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Synthesis and Physicochemical Studies of the Compounds Based on $[Ni(Dien)(NH_3)_3]^{2+}$ and $[Ni(Trien)(NH_3)_2]^{2+}$ Cations and $[Re_6Se_8(CN)_4(OH)_2]^{4-}$ Octahedral Cluster Anion

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Abstract—Two new compounds, $[Ni(Dien)(NH_3)_3]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 5.5H_2O$ (**I**) and $[Ni(Trien)(NH_3)_2]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 4.67H_2O$ (**II**) (Dien is diethylenetriamine and Trien is triethylenetetraamine), are synthesized by layering solutions of $NiCl_2 \cdot 6H_2O$ in aqueous ammonia with the addition of the corresponding amines onto an aqueous solution of cluster salt $Cs_{2.75}K_{1.25}[Re_6Se_8(CN)_4(OH)_2] \cdot H_2O$. The structures of both complexes are determined by single-crystal X-ray diffraction analysis (CIF files CCDC 1446999 (**I**) and 1447000 (**II**)). The compounds are characterized by IR spectroscopy and elemental and thermogravimetric analyses.

Keywords: octahedral cluster, rhenium, nickel, crystal structure

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INTRODUCTION

The chemistry of octahedral cluster rhenium complexes with $[Re_6Q_8(CN)_6]^{4-/3-}$ (Q = S, Se, and Te) and $[Re_6Q_8(OH)_6]^{4-}$ (Q = S, Se) anions has intensively been studied in the recent years [1–3]. Interest in these complexes is due to such properties as solubility, a high resistance to thermal and chemical actions, a possibility of changing and modifying the ligand environment, the occurrence of redox transformations, and long-lived luminescence in the solid state and in solutions. In addition, owing to free donor atoms in terminal ligands, these complexes can be used as nanosized building units for the synthesis of coordination polymers with cations of various metals. This property was clearly demonstrated, for example, in a number of studies describing cyano-bridged polymers of various dimensionality built of anionic chalcocyanide complexes $[Re_6Q_8(CN)_6]^{4-/3-}$ (Q = S, Se, and Te) and diverse transition metal cations [4–19]. Complexes $[Re_6Q_8(OH)_6]^{4-}$ (Q = S, Se) were found to be convenient precursors for ligand exchange reactions, since apical OH^- ligands are relatively easily substituted by ligands of other types, for example, anions of carboxylic acids or organic molecules [20–23]. In addition, complexes $[Re_6Q_8(OH)_6]^{4-}$ were shown to be coordinated to alkaline-earth and transition metal cations to form polymeric structures similarly to the cyano complexes [24–26].

Heteroligand octahedral rhenium clusters *trans*-$[Re_6S_8(CN)_{6-n}(OH)_n]^{4-}$ (n = 2, 4) and *trans*-$[Re_6Se_8(CN)_4(OH)_2]^{4-}$ have recently been synthesized [27–29]. An important feature of these compounds is the presence of both types of ligands (CN^- and OH^-) with different functionalities, which makes them interesting objects for investigation. In our recent publications, we studied the reactions of these anions with some cationic Ni(II) and Cu(II) complexes with amines. A series of the compounds synthesized to the present time includes molecular $\{[Ni(NH_3)_5]_2[Re_6Se_8(CN)_4(OH)_2]\} \cdot 6H_2O$ [30] and $\{[Cu(CH_3NH_2)_4]_2[Re_6Se_8(CN)_4(OH)_2]\}$ [28]; ionic $[Cu(NH_3)_5]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 8H_2O$ [28], $[Ni(En)_3]_2[Re_6S_8(CN)_4(OH)_2] \cdot 5.5H_2O$, and $[Ni(En)_3]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 10H_2O$ [31]; or chain polymeric $[Cu(En)_2(H_2O)_2][\{Cu(En)_2\}Re_6S_8(CN)_4(OH)_2] \cdot 5.5H_2O$ and $[Cu(En)_2(H_2O)_2][\{Cu(En)_2\}Re_6Se_8(CN)_4(OH)_2] \cdot 5H_2O$ compounds [32]. Continuing these works, in order to study the influence of the ligand environment of the transition metal cation on the structures of the formed compounds, we carried out the reactions of the octahedral cluster chalcocyanohydroxo complex of rhenium *trans*-$[Re_6Se_8(CN)_4(OH)_2]^{4-}$ with Ni^{2+} cations coordinated by diethylenetriamine and triethylenetetraamine, respectively. Two new compounds of the ionic type were synthesized and characterized: $[Ni(Dien)(NH_3)_3]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 5.5H_2O$ (**I**) and $[Ni(Trien)(NH_3)_2]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 4.67H_2O$ (**II**).

EXPERIMENTAL

The starting cluster salt $Cs_{2.75}K_{1.25}[Re_6Se_8(CN)_4(OH)_2] \cdot H_2O$ was synthesized using a previously described procedure [28], and other reagents were used as purchased. The IR spectra of the samples were recorded on a Scimitar FTS 2000 FT-IR spectrometer in a range of 4000–400 cm^{-1} (KBr pellets). Elemental analyses to C, H, N, and S were carried out on a MICRO CORDER JM10 instrument. Thermogravimetric analysis (TG) was conducted on a NETZSCH TG 209 F1 Iris Thermo Microbalance instrument in a helium flow (30 mL/min) with a heating rate of 20 K/min.

Synthesis of compound I. A solution of $NiCl_2 \cdot 6H_2O$ (16 mg, 0.07 mmol) and diethylenetriamine (0.15 mL, 1.38 mmol) in a 30% aqueous solution of ammonia (10 mL) were carefully layered onto a solution of $Cs_{2.75}K_{1.25}[Re_6Se_8(CN)_4(OH)_2] \cdot H_2O$ (20 mg, 0.01 mmol) in water (5 mL) in a glass tube. On the next day the resulting orange crystals were filtered off and dried on a paper filter. Yield, 9 mg (44%).

IR, ν, cm^{-1}: 957 $\delta_{rocking}(CH_2)$, 1039 $\nu(OH_w)$, 1590 $\delta(H_2O)$ and $\delta(NH_2)$, 2099 ν(CN), 3266 and 3320 $\nu_{as}(NH)$, 3441 $\nu(OH^-)$.

For $C_{12}H_{57}N_{16}O_{7.5}Se_8Ni_2Re_6$

anal. calcd., %:	C, 6.0;	H, 2.4;	N, 9.3.
Found, %:	C, 5.9;	H, 2.4;	N, 8.8.

Synthesis of compound II was carried out similarly to that of compound **I** with the only distinction that a solution of $NiCl_2 \cdot 6H_2O$ (16 mg, 0.07 mmol) and triethylenetetraamine (20 mg, 1.37 mmol) in a 30% aqueous solution of ammonia (10 mL) was used as a layered solution. Orange needle-like crystals formed 2 days after were filtered off and dried on a paper filter. Yield, 8 mg (38%).

IR, ν, cm^{-1}: 950 $\delta_{rocking}(CH_2)$, 1050 $\nu(OH_w)$, 1590 $\delta(H_2O)$ and $\delta(NH_2)$, 2099 ν(CN), 3267 and 3316 $\nu_{as}(NH)$, 3426 $\nu(OH^-)$.

For $C_{16}H_{59.33}N_{16}O_{6.67}Se_8Ni_2Re_6$

anal. calcd., %:	C, 7.8;	H, 2.4;	N, 9.1.
Found, %:	C, 7.8;	H, 2.5;	N, 9.0.

X-ray diffraction analysis. The structures of complexes **I** and **II** were determined using a standard procedure on a Bruker-Nonius X8 Apex automated four-circle diffractometer equipped with a CCD two-coordinate detector at 296(2) K for **I** and 150(2) K for **II** (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). An absorption correction was applied by intensities of equivalent reflections (SADABS) [33]. The crystal structures were solved by a direct method. The positions of hydrogen atoms of the diethylenetriamine and triethylenetetraamine molecules and the hydroxo groups of the cluster anions were calculated geometrically and refined in the rigid body approximation. Hydrogen atoms of the crystallization water molecules were not localized. The final refinement was performed by full-matrix least squares in the anisotropic approximation for all non-hydrogen atoms. All calculations were performed using the SHELXTL program package [33], and the figures were drawn using the DIAMOND program [34]. The crystallographic characteristics and details of diffraction experiments are presented in Table 1. The coordinates of atoms and thermal parameters were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC 1446999 (**I**) and 1447000 (**II**); www.ccdc.cam.ac.uk/data_request/cif) and can be requested from the authors.

RESULTS AND DISCUSSION

Compounds **I** and **II** were synthesized by the cross diffusion method: an ammonia solution of $NiCl_2$ + Dien or $NiCl_2$ + Trien was carefully layered onto an aqueous solution of cluster salt $Cs_{2.75}K_{1.25}[Re_6Se_8(CN)_4(OH)_2] \cdot H_2O$ in a thin glass tube. This method was earlier used successfully to obtain compounds of various dimensionalities in the systems containing cluster chalcocyanide complexes of rhenium and diverse cationic transition metal complexes [35–38]. This work is a continuation of the study of the reactions of the anionic octahedral chalcocyanohydroxo rhenium complexes *trans*-$[Re_6Q_8(CN)_4(OH)_2]^{4-}$ (Q = S, Se) containing terminal ligands CN^- and OH^- of two types with ammine complexes of transition metals. In the previous works, we described a series of compounds with $[Ni(NH_3)_5]^{2+}$, $[Cu(CH_3NH_2)_4]^{2+}$, $[Cu(NH_3)_5]^{2+}$, $[Ni(En)_3]^{2+}$, $[Cu(En)_2(H_2O)_2]^{2+}$, and $[Cu(En)_2]^{2+}$ complex cations. It is interesting to extend this series of compounds from the viewpoint of establishing the structure-forming influence of complex transition metal cations with different compositions of the coordination sphere. For this purpose, we used chelate ligands of different denticites, namely Dien and Trien. Similarly to the compounds containing the $[Ni(En)_3]^{2+}$ complex cation, synthesized complexes **I** and **II** are ionic.

Compounds **I** and **II** can be assigned to isotypical. They are monoclinic with similar unit cell parameters (space group $P2_1/c$, $Z = 2$; Table 1). The independent

Table 1. Crystallographic characteristics and details of diffraction experiments and refinement for structures **I** and **II**

Parameter	Value	
	I	**II**
FW	2412.04	2449.07
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a, Å	8.7101(6)	8.6292(7)
b, Å	14.9235(10)	15.5819(13)
c, Å	17.9973(13)	18.0364(15)
β, deg	97.552(2)	97.093(2)
V, Å^3	2319.1(3)	2406.6(3)
Z	2	2
ρ_{calcd}, g cm^{-3}	3.454	3.380
$\mu_{MoK\alpha}$, mm^{-1}	22.712	21.887
Crystal size, mm	0.25 × 0.12 × 0.10	0.17 × 0.04 × 0.04
θ Range, deg	2.66–27.54	1.73–27.55
Ranges of indices h, k, l	$-9 \leqslant h \leqslant 11$, $-13 \leqslant k \leqslant 19$, $-22 \leqslant l \leqslant 23$	$-10 \leqslant h \leqslant 11$, $-20 \leqslant k \leqslant 13$, $-23 \leqslant l \leqslant 21$
Number of measured reflections:		
collected	13468	14270
unique (N) [R_{int}]	5276 (0.050)	5528 (0.047)
with ($I > 2\sigma(I)$ (N_o)	3900	3548
Number of refined parameters	239	255
F(000)	2158	2197
R_1, wR_2 on N_o	0.0578, 0.1551	0.0528, 0.1122
R_1, wR_2 on N	0.0809, 0.1659	0.1008, 0.1229
GOOF	1.067	1.053
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	5.828/–2.663	2.970/–1.910

part of the unit cell contains one complex cation $[Ni(Dien)(NH_3)_3]^{2+}$ (Fig. 1a) or $[Ni(Trien)(NH_3)_2]^{2+}$ (Fig. 1b), half a centrosymmetric cluster anion $[Re_6Se_8(CN)_4(OH)_2]^{4-}$, and water molecules in three positions with different site occupancies, which results in a lower content of hydrate water in compound **II**. The latter is primarily related to the disordering of the CN^- and OH^- terminal ligands of the cluster anion in structure **II** resulting in a decrease in the cell volume accessible for filling with solvent molecules and in some tendency of these compounds to dehydration. The structure of the $[Re_6Se_8(CN)_4(OH)_2]^{4-}$ cluster anion in compounds **I** and **II** (Fig. 2) is similar to the structure of this anion in the starting salt $Cs_{2.75}K_{1.25}[Re_6Se_8(CN)_4(OH)_2] \cdot H_2O$ and in a series of other compounds [28, 30–32]. This is typical of octahedral chalcogenide clusters $[Re_6Q_8L_6]^n$. The octahedron of the rhenium atoms is inscribed into the cube of the selenium atoms in such a way that each Se atom is the μ_3-ligand. In addition, each of four rhenium atoms is coordinated by the CN^- terminal ligands, and two other rhenium atoms are coordinated by the OH^- ligands arranged in the *trans* positions relative to each other. The interatomic distances in the cluster anions of compounds **I** and **II** are presented in Table 2 and compared with the distances in the $[Re_6Se_8(CN)_4(OH)_2]^{4-}$ [28], $[Re_6Se_8(CN)_6]^{4-}$ [39], and $[Re_6Se_8(OH)_6]^{4-}$ [40, 41] anions in some known compounds.

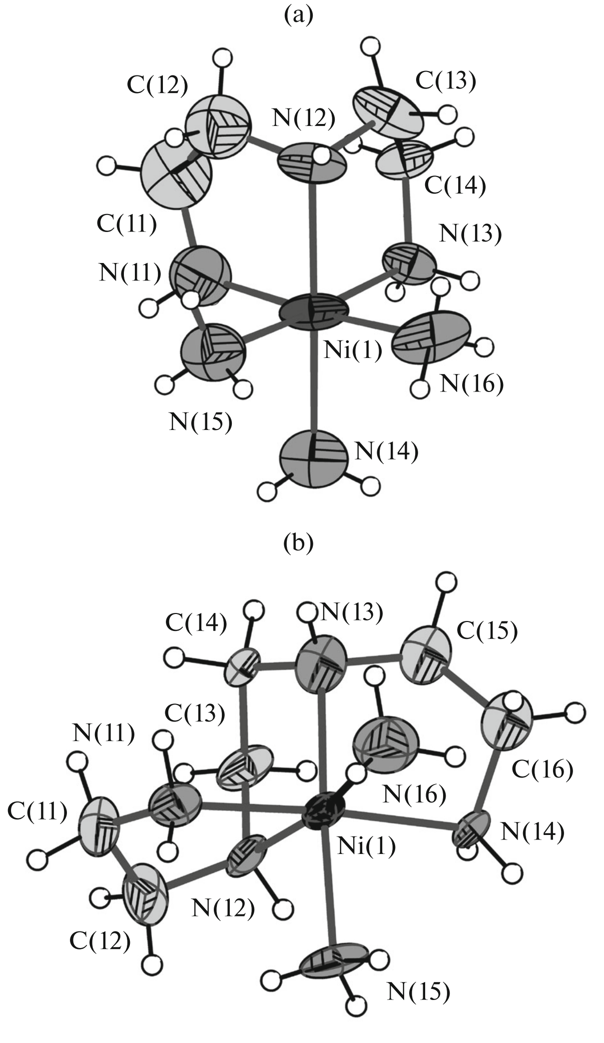

Fig. 1. Structures of cations (a) $[Ni(Dien)(NH_3)_3]^{2+}$ and (b) $[Ni(Trien)(NH_3)_2]^{2+}$. Thermal ellipsoids of 30% probability are presented.

The cationic part of compounds **I** and **II** is presented by the $[Ni(Dien)(NH_3)_3]^{2+}$ and $[Ni(Trien)(NH_3)_2]^{2+}$ complex cations containing one amine molecule that occupies three (in **I**) and four (in **II**) coordination sites of the central Ni atom (Ni–N(Dien) 2.00(3)–2.109(19), Ni–N(Trien) 2.061(18)–2.14(2) Å). The coordination environment of the Ni(1) atom is completed to the octahedral one by the N atoms of the ammonia molecules (Ni–$N(NH_3)$ 2.09(3)–2.16(3) Å in **I** and 2.10(2)–2.134(15) Å in **II**). The N–Ni–N angles lie in the following ranges: 80.7(8)°–91.6(10)° (**I**) and 82.0(7)°–85.5(7)° (**II**) for chelate angles, 87.6(12)°–95.7(9)° (**I**) and 87.5(8)°–96.4(6)° (**II**) for *cis* angles, and 173.1(9)°–176.4(9)° (in **I**) and 171.0(7)°–175.4(8)° (in **II**) for *trans* angles.

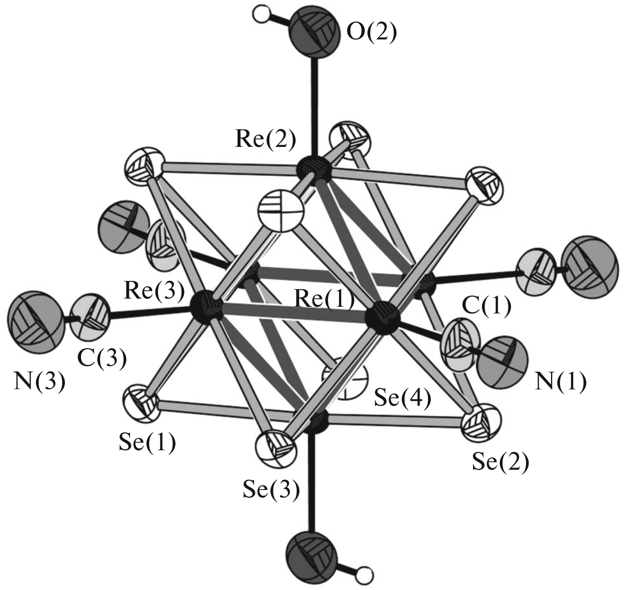

Fig. 2. Cluster anion *trans*-$[Re_6Se_8(CN)_4(OH)_2]^{4-}$ in structures **I** and **II**. Thermal ellipsoids of 50% probability are presented.

The general view of structures **I** and **II** is shown in Fig. 3. The complex cations, cluster anions, and crystallization water molecules are unified by a system of hydrogen bonds O–H···O, O–H···N, N–H···O, and N–H···N including water molecules, hydroxide and cyanide ligands of the cluster anions, and coordinated Dien, Trien, and ammonia molecules (minimum distances O···O ~2.7, O···N ~2.9, and N···N ~3.3 Å). Hydrogen bonds O–H···N–C (O···N 2.9865(5) Å) between the CN^- and OH^- ligands of the nearest cluster anions in structure **II** resulting in the formation of anionic chains parallel to the *x* axis (Fig. 4) should specially be mentioned. This type of binding of the cluster chalcocyanohydroxo complex has recently been described for a series of similar compounds [31, 32]. In this work, we attempted to study the factors accompanying the formation of intercluster binding interactions. For this purpose, we used complex formation with chelate Dien and Trien ligands exerting a direct effect on the ability of the complex cation to act as a donor of hydrogen bonds and thus governing (under other equivalent conditions) the topology of intermolecular interactions. However, it should be acknowledged that no explicit relationship was revealed to date between the composition of the compounds based on $[Re_6Q_8(CN)_4(OH)_2]^{4-}$ anions (Q = S, Se) and the presence (absence) of intercluster interactions O–H···N–C in their structure. It is most likely that this type of hydrogen bonds should be considered as one of manifestations of packing effects, which is probabilistic in character.

The thermal decomposition of compounds **I** and **II** was studied by the TG method of the corresponding powder samples. The total mass loss occurs in three

Table 2. Selected geometric characteristics of cluster anions $[Re_6Se_8(CN)_4(OH)_2]^{4-}$, $[Re_6Se_8(CN)_6]^{4-}$, and $[Re_6Se_8(OH)_6]^{4-}$ in complexes **I** and **II** and selected known compounds

Compound	*d*, Å		
	Re–Re	Re–(μ_3-Q)	Re–O, Re–C
I	2.6219(8)–2.6354(8)	2.5213(16)–2.5422(17)	2.110(14), 2.054(16), 2.108(18)
II	2.6205(8)–2.6328(9)	2.5153(16)–2.5347(16)	2.078(11), 2.107(15), 2.132(15)
$Cs_{2.75}K_{1.25}[Re_6Se_8(CN)_4(OH)_2] \cdot H_2O$ [28]	2.6160(11)–2.6239(12)	2.518(2)–2.532(2)	2.110(14), 2.01(2), 2.05(2)
$K_4[Re_6Se_8(CN)_6] \cdot 3.5H_2O$ [39]	2.624(1)–2.642(1)	2.516(1)–2.538(1)	2.09(2)–2.12(2)
$K_4[Re_6Se_8(OH)_6] \cdot 8H_2O$ [40]	2.6085(6)–2.6155(6)	2.527(1)–2.545(1)	2.081(6)–2.088(6)
$Cs_4[Re_6Se_8(OH)_6] \cdot 8H_2O$ [41]	2.6103(11)–2.6210(12)	2.522(2)–2.560(2)	2.059(9)–2.092(9)
$\{[Ni(NH_3)_5]_2[Re_6Se_8(CN)_4(OH)_2]\} \cdot 6H_2O$ [30]	2.6147(4)–2.6271(4)	2.5159(8)–2.5395(8)	2.080(5), 2.090(7), 2.106(6)
$[Ni(En)_3]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 10H_2O$ [31]	2.6196(6)–2.6333(5)	2.5168(11)–2.5361(11)	2.106(7), 2.077(11), 2.091(11)
$[Ni(NH_3)_2(En)_2]_2[\{Ni(NH_3)_4\}Re_6Se_8(CN)_6]Cl_2 \cdot 2H_2O$ [11]	2.6270(7)–2.6330(3)	2.5247(8)–2.5275(8)	2.08(1)–2.104(9)
$[Cu(NH_3)_5]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 8H_2O$ [28]	2.6198(6)–2.6311(6)	2.5140(12)–2.5327(11)	2.068(8), 2.100(10), 2.103(11)
$[\{Cu(CH_3NH_2)_4\}_2Re_6Se_8(CN)_4(OH)_2]$ [28]	2.6153(18)–2.6261(9)	2.512(2)–2.533(3)	2.13(3), 2.01(5), 2.12(4)
$[Cu(En)_2(H_2O)_2][\{Cu(En)_2\}Re_6Se_8(CN)_4(OH)_2] \cdot 5H_2O$ [32]	2.113(9)–2.6365(8)	2.5133(14)–2.533(18)	2.092(8), 2.053(16), 2.082(15)
$[\{Cu(H_2O)_{0.5}(En)_2\}Cu(En)_2\}Re_6Se_8(CN)_6] \cdot 3H_2O$ [11]	2.624(2)–2.647(2)	2.505(4)–2.542(4)	2.06(4)–2.18(4)
$[Cu(NH_3)(Trien)]_2[Re_6Se_8(CN)_6]$ [35]	2.5253(10)–2.6396(7)	2.5155(10)–2.5338(11)	2.104(10)

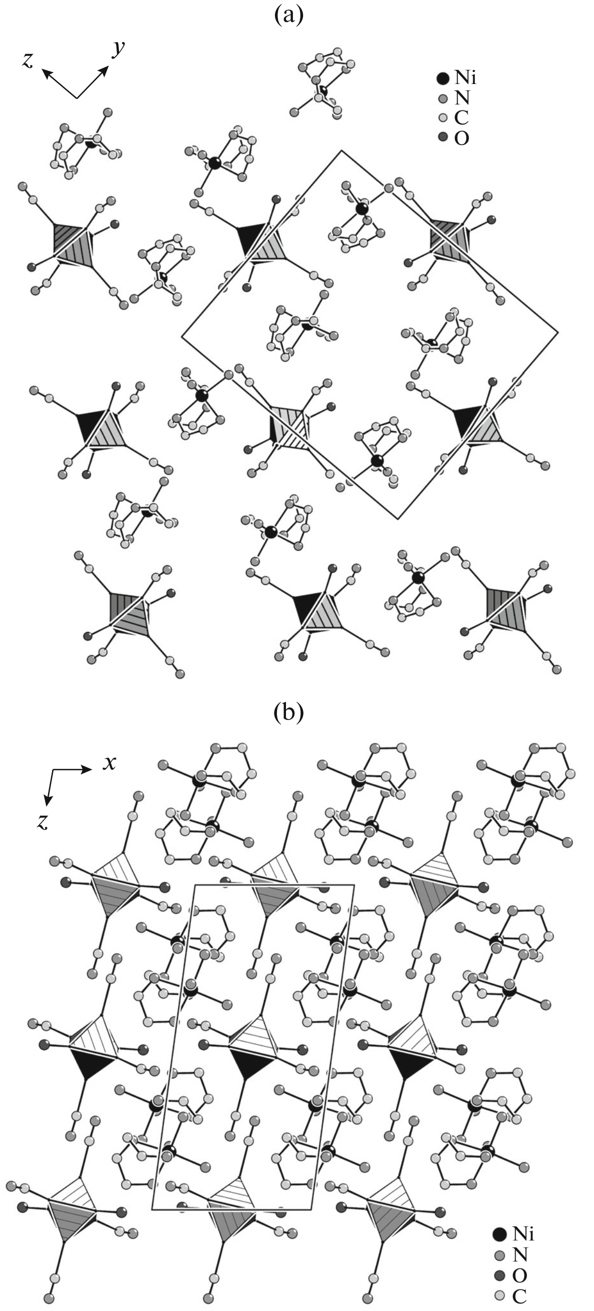

Fig. 3. General view of structures (a) **I** along the *x* axis and (b) **II** along the *y* axis. Cluster cores $\{Re_6Se_8\}^{2+}$ are shown as octahedra. Hydrogen atoms and solvate water molecules are omitted.

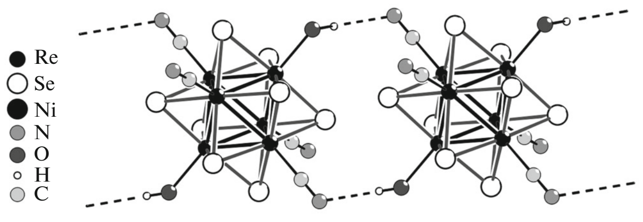

Fig. 4. Fragment of the chain formed due to the O–H···N–C hydrogen bonds between the adjacent $[Re_6Se_8(CN)_4(OH)_2]^{4-}$ anions in structure **II**.

stages corresponding to the removal of water, ammonia, Dien (for **I**), and Trien (for **II**) molecules and decomposition of the cluster cores (Fig. 5). The samples begin to decompose almost simultaneously with the temperature increase. At the first stage, the removal of water and ammonia molecules ceases at 150°C for **I** (calcd. 8%) and 120°C for **II** (calcd. 6%). A mass loss of 7% in a range of 200–420°C corresponds to the loss of Dien molecules (calcd. 8.5%). A mass loss of 9% in a range of 220–430°C corresponds to the loss of Trien molecules (calcd. 12%). Further heating decomposes the cluster cores. The intercluster O–H···N–C interactions result in the formation of ordered associates (chains or their fragments) consisting of cluster anions, which decreases, most likely, the strength and branching of the network of hydrogen bonds. This is also accompanied by a decrease in the temperature necessary for the complete removal of

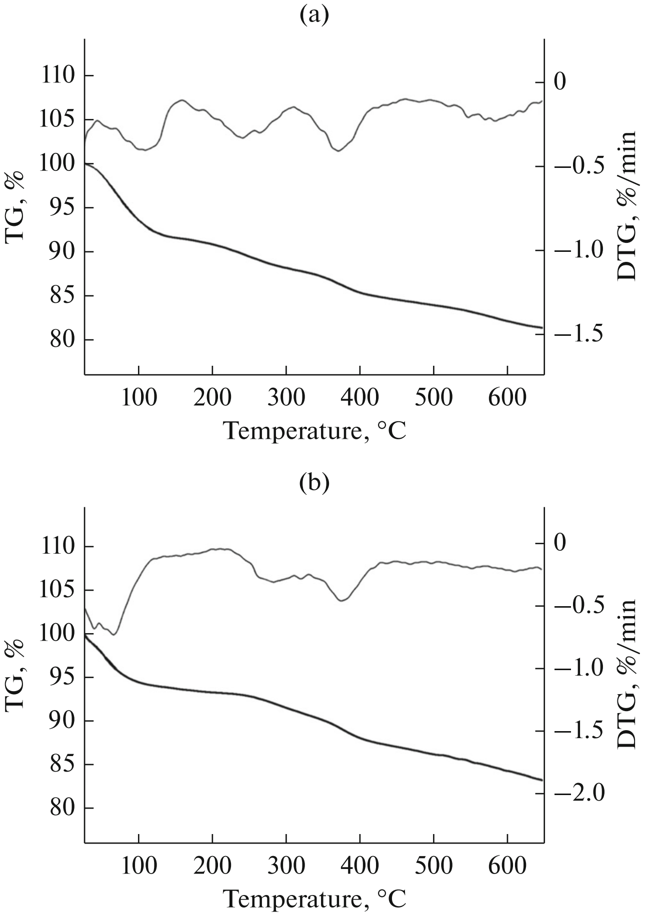

Fig. 5. The TG and DTG curves for compounds (a) **I** and (b) **II**.

water and ammonia molecules. On the one hand, the water and ammonia molecules in compound **II** are removed at a lower temperature than in compound **I**. On the other hand, dehydration is regularly observed at a significantly higher temperature (180°C) for the earlier synthesized compound $[Ni(En)_3]_2[Re_6Se_8(CN)_4(OH)_2] \cdot 10H_2O$ characterized by the absence of binding interactions between the adjacent cluster anions [31].

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