was described earlier [16]. The slow evaporation of a solution of the compound in acetone affords crystals of

**Table 3.** Selected interatomic distances and bond angles in the structure of compound **III**

Bond	$d, \text{\AA}$	Angle	$\omega, \text{deg}$
N–O	1.124(2)	RuNO	180.0(2)
Ru–N <sub>NO</sub>	1.760(2)	N <sub>Py</sub> RuN <sub>Py</sub>	87.73(4)–92.30(4)
Ru–N <sub>Py</sub> (av.)	2.110(1)	N <sub>Py</sub> RuCl	87.05(3)–90.36(3)
Ru–Cl	2.3237(4)		

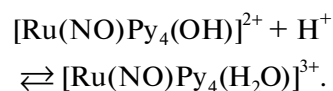
solvate **I**, whose structure was determined by X-ray structure analysis.

The geometries of *trans*-[Ru(NO)Py<sub>4</sub>(OH)]<sup>2+</sup> in compound **I** and in similar hydrate are close, but the structure of compound **I** contains complex species of two types (**A** containing the central atom Ru(1) and **B** containing Ru(2), Fig. 1). The interatomic distances (Table 2) in these complexes differ insignificantly and are consistent, as a whole, with the published data [16].

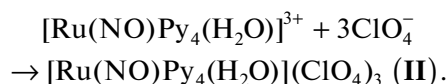
The coordination polyhedron of each complex **A** and **B** is a distorted octahedron formed by four nitrogen atoms of the pyridine molecules in the equatorial plane, nitrogen atoms of the nitroso groups, and oxygen of the hydroxide ion. The planes of the Py rings are arranged according to the “propeller” type at dihedral angles of 60°–70° to the equatorial planes. The non-equivalent complexes **A** and **B** are packed in crystal in such a way that their N–Ru–O axes are oppositely directed and the planes of the Py rings are parallel in pairs (two pyridine molecules are coaxial). The planes of the pyridine rings in complexes **A** and **B** deviate insignificantly (0.5°–2°) from the corresponding Ru–N axes, except for the coaxial Py ligands for which this deviation reaches 9° and 7°, respectively.

The ONRu bond angles for complexes **A** and **B** are 174.2° and 177.0°, and NRuO are 179.1° and 179.6°, respectively. The NRuN bond angles at the Ru(1) and Ru(2) atoms deviate from ideal values (90°) by 0.1°–2.8° (for **A**) and 0.9°–3.5° (for **B**). These values agree with the published data for the known nitrosoruthenium complexes of similar structure [17–20].

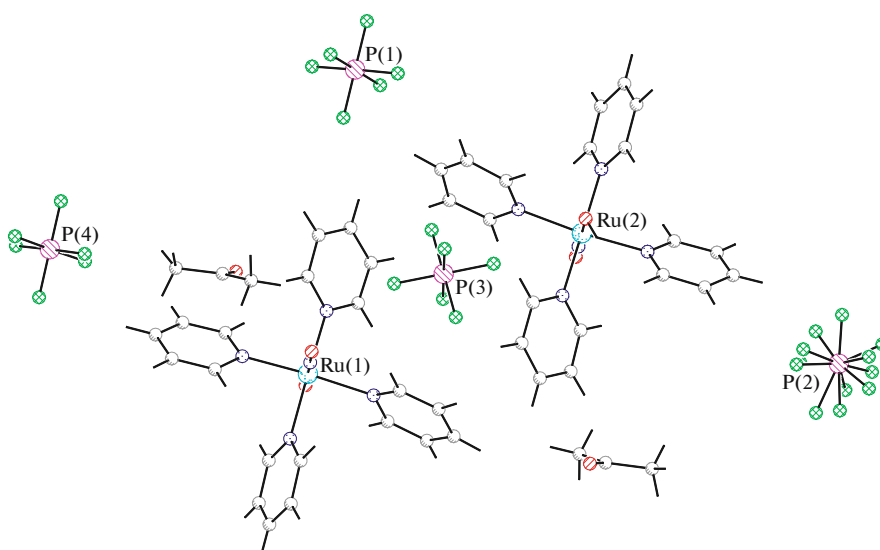
As we expected, the reactions of the hydroxo complex *trans*-[Ru(NO)(Py)<sub>4</sub>(OH)]<sup>2+</sup> with solutions of strong acids are accompanied by the protonation of coordinated hydroxide ions



In the case chloric acid, the gradual evaporation of the reaction solution does not result in the coordination of the ClO<sub>4</sub><sup>−</sup> ion to the Ru atom and the following aqua complex in the form of perchlorate salt is formed:



When hydrochloric acid is used, the slow evaporation of the reaction solution at ambient temperature affords yellow-orange crystals of compound **III** containing chloride ions in the internal sphere

**Fig. 1.** Packing of structural units in the crystal of compound **I**.

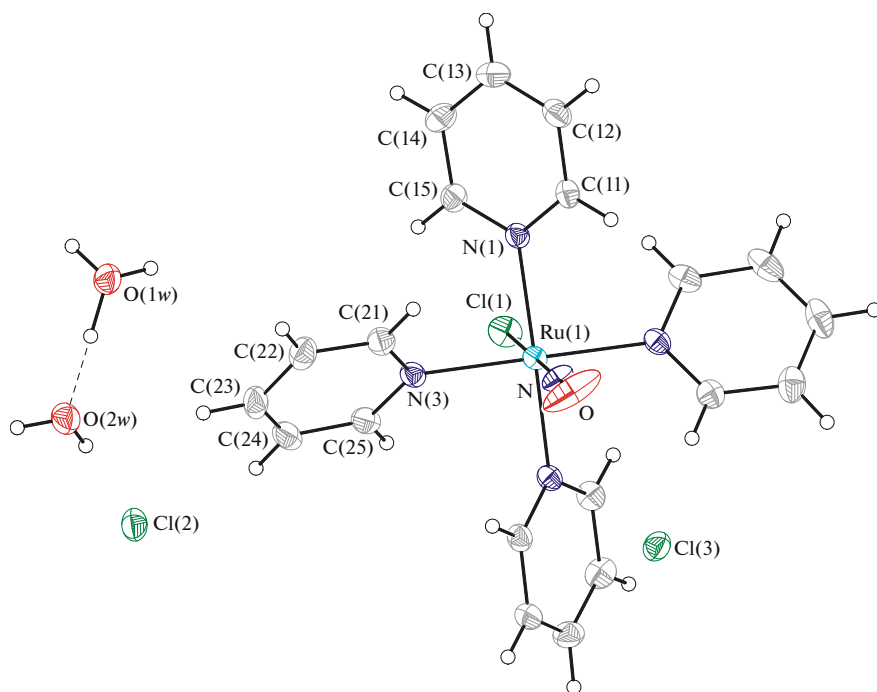
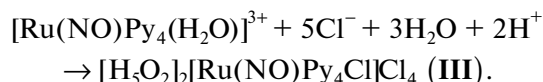
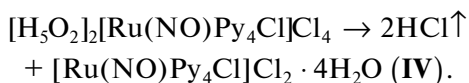


Fig. 2. Structure of the complex cation with the outer-sphere environment in compound **III**.



It has previously been reported that the hydroxo complex *trans*-[Ru(NO)(Py)<sub>4</sub>(OH)]<sup>2+</sup> quantitatively transforms into *trans*-[Ru(NO)Py<sub>4</sub>Cl]<sup>2+</sup> on heating to 80°C [11]. However, as our experiments showed, it is preferential to synthesize this complex at ambient temperature, because the substitution of coordinated pyridine molecules can occur on heating [13, 21].

According to the data of IR spectroscopy, X-ray diffraction analysis, and gravimetry, on storage in air compound **III** loses hydrogen chloride molecules, which was used for the preparation of chloro complex **IV**, whose structure has previously been described [9]



The IR spectra of all compounds obtained exhibit intense bands at 1850–1920 cm<sup>-1</sup> corresponding to the ν(NO) stretching vibrations. These values are in the range characteristic of the most part of nitrosoruthenium complexes containing the diamagnetic Ru(II) center and linearly coordinated NO<sup>+</sup> species [22, 23]. The IR spectra of compounds **I–IV** also contain bands characteristic of coordinated pyridine molecules [24, 25]: narrow medium- and low-intensity ν(CH) bands at 3100–3000 cm<sup>-1</sup>, narrow medium- and high-intensity ν(C<sub>arom</sub>–C<sub>arom</sub>) and ν(C<sub>arom</sub>–N<sub>arom</sub>) bands at

1600–1350 cm<sup>-1</sup>, and medium- and high-intensity δ(CH<sub>pl</sub>) and δ(CH<sub>out-of-pl</sub>) bands at 1240–970 and 760–630 cm<sup>-1</sup>, respectively. In addition, the spectra of compounds **I** and **II** contain the medium- and low-intensity bands caused by the presence of the hydroxide ion (for **I**) or water molecules (for **II**) in the internal sphere.

The structure of compound **III** was determined by X-ray structure analysis. Its crystal structure is built of the centrosymmetric complex cations *trans*-[Ru(NO)Py<sub>4</sub>Cl]<sup>2+</sup>, anions Cl<sup>-</sup>, and cations H<sub>5</sub>O<sub>2</sub><sup>+</sup>. The structure of the complex cation with the numeration of atoms and thermal vibration ellipsoids is shown in Fig. 2.

The square environment in the equatorial plane of the complex cation is formed by four nitrogen atoms of the pyridine molecules. The octahedral coordination mode of the ruthenium atom is supplemented by the nitroso group and chloride ion located in the *trans*-position to each other. The Ru–N<sub>Py</sub>, Ru–Cl, Ru–N<sub>NO</sub>, and N–O distances in this complex are characteristic of other known nitrosoruthenium complexes [9, 13, 26, 27]. The deviation of the bond angles at the ruthenium atom from 90° does not exceed 2.3°.

The structure of compound **III** contains layers parallel to the *yz* plane (Fig. 3). The interlayer space is occupied by H<sub>5</sub>O<sub>2</sub><sup>+</sup> and Cl<sup>-</sup>. The O⋯O distance in the H<sub>5</sub>O<sub>2</sub><sup>+</sup> cation is ~2.45 Å. The outer-sphere chloride ions form three short contacts with the π-systems of two pyridine molecules (Cl⋯Py 3.6–3.7 Å). The

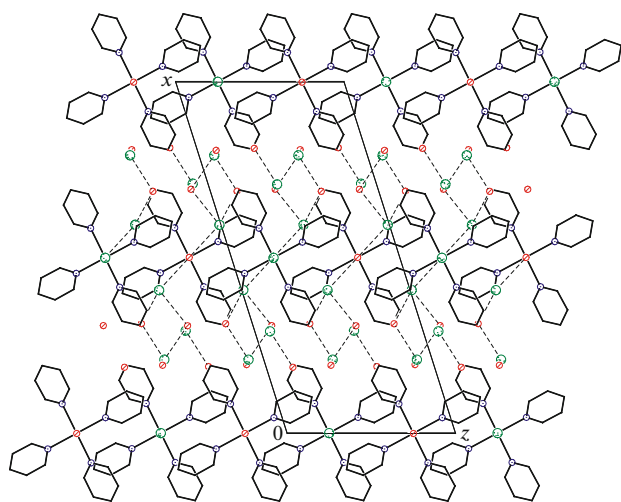


Fig. 3. Packing of particles in the structure of compound III along the  $y$  axis.

$\text{Cl}\cdots\text{O}$  distances between the  $\text{H}_3\text{O}_2^+$  particles and chloride ions are 2.93 and 3.06 Å, and the shortest  $\text{Ru}\cdots\text{Ru}$  distance is 7.7 Å.

To conclude, the slow evaporation of an acetone solution containing  $\text{trans-}[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})]^{2+}$  cations and hexafluorophosphate anions gave crystals of  $\text{trans-}[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})](\text{PF}_6)_2 \cdot (\text{CH}_3)_2\text{CO}$ . The reactions of the hydroxotetrapyrindine complex  $\text{trans-}[\text{Ru}(\text{NO})\text{Py}_4(\text{OH})]\text{Cl}_2 \cdot \text{H}_2\text{O}$  with solutions of strong acids at ambient temperature afford the aqua or acido complexes depending on the nature of the acid. In the case of using chloric acid, we isolated the aqua complex  $\text{trans-}[\text{Ru}(\text{NO})\text{Py}_4(\text{H}_2\text{O})](\text{ClO}_4)_3$ , and the chloride complex  $[\text{H}_3\text{O}_2]_2[\text{Ru}(\text{NO})\text{Py}_4\text{Cl}]\text{Cl}_4$  was isolated from a solution of hydrochloric acid. When this complex is kept in air at ambient temperature, the hydrogen chloride molecules are removed and  $\text{trans-}[\text{Ru}(\text{NO})\text{Py}_4\text{Cl}]\text{Cl}_2 \cdot 4\text{H}_2\text{O}$  is formed.

#### ACKNOWLEDGMENTS

The authors are grateful to N.I. Alferova for recording IR spectra and to N.P. Korotkevich for the detection of powder diffraction patterns.

This work was supported by the Complex program for basic research of the Siberian Branch of the Russian Academy of Sciences, project no. II.1.16.1.

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Translated by E. Yablonskaya