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Synthesis, Some Properties, and Crystalline Modifications of *fac*-$[Ru(NO)(Py)_2Cl_3]$

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Abstract—According to the data of ^{1}H NMR spectroscopy, *trans*-hydroxochloro complexes containing from two to four pyridine molecules in the internal sphere are formed on the heating of a dilute aqueous solution of $K_2[Ru(NO)Cl_5]$ with pyridine. The evaporation of the reaction solution with concentrated hydrochloric acid gives *fac*-$[Ru(NO)(Py)_2Cl_3]$ (**I**) in a yield of ~90%. The structures of two crystalline modifications of this complex are determined by X-ray diffraction analysis (CIF files CCDC nos. 1452208 (**Ia**) and 1452207 (**Ib**)). IR spectroscopy shows that the irradiation of complex **I** ($\lambda \sim 450$ nm, $T = 80$ K) results in photoisomerization with the formation of the metastable state MS1 in which the nitroso group is coordinated by the oxygen atom. The activation parameters of the photoisomerization are determined from the data of differential scanning calorimetry (DSC). Compound *trans*-$[Ru(NO)Py_4(OH)]Cl_2 \cdot H_2O$ is isolated in a yield of ~70% on reflux of complex **I** with a pyridine excess in an aqueous solution, and the presence of molecules of water of crystallization in this compound is confirmed by thermal gravimetry (TG) and IR spectroscopy.

Keywords: ruthenium nitrosyl, amino complexes, chloro complexes, pyridine, X-ray diffraction analysis, ^{1}H NMR spectroscopy, IR spectroscopy, thermogravimetry, photoisomerization, differential scanning calorimetry

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INTRODUCTION

Ruthenium nitroso complexes are actual objects of the study of modern coordination chemistry due to several reasons. First, these compounds are biologically active and considered as additional sources and low-toxicity regulators of the nitrogen monoxide concentration in biological systems [1, 2].

Second, the photochemical transformations of the ruthenium nitroso complexes [3, 4] give prospects for the synthesis of hybrid materials based on them and combining the photochromic properties with electric conductivity or magnetism [5, 6]. Among the presently known nitrosoruthenium compounds, the pyridine complexes $[Ru(NO)(Py)_4Cl](PF_6)_2 \cdot 0.5H_2O$ [7, 8] and $[Ru(NO)(Py)_2(NO_2)_2(OH)]$ [9] are characterized by the highest populations of the photogenerated metastable states. Published data on the studies of the photoisomerization of other pyridine complexes of nitrosoruthenium are lacking.

Data on the synthesis and structure of *fac*-$[Ru(NO)Py_2Cl_3]$ (**I**) are scarce. Compounds containing the *trans*-$[Ru(NO)Py_4(OH)]^{2+}$ complex cation can be obtained by the reaction of the ruthenium(II) dinitrotetrapyridine complex with chloric [10, 11] or trifluoroacetic [12] acids as follows:

$$\begin{aligned}&trans\text{-}[Ru(Py)_4(NO_2)_2] + 2H^+\\ &\rightarrow trans\text{-}[Ru(NO)(Py)_4(OH)]^{2+}\\ &+ 1/2H_2O + 1/2NO + 1/2NO_2.\end{aligned}$$

Only compounds with ClO_4^- and PF_6^- anions in the external sphere can be isolated in high yields (70–85%) in the solid phase from aqueous solutions, and the structure of the compound with PF_6^- was determined by X-ray diffraction analysis [13]. The chloride salt can be obtained only by the reaction of $[Ru(Py)_4(NO_2)_2]$ with trifluoroacetic acid in acetonitrile followed by the precipitation of the product with tetrabutylammonium chloride [12].

This work is the continuation of our studies [14] devoted to the search for synthesis procedures and the establishment of the structures of the ruthenium nitroso complexes with coordinated pyridine molecules. The purpose of this work is the development of procedures for the synthesis of compounds **I** and *trans*-$[Ru(NO)Py_4(OH)]Cl_2 \cdot H_2O$ (**II**) in high yields, spectral characterization of these compounds, establishment of the structures of the crystalline modifications of the dipyridine complex by X-ray diffraction analysis, and the study of its photoisomerization.

EXPERIMENTAL

The starting $K_2[Ru(NO)Cl_5]$ was obtained from commercially available ruthenium trichloride hydrate using a described procedure [15]. Other reagents and solvents were used as received (all of them were not lower than reagent grade).

Synthesis of complex I. A weighed sample (2.0 g, 5.2×10^{-3} mol) of $K_2[Ru(NO)Cl_5]$ was placed in a beaker and dissolved in ~1 L of distilled water, and pyridine (~5 mL) was added. The beaker was covered with a watch glass, and the reaction mixture was boiled for ~20 min. The obtained solution was evaporated on a rotary evaporator to wet salts. Concentrated hydrochloric acid (~10 mL) was added to the obtained mixture, and the resulting mixture was evaporated to dryness. The procedure of evaporation with the acid was repeated once more. Then distilled water (~10 mL) was added to dissolve KCl, and a red product was filtered off on a glass porous filter (40 pores). The precipitate was washed with cold water (~5 mL), ethanol, and ether. The yield of compound **I** was ~90%.

Single crystals of compound **Ia** suitable for X-ray diffraction analysis were obtained by the slow evaporation of a solution of compound **I** in DMF, and single crystals of compound **Ib** were obtained similarly from a saturated aqueous solution.

IR, ν, cm^{-1}: 3110–3030 ν(CH); 1867 ν(NO); 1606, 1482, 1449, 1352 $\nu(C_{arom}-C_{arom})$, $\nu(C_{arom}-N_{arom})$; 1241, 1209, 1156, 1093, 1066, 1018 $\delta(CH_{sh})$; 756, 689, 648 $\delta(CH_{out\text{-}of\text{-}plane})$; 605, 600 $\nu(Ru-N_{NO})$, δ(RuNO); 449 $\nu(Ru-N_{Py})$; 343, 329, 315 ν(Ru–Cl); 252, 222 $\delta(N_{Py}-Ru-N_{Py})$. ^1H NMR (DMSO), δ, ppm: 9.21 d (4H, $H^{2,6}$), 8.72 t (2H, H^4), 8.23 t (4H, $H^{3,5}$).

For $C_{10}H_{10}N_3OCl_3Ru$

anal. calcd., %:	C, 30.36;	H, 2.55;	N, 10.62;	Cl, 26.88.
Found, %:	C, 30.3;	H, 2.9;	N, 10.5;	Cl, 26.9.

Synthesis of complex II. Distilled water (~600 mL) and an excess of pyridine (~30 mL) were added to a weighed sample (~2.0 g, 5.1×10^{-3} mol) of preliminarily grinded compound **I**. The reaction mixture was refluxed for ~10 min. The obtained yellow solution was evaporated on a rotary evaporator to a red-orange oily residue (~5 mL), which was transferred to a beaker with the addition of ethanol (~20 mL). An ethanolic solution was evaporated at room temperature to ~5 mL, and a threefold volume of THF was added. After 10–15 min, a bright yellow precipitate that formed was filtered off. The yield of compound **II** was ~70%.

IR, ν, cm^{-1}: 3440, 3350, 3150 ν(OH); 3100–3000 ν(CH); 1848 ν(NO); 1635 δ(HOH); 1607, 1570, 1485, 1449, 1360 $\nu(C_{arom}-C_{arom})$, $\nu(C_{arom}-N_{arom})$; 1215, 1153, 1067, 1015 $\delta(CH_{sh})$; 949, 885 δ(RuOH); 783, 768, 696, 660, 642 $\delta(CH_{out\text{-}of\text{-}plane})$; 596 $\nu(Ru-N_{NO})$, δ(RuNO); 565 $\nu(Ru-O_{OH})$; 463 $\nu(Ru-N_{Py})$; 298, 287, 261, 239 $\delta(N_{Py}-Ru-N_{Py})$. ^1H NMR (DMSO), δ, ppm: 9.08 d (8H, $H^{2,6}$), 8.79 t (4H, H^4), 8.25 t (8H, $H^{3,5}$). ^1H NMR (H_2O), δ, ppm: 8.45 d (8H, $H^{2,6}$), 8.17 t (4H, H^4), 7.67 t (8H, $H^{3,5}$), 6.99 (1H, OH).

For $C_{20}H_{23}N_5O_3Cl_2Ru$

anal. calcd., %:	C, 43.41;	H, 4.19;	N, 12.65;	Cl, 12.81.
Found, %:	C, 41.5;	H, 4.4;	N, 12.1;	Cl, 12.7.

The IR spectra of the samples in KBr pellets were recorded on a Scimitar FTS 2000 FT-IR spectrometer in a range of 4000–375 cm^{-1} and on a Vertex 80 FT-IR spectrometer in the range 600–80 cm^{-1}. To detect photoisomerization, the substance as pellets with KBr was irradiated with a laser (450 nm, 100 mW) for 10 min and then the IR spectrum was rapidly recorded.

Analyses to C, H, and N were carried out on a EURO EA3000 analyzer, and Cl was analyzed using the Scheniger volumetric method with preliminary burning in oxygen [16].

The ^1H NMR spectra of solutions of the complexes were recorded on a Bruker Avance III spectrometer at a frequency of 500.0 MHz.

Thermogravimetry (TG) was carried out on a TG 209 F1 Iris® thermobalance (NETZSCH) (heating rate 10 K/min in a helium flow, helium flow rate 30 mL/min, Al_2O_3 crucible, weighed sample ~10 mg). The experimental results were processed using the OriginPro program package [17].

A NETZSCH DSC 204 F1 Phoenix differential scanning calorimeter was used to determine the kinetic parameters of the backward isomerization of the MS1 metastable state. A weighed sample (1–3 mg) was placed in an aluminum boat, cooled in nitrogen vapors, irradiated with a laser (450 nm, 100 mW) for 10 min, and rapidly transferred to the preliminarily cooled calorimeter. The samples were heated at different rates (12 and 15 K/min) in an argon flow (25 mL/min). The calorimeter was calibrated by the melting of standard samples (C_6H_{12}, Hg, KNO_3, In), and the experimental data were processed in the Netzsch Proteus Analysis program package [18].

The X-ray phase analysis of the grinded crystals was carried out on a DRON-3M diffractometer (R = 192 mm, CuK_α radiation, Ni filter, scintillation detector with amplitude discrimination). The samples were deposited as thin layers on the smooth side of a standard quartz cell. The diffraction patterns of the compounds were completely indexed by the data of single-

Table 1. Crystallographic data and the experimental and refinement parameters for the structures of compounds **Ia** and **Ib**

Parameter	Value	
	Ia	**Ib**
FW	395.63	395.63
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a, Å	15.4631(7)	8.006(2)
b, Å	6.8609(3)	12.758(3)
c, Å	14.5966(6)	14.308(3)
β, deg	116.473(2)	98.810(13)
V, Å^3	1386.19(10)	1444.1(6)
Z	4	4
ρ_{calcd}, g/cm^3	1.896	1.820
μ, mm^{-1}	1.699	1.631
F(000)	776	776
Crystal size, mm	0.25 × 0.20 × 0.18	0.18 × 0.15 × 0.10
θ Range, deg	2.79–33.34	2.88–30.38
Ranges of reflection indices	$-10 \le h \le 18$, $-10 \le k \le 3$, $-10 \le l \le 18$	$-11 \le h \le 8$, $-17 \le k \le 18$, $-20 \le l \le 16$
Number of measured reflections	2508	15766
Number of independent reflections	2203	4331
R_{int}	0.0126	0.0329
Completeness of data collection, %	52.1 (θ = 25.25)	99.8 (θ = 25.25)
Number of refined parameters	164	173
GOOF on F^2	1.078	1.039
R factor ($I > 2\sigma(I)$)	$R_1 = 0.0312$, $wR_2 = 0.0845$	$R_1 = 0.0303$, $wR_2 = 0.0658$
R factor (over all data)	$R_1 = 0.0385$, $wR_2 = 0.0875$	$R_1 = 0.0467$, $wR_2 = 0.0695$
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.614/–0.556	0.531/–0.307

crystal studies, which confirmed that the isolated products were single-phase.

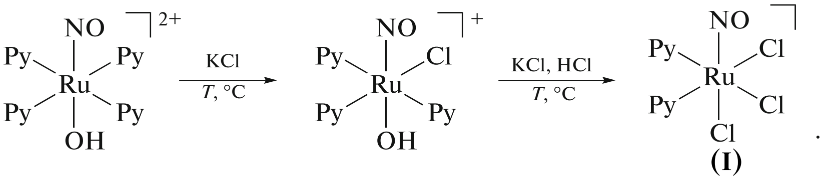
X-ray diffraction analysis. Unit cell parameters were determined and a set of experimental intensities was obtained on an X8 APEX Bruker automated diffractometer (MoK_α radiation, graphite monochromator, two-coordinate CCD detector). The structures were solved by the heavy atom method and refined in the anisotropic approximation of non-hydrogen atoms. Hydrogen atoms were specified geometrically in the idealized positions. All calculations were performed using the SHELX-97 program package [19].

The crystallographic characteristics and the main refinement parameters for compounds **Ia** and **Ib** are presented in Table 1. The data on the structures of compounds **Ia** and **Ib** were deposited with the Cambridge Crystallographic Data Centre (CIF files nos. 1452208 and 1452207, respectively; www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

Attempts to obtain the ruthenium nitrosopyridine complexes by the reaction of $K_2[Ru(NO)Cl_5]$ with an excess of pyridine in the absence of any solvent were unsuccessful: no visible changes occurred either at room temperature or on prolonged reflux.

The $[Ru(NO)Cl_5]^{2-}$ ion is activated in an aqueous solution on heating, and the chloroaqua complexes exhibit weakly acidic properties [15, 20]. According to the data of ^{1}H NMR spectroscopy, the formation of the pyridine complexes in the reaction of a dilute solution of $K_2[Ru(NO)Cl_5]$ with an excess of pyridine is observed already 5 min after the beginning of heating, and the concentration of the *trans*-$[Ru(NO)Py_4(OH)]^{2+}$ form becomes maximum in 10–15 min of reflux. The subsequent evaporation of the reaction solution in the presence of an excess of chloride ions in an acidic medium results in the formation of compound **I**



(I)

This scheme is also confirmed by the fact that compound *mer*-$[Ru(NO)Py_3Cl(OH)]Cl \cdot 1.5H_2O$, whose structure was determined by X-ray diffraction analysis [21], was isolated earlier from a similar system.

The evaporation of the reaction solution obtained by the reaction of $K_2[Ru(NO)Cl_5]$ with a pyridine excess affords compound **II** in a yield lower than 30%. Probably, such a low yield is due to the occurrence of the above mentioned processes of the substitution of the coordinated pyridine molecules by chloride ions and the formation of polymer species with bridging hydroxide ions [22, 23]. As it turned out, it is more efficient to use complex **I** as the initial ruthenium-containing compound for the synthesis of complex **II**.

The ^1H NMR spectra of solutions of compounds **I** and **II** exhibit three groups of signals assigned to three types of ^1H nuclei in the *ortho*, *para*, and *meta* positions of the coordinated pyridine molecules. The values of $\delta(^1H)$ range from 7 to 10 ppm, which is a characteristic range for the pyridine complexes of nitrosoruthenium [10, 24].

The IR spectra of the obtained compounds contain intense bands of ν(NO) stretching vibrations in the range characteristic of the most part of the ruthenium(II) nitroso complexes [25–27] and bands of low, medium, and high intensity caused by the presence of the coordinated pyridine molecules [28, 29]. In addition, the spectrum of compound **II** contains medium- and low-intensity bands of stretching vibrations of water molecules in a range of 3440–3150 cm^{-1}.

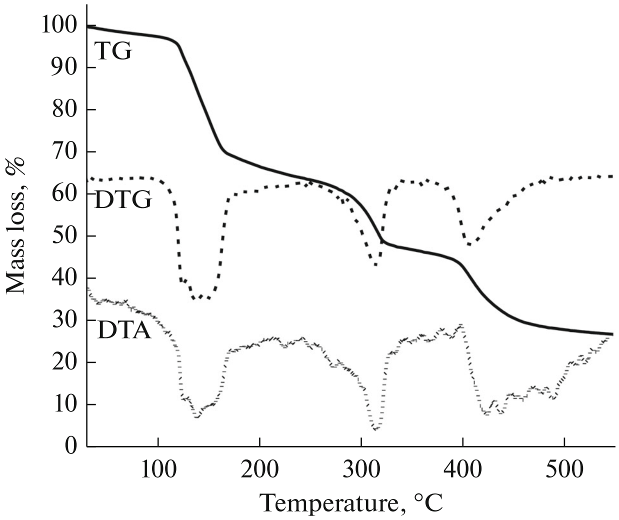


Fig. 1. TG curves for *trans*-$[Ru(NO)Py_4(OH)]Cl_2 \cdot H_2O$.

The presence of molecules of water of crystallization in tetrapyridine complex **II** is also confirmed by the data of TG analysis (Fig. 1) carried out in a helium atmosphere. One molecule of water of crystallization is removed (a value of 3.2% was both calculated and found from the mass loss curve) on the heating of compound **II** to ~110°C. The further heating to ~170°C results in the detachment of two pyridine molecules (calculated: 28.6%, found from the TG curve: 27.7%) and affords *trans*-$[Ru(NO)(Py)_2Cl_2(OH)]$ as an intermediate. The subsequent thermolysis stages are poorly separated, and the final thermolysis product contains, according to the X-ray phase analysis data, metallic ruthenium and a phase amorphous to X-rays (probably, carbon).

Dipyridine complex **I** was crystallized from different solvents as crystalline modifications **Ia** (from DMF) and **Ib** (from water). The structure of the *fac*-$[Ru(NO)(Py)_2Cl_3]$ complex species with the numeration of atoms and thermal vibration ellipsoids is presented in Fig. 2. Selected interatomic distances and angles are given in Table 2.

Two pyridine molecules in the complex species are arranged in the *cis* position to each other, and the square environment in the equatorial plane is supplemented by two chloride ions. The average values of similar bond lengths in species **Ib** are much higher than those in **Ia** except for Ru(1)–N and Ru(1)–Cl(1). The most differences in interatomic distances are observed along the O–N–Ru(1)–Cl(3) axis. On going from **Ib** to **Ia**, the contraction along this axis is ~0.07 Å, the sum of interatomic distances along the N(1)–Ru(1)–Cl(2) axis decreases by ~0.03 Å, and that along the N(2)–Ru(1)–Cl(1) axis remains unchanged. The Ru–N(Py), Ru–Cl, Ru–N(NO), and N–O distances in these crystalline modifications are characteristic of the known ruthenium nitroso complexes [30–32].

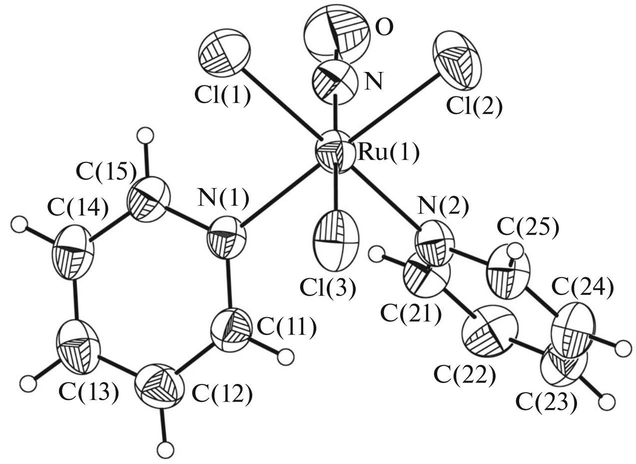


Fig. 2. Structure of the *fac*-[Ru(NO)Py_2Cl_3] complex species.

The coordination polyhedron of the ruthenium atom is a slightly distorted octahedron RuN_3Cl_3. The deviations of the bond angles at the ruthenium atom from 90° do not exceed 2.7° in **Ia** and 3.8° in **Ib**. The ONRu(1) bond angle in both structures is close to 180°. The pyridine molecules are inclined to the equatorial plane at angles of 45°–55° in **Ia** and 40°–45° in **Ib**. The central atom in the complex species is shifted from the equatorial plane to the NO group by 0.05 Å in **Ia** and 0.07 Å in **Ib**.

Both variants of the structure of compound **I** are of the insular type. They contain short C–H···Cl contacts (Cl···H(Py) ~2.8 Å). The shortest Ru···Ru distances in the structures of compounds **Ia** and **Ib** are ~6.7 and ~6.9 Å, respectively.

The photochemical transformations that occurred under the irradiation of the polycrystalline sample of compound **I** were studied by IR spectroscopy and DSC. An additional band at 1730 cm^{-1}, which can be interpreted as ν(NO) for the MS1 state [8, 9], appears in the IR spectrum of the irradiated sample (Fig. 3).

The DSC method was used to study the kinetics of the backward isomerization of the MS1 state. The measurements were carried out for three different weighed samples at different heating rates. The obtained temperature dependence of the heat flow (Fig. 4) is approximated by the first-order kinetic equation

$$\dot{H} = H_{\mathrm{tot}} k_0 \exp\left(-\frac{E_{\mathrm{a}}}{k_{\mathrm{B}}T} - \frac{k_0}{q}\int_{T_0}^{T} e^{-\frac{E_{\mathrm{a}}}{k_{\mathrm{B}}T'}} dT\right),$$

where $\dot{H}$ is the heat flow, H_{tot} is the total effect, k_0 is the preexponential factor, E_{a} is the activation energy, q is the heating rate, k_{B} is the Boltzmann constant, and T_0 is the initial temperature.

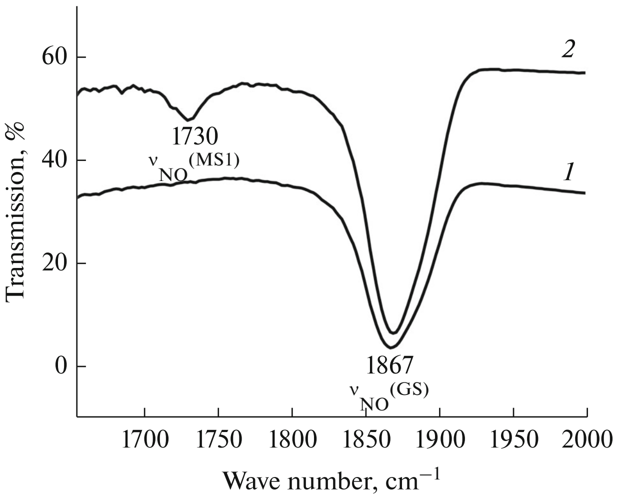


Fig. 3. IR spectra of *fac*-[Ru(NO)Py_2Cl_3]: (*1*) in the ground state (GS) and (*2*) after irradiation (GS + MS1) in the range of ν(NO) stretching vibrations.

All curves were calculated in a range of 178–218 K, and E_a and k_0 were found to be 62.5(7) kJ/mol and 4.7(8) × 10^{14} s^{-1}, respectively. Using these data, one can calculate the maximum decomposition temperature (T_d) of MS1, which is determined at a reaction rate constant of 10^{-3} s^{-1} [33]. For the studied compound, T_d = 185 K.

Thus, it was established that dipyridine complex **I** can be isolated in a high yield (~90%) from the reaction mixture obtained by the reaction of an aqueous

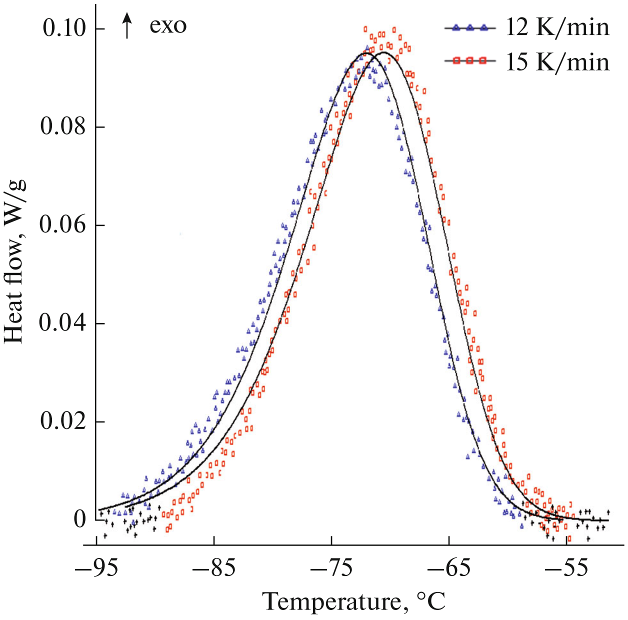


Fig. 4. Calculated (solid lines) and experimental (points) DSC data for irradiated *fac*-[Ru(NO)Py_2Cl_3].

Table 2. Selected interatomic distances and angles in the structures of compounds **Ia** and **Ib**

Bond	*d*, Å	
	Ia	**Ib**
Ru(1)–N	1.784(3)	1.766(2)
Ru(1)–N(1)	2.108(3)	2.1238(16)
Ru(1)–N(2)	2.111(3)	2.1195(17)
Ru(1)–Cl(1)	2.3848(11)	2.3778(7)
Ru(1)–Cl(2)	2.3586(10)	2.3782(7)
Ru(1)–Cl(3)	2.3038(10)	2.3415(8)
N–O	1.062(4)	1.115(3)
Angle	**ω, deg**	
	Ia	**Ib**
NRu(1)N(1)	92.68(13)	93.79(8)
NRu(1)N(2)	92.29(15)	92.41(8)
NRu(1)Cl(1)	90.38(13)	89.98(7)
NRu(1)Cl(2)	89.92(11)	89.74(7)
NRu(1)Cl(3)	177.93(10)	178.55(7)
N(1)Ru(1)Cl(1)	90.38(13)	89.27(5)
N(1)Ru(1)Cl(3)	88.73(7)	87.13(5)
N(2)Ru(1)Cl(1)	177.22(7)	177.61(5)
N(2)Ru(1)Cl(3)	89.25(8)	88.71(5)
N(2)Ru(1)N(1)	89.21(12)	90.63(6)
Cl(1)Ru(1)Cl(2)	91.05(4)	90.31(3)
Cl(1)Ru(1)Cl(3)	88.10(4)	88.90(3)
Cl(2)Ru(1)Cl(3)	88.70(4)	89.34(3)
Cl(2)Ru(1)N(1)	177.24(8)	176.45(5)
Cl(2)Ru(1)N(2)	89.75(8)	89.65(5)
ONRu(1)	176.6(4)	176.8(2)

solution of $K_2[Ru(NO)Cl_5]$ diluted with respect to ruthenium with a pyridine excess. To obtain tetrapyridine complex **II**, it is more efficient to use complex **I** instead of $K_2[Ru(NO)Cl_5]$ as the starting ruthenium-containing reagent. The formation of the isonitrosyl state Ru–O–N (MS1) was detected and the kinetic parameters of this isomerization were determined in the course of the photochemical studies carried out for compound **I**.

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