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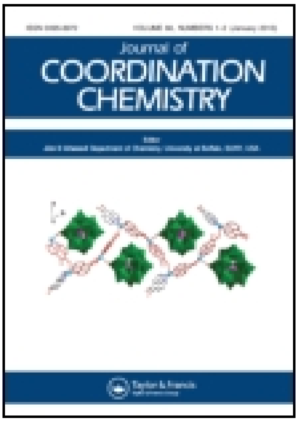

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Ionic coordination complexes based on $[Re_6S_8(CN)_4L_2]^{n-}$ ($L = OH^-$, NH_3; $n = 2, 3$) cluster anions, and Ni(II) and Cd(II) ammine cations

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Ionic coordination complexes based on $[Re_6S_8(CN)_4L_2]^{n-}$ ($L = OH^-$, NH_3; $n = 2, 3$) cluster anions, and Ni(II) and Cd(II) ammine cations

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Two new complexes containing M(II) ammine cations (M = Ni, Cd) and octahedral rhenium(III) thiocyanoammine and thiocyanohydroxoammine cluster anions, $[Ni(NH_3)_6][Re_6S_8(CN)_4(NH_3)_2]\cdot 2H_2O$ (**1**) and $[Cd(NH_3)_6][\{Cd(NH_3)_5\}\{Re_6S_8(CN)_4(OH)(NH_3)\}]_2\cdot 5H_2O$ (**2**), have been synthesized by hydrothermal reactions starting from $Cs_{1.83}K_{2.17}[Re_6S_8(CN)_4(OH)_2]\cdot 2H_2O$. The compounds were structurally characterized by single-crystal X-ray diffraction analysis, elemental analysis, energy dispersive spectroscopy, and IR spectroscopy. Both compounds adopt monoclinic crystal structures composed from discrete ionic species which are held together by multiple hydrogen bonds between CN^-, OH^-, and NH_3 ligands and lattice water. **2** consists of $\{Cd(NH_3)_5\}^{2+}$ attached to the OH group of the $[Re_6S_8(CN)_4(OH)(NH_3)]^{3-}$ cluster anion via the Re–OH–Cd linkage.

Keywords: Octahedral rhenium cluster; Nickel; Cadmium; Ammine complex; Crystal structure

1. Introduction

It has been shown in numerous studies that octahedral rhenium chalcocyanide anionic complexes $[Re_6Q_8(CN)_6]^{3-/4-}$ (Q = S, Se, Te) represent excellent rigid building blocks for the design of solid-state architectures of different dimensionalities [1–6]. The interaction of $[Re_6Q_8(CN)_6]^{4-}$ with multivalent metal ions (transition metals and lanthanides) in an aqueous solution can be described as a successive replacement of some water molecules in the starting aqua complex $[M(H_2O)_x]^{2+/3+}$ by N atoms of the ambidentate cyanide ligands. In most cases, these reactions lead to the formation of insoluble compounds possessing densely packed 3-D framework structures in which up to all six cyano groups of the anion are involved in the Re–CN–M bridging interactions. One of the ways that provides the possibility of adjusting the structure dimensionality is the employment of co-ligands which is able to compete with cluster anion's cyano groups for the metal coordination sites. Among these, the most known site-blocking agents are ammonia [7], polyamines (ethylenediamine [8–10], diethylenetriamine [11], triethylenetetramine [12]), and alcohols (methanol [13], glycerol [14], erythritol [15]). It is noteworthy, that the low-dimensional (composed of discrete ions and 1-D polymeric) complexes obtained from $[Re_6Q_8(CN)_6]^{4-}$ anions and

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*Corresponding author. Email: smolentsev@ngs.ru transition metal cations in the absence of any competing ligands are represented by only a few examples in literature [16–18].

On the other hand, the partially substituted anionic complexes $[Re_6S_8(CN)_4L_2]^{n-}$ (L are weaker donor ligands than CN^-) having a lower amount of ambidentate cyanide ligands are considered to be more appropriate building blocks for the specific construction of low-dimensional architectures. However, this research field is still far from being well explored. In this work, we focus on the investigation of coordination behavior of the $[Re_6S_8(CN)_4(OH)_2]^{4-}$ cluster anion [19] towards the divalent transition metal cations in aqueous ammonia under hydrothermal conditions. Here we report synthesis and characterization of two new compounds having an ionic structure, $[Ni(NH_3)_6][Re_6S_8(CN)_4(NH_3)_2]\cdot 2H_2O$ (**1**) and $[Cd(NH_3)_6][\{Cd(NH_3)_5\}\{Re_6S_8(CN)_4(OH)(NH_3)\}]_2\cdot 5H_2O$ (**2**).

2. Experimental

2.1. *Materials and instruments*

The starting compound $Cs_{1.83}K_{2.17}[Re_6S_8(CN)_4(OH)_2]\cdot 2H_2O$ was prepared as described [19]. All other reagents and solvents were used as purchased without purification. Elemental analysis was carried out with a Euro Vector EA 3000 CHNS-analyzer. Infrared spectrum (in KBr pellets) was recorded using a Scimitar FTS 2000 spectrometer from 4000 to 375 cm^{-1}. Energy dispersive spectroscopy (EDS) was performed on an EDAX equipped (JEOL EX-23000BU) JEOL JSM-6700F field emission scanning electron microscope.

2.2. *Synthesis and characterization*

2.2.1. Synthesis of 1. Twenty milligram (0.01 mM) of $Cs_{1.83}K_{2.17}[Re_6S_8(CN)_4(OH)_2]\cdot 2H_2O$ was dissolved in 5 mL of water. To the resulting solution, a solution of $NiCl_2$ (0.02 M) in concentrated aqueous ammonia was added until the precipitation was complete. The amorphous precipitate together with about 4 mL of mother liquor was transferred to a glass tube, which was sealed and heated at 130 °C for two days. After cooling, orange needle crystals of **1** were filtered off, washed with water and ethanol, and dried in air. Yield 14.6 mg, 81%. EDS: Ni : Re : S = 0.99 : 6 : 8.02. Anal. Calcd for $C_4H_{28}N_{12}NiO_2Re_6S_8$: C, 2.8; H, 1.6; N, 9.8; S, 15.0%. Found: C, 2.8; H, 1.7; N, 9.8; S, 15.5 %. IR, cm^{-1}: $\nu(NH_3)$ 3400–3100, 1605, 1243, 721; $\nu(OH_{aq})$ and $\delta(HOH)$ ~3400 and 1605 (overlap with vibrations of NH_3); $\nu(CN)$ 2118; $\nu(ReS)$ 470.

2.2.2. Synthesis of 2. The procedure was similar to that for **1**, except 0.02 M solution of $Cd(CH_3COO)_2$ in concentrated aqueous ammonia was used instead of the $NiCl_2$ solution. Yellow–orange needle crystals of **2** were the final product. Yield 16.4 mg, 90%. EDS: Cd : Re : S = 1.58 : 6 : 7.95. Anal. Calcd for $C_8H_{66}Cd_3N_{26}O_7Re_{12}S_{16}$: C, 2.6; H, 1.8; N, 9.8; S, 13.8%. Found: C, 2.6; H, 1.9; N, 9.7; S, 14.1%. IR, cm^{-1}: $\nu(NH_3)$ 3400 – 3100, 1613, 1230, 715; $\nu(OH)$ 3580; $\nu(OH_{aq})$ and $\delta(HOH)$ ~3400 and 1615 (overlap with vibrations of NH_3); $\nu(CN)$ 2130; $\nu(MOH)$ 995; $\nu(ReS)$ 468.

2.3. *Crystal structure determination*

Needle-shaped crystals of **1** and **2** were selected under a microscope and mounted to the tip of the thin glass fiber with epoxy resin. X-ray intensity data were collected on a Bruker X8

Table 1. Crystal data, data collection and refinement parameters for **1** and **2**.

Empirical formula	$C_4H_{28}N_{12}NiO_2Re_6S_8$	$C_8H_{66}Cd_3N_{26}O_7Re_{12}S_{16}$
Formula weight	1708.77	3723.43
Crystal size (mm^3)	0.30 × 0.02 × 0.02	0.12 × 0.05 × 0.05
Temperature (K)	100(2)	150(2)
Crystal system	Monoclinic	Monoclinic
Space group	*C2/m*	$P2_1/c$
a (Å)	12.9231(14)	8.6759(3)
b (Å)	10.4042(11)	16.9708(6)
c (Å)	12.1429(13)	22.6597(7)
β (°)	112.265(4)	90.1640(10)
Volume (Å^3)	1510.9(3)	3336.3(2)
Z	2	2
D_{Calcd} (g cm^{-3})	3.756	3.706
Absorption coefficient (mm^{-1})	25.122	23.147
F(000)	1516	3304
θ Range (°)	2.59–32.12	2.35–27.51
h, k, l ranges	$-9 \le h \le 19$, $-13 \le k \le 9$, $-17 \le l \le 11$	$-11 \le h \le 10$, $-21 \le k \le 22$, $-29 \le l \le 22$
Reflections collected	5598	29,060
Unique reflections	2011 (R_{int} = 0.0373)	7649 (R_{int} = 0.0532)
Observed reflections [$I > 2\sigma(I)$]	1623	5999
Parameters refined	98	343
$R[F^2 > 2\sigma(F^2)]$	$R_1 = 0.0323$, $wR_2 = 0.0548$	$R_1 = 0.0336$, $wR_2 = 0.0604$
$R(F^2)$ (all data)	$R_1 = 0.0503$, $wR_2 = 0.0597$	$R_1 = 0.0525$, $wR_2 = 0.0638$
Goodness-of-fit on F^2	1.051	1.023
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	2.333, –2.680	2.370, –1.671

Apex CCD diffractometer at 100(2) and 150(2) K for **1** and **2**, respectively, using graphite monochromated Mo Kα radiation (λ = 0.71073 Å). The standard technique was used (combined φ- and ω-scans of narrow frames). Data reduction and multi-scan absorption were carried out using SADABS [20]. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 using the SHELXTL software package [20]. All non-hydrogen atoms were refined anisotropically. Hydrogens of NH_3 groups were placed in geometrically idealized positions and refined as riding with U_{iso}(H) = 1.5U_{eq}(N); hydrogen of OH group (in **2**) was found in a difference Fourier map and refined isotropically with U_{iso}(H) = 1.2U_{eq}(O) and O–H distance restrained to 0.82(2) Å; hydrogens of crystallization water molecules were not located. The residual electron densities were found between –2.68 and 2.33 eÅ^{-3} (**1**), and –1.67 and 2.37 eÅ^{-3} (**2**), which fall within the expected ranges. Crystallographic data together with selected refinement details are given in table 1. The ranges of bond lengths and corresponding average values are given in table 2.

Table 2. Selected interatomic distances for **1** and **2** (min-max, average, Å).

Distance	**1**	**2**
Re–Re	2.5927(5)–2.6050(6), 2.597(5)	2.5904(5)–2.6050(5), 2.598(5)
Re–S	2.392(3)–2.418(2), 2.407(8)	2.401(2)–2.422(2), 2.412(6)
Re–C	2.116(8)	2.104(9)–2.116(10), 2.111(5)
C–N	1.152(10)	1.149(11)–1.157(12) 1.153(4)
Re–N	2.164(11)	2.172(7)
Re–O	–	2.072(6)
M–N	2.135(7)–2.140(10), 2.137(3)	2.304(10)–2.405(9), 2.36(4)
M–O	–	2.304(6)

3. Results and discussion

3.1. *Synthesis*

The reactivity study of the $[Re_6S_8(CN)_4(OH)_2]^{4-}$ anion carried out in the systems containing more than 10^{-3} M of Ni^{2+} or Cd^{2+} in an aqueous or ammonia-aqueous medium revealed that the mixing of the corresponding solutions leads to the immediate precipitation of insoluble amorphous products. The mixing of dilute solutions causes no immediate precipitation, but on long standing the precipitates were formed when the solvent was allowed to evaporate. According to the physico-chemical analyzes (EDS, thermogravimetry, IR spectroscopy), the composition of the products can be described as $M_2[Re_6S_8(CN)_4(OH)_2]{\cdot}xH_2O$ (M = Ni, Cd). In other words, the $[Re_6S_8(CN)_4(OH)_2]^{4-}$ anion shows no changes in the ligand environment when treated with aqueous ammonia at room temperature. In the present study, it was demonstrated that freshly precipitated amorphous solids taken with small amounts of mother liquor are successfully dissolved under hydrothermal conditions and the substitution of the OH groups by NH_3 molecules occurs during the process. Therefore, it can be seen that almost covalently bound hydroxide ligands, while being relatively labile, require rather hard synthetic conditions to be replaced by ammonia. It is interesting to note that the first (and only) example of ammine-containing rhenium cluster compound, $[Re_6Se_7Br(NH_3)_6][Re_6Se_7BrBr_6]{\cdot}12H_2O$ [21], was obtained by facile substitution of six terminal bromide ligands in the $[Re_6Se_7BrBr_6]^{3-}$ anion at ambient conditions. Another interesting point is the different number of OH groups affected by substitution with ammonia in the $[Re_6S_8(CN)_4(OH)_2]^{4-}$ anion during the synthesis of the title complexes. In particular, the synthesis of **1** is accompanied by substitution of two hydroxide ligands of anion; while in the case of **2,** only one is replaced. Such a behavior of the anion can be, though very approximately, accounted for on the basis of stability constants of the complexes which Ni^{2+} and Cd^{2+} ions form in the presence of ammine ligands. According to the literature data [22], the Ni^{2+} is readily coordinated by up to six NH_3 molecules to give the stable ion $[Ni(NH_3)_6]^{2+}$, while the analogous ion $[Cd(NH_3)_6]^{2+}$ is much less prevalent as compared to the $[Cd(NH_3)_4(H_2O)_2]^{2+}$ and $[Cd(NH_3)_5(H_2O)]^{2+}$ forms (step-wise stability constants β_5 and β_6 are only 0.50 and 0.02, respectively). This opens some possibility for the Cd^{2+} to be partially involved in coordination with hydroxide ligands of the $[Re_6S_8(CN)_4(OH)_2]^{4-}$ anion, thus making the bridging OH group insusceptible to further replacement. This situation is illustrated by the formation of bulky anionic complex $[\{Cd(NH_3)_5\}\{Re_6S_8(CN)_4(OH)(NH_3)\}]^-$ observed in the structure of **2** along with discrete $[Cd(NH_3)_6]^{2+}$ cations. It is worth mentioning that molar conductivity measurements (important tool in characterization of ionic complexes in solutions) cannot be performed for **1** and **2**. Upon dissolution of the compounds in water, the reverse substitution of coordinated NH_3 molecules by H_2O or OH^- (if concentrated aqueous ammonia is added) is observed. Therefore, the aqueous solutions of **1** and **2** do not reflect the proper ionic composition. The determination in non-aqueous solvents is also impossible due to the insolubility of the compounds.

3.2. *Crystal structures*

Compound **1** crystallizes in the monoclinic space group *C2/m* with 2 formula units per unit cell. The $[Ni(NH_3)_6]^{2+}$ cations and $[Re_6S_8(CN)_4(NH_3)_2]^{2-}$ cluster anions have *2/m* symmetry, while water molecules of crystallization are located in general positions. The

$[Re_6S_8(CN)_4(NH_3)_2]^{2-}$ shows the typical structure for octahedral rhenium cluster complexes (figure 1). Specifically, the Re atoms form an almost perfect octahedron with each of its triangular faces capped by a μ_3 bridging S atom, and CN^- and NH_3 ligands occupying the axial positions. Due to the imposed symmetry, the majority of the bond lengths and bond angles in the aforementioned ions are equal within esd's (table 2). The overall geometry of the anion is in good agreement with that reported earlier for the series of compounds based on the $[Re_6S_8(CN)_6]^{4-}$ and $[Re_6S_8(CN)_4(OH)_2]^{4-}$, including the starting $Cs_{1.83}K_{2.17}[Re_6S_8(CN)_4(OH)_2]\cdot 2H_2O$. A noteworthy characteristic of the $[Re_6S_8(CN)_4(NH_3)_2]^{2-}$ anion is two ammine ligands coordinated to the *trans*-positions of the rhenium cluster core. Both Re–N distances are equal to 2.164(11) Å which, as expected, is consistent with the value of 2.135 (10) Å found in $[Re_6Se_7Br(NH_3)_6]^{3+}$, the only known rhenium ammine cluster complex. In the structure, the ions interact with each other through hydrogen bonding between CN^- and NH_3 ligands and lattice water molecules, as well as through weaker electrostatic and van der Waals forces. The structure of **1** can be depicted as a pseudo-cubic NaCl-type packing in which the $[Ni(NH_3)_6]^{2+}$ cations and cluster anions occupy the positions of Na^+ and Cl^-, respectively. A complete packing diagram of **1** is presented in figure 2.

Compound **2** crystallizes in the monoclinic space group $P2_1/c$ and the unit cell contains 2 formula units. The $[Cd(NH_3)_6]^{2+}$ cation is centrosymmetric, while all other atoms including lattice water oxygen atoms are located in general positions. The anionic part of the structure, $[\{Cd(NH_3)_5\}\{Re_6S_8(CN)_4(OH)(NH_3)\}]^-$, consists of notably distorted fragment $\{Cd(NH_3)_5\}^{2+}$ coordinated to the OH group of the cluster anion $[Re_6S_8(CN)_4(OH)(NH_3)]^{3-}$. As seen in figure 3, the cluster anion is almost identical in structure to that in **1** except it has an OH group in place of one of the NH_3 ligands. The Re–N distance of 2.172(7) Å shows no significant difference with those given above. The most interesting feature to be considered is the Re–OH–Cd linkage, whose geometry is characterized by the following values: Re–O 2.072(6) Å; Cd–O 2.304(6) Å; ∠Re–O–Cd 128.7(3)°. It closely resembles the Re–OH–Cu bridges observed in recently reported coordination polymer $[\{Cu(NH_3)_3\}_2\{Re_6S_8(OH)_4(\mu\text{-}OH)_2\}]\cdot 8H_2O$ [23] in which these interactions participate in the formation of a 2-D layer structure. The most notable difference between these two bridging geometries resides in the rather large value of the Re–O–Cd angle, being about 18° larger

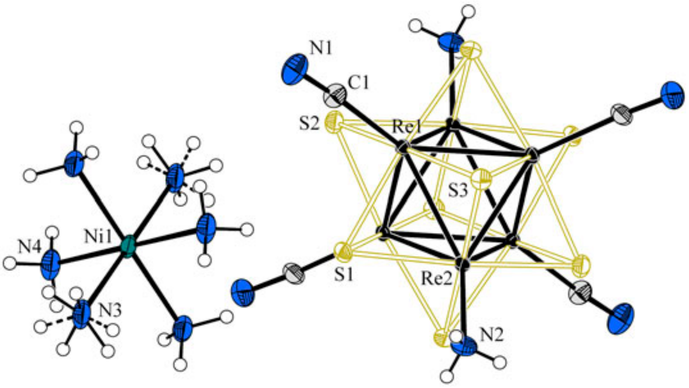

Figure 1. Displacement ellipsoid plot (at the 50% probability level) of $[Ni(NH_3)_6]^{2+}$ and $[Re_6S_8(CN)_4(NH_3)_2]^{2-}$ in **1**. Only the asymmetric unit is labeled. Hydrogens of two NH_3 groups in $[Ni(NH_3)_6]^{2+}$ are disordered by symmetry (shown by dashed lines).

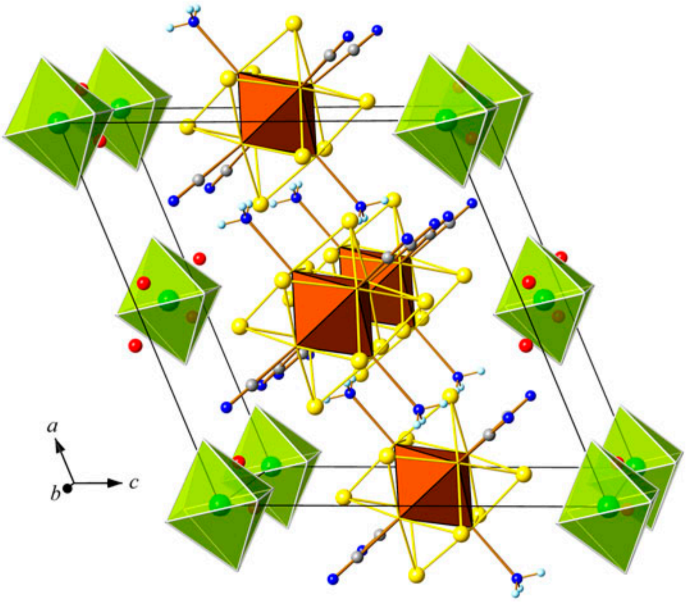

Figure 2. Packing diagram of **1** viewed along the *b* axis. $[Ni(NH_3)_6]^{2+}$ and $\{Re_6\}$ clusters are represented as translucent bright green and opaque brown octahedra, respectively. Hydrogen bonds are not shown (see http://dx.doi.org/10.1080/00958972.2013.865025 for color version).

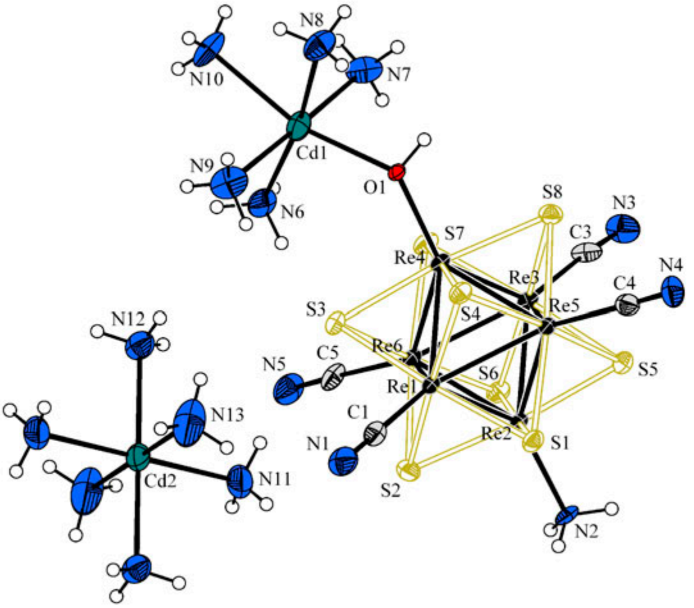

Figure 3. Displacement ellipsoid plot (at the 50% probability level) of $[Cd(NH_3)_6]^{2+}$ and $[\{Cd(NH_3)_5\}\{Re_6S_8(CN)_4(OH)(NH_3)\}]^-$ in **2**. Only the asymmetric unit is labeled.

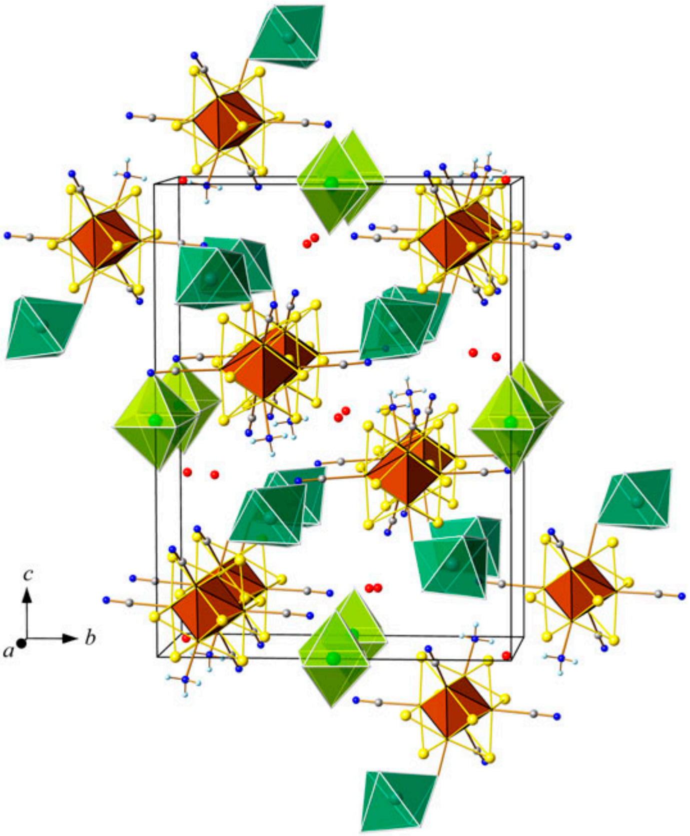

Figure 4. Packing diagram of **2** viewed along the *a* axis. $[Cd(NH_3)_6]^{2+}$, $\{Cd(NH_3)_5(OH)\}^{+}$ and $\{Re_6\}$ clusters are depicted as translucent bright green, teal green and opaque brown octahedra, respectively. Hydrogen bonds are not shown (see http://dx.doi.org/10.1080/00958972.2013.865025 for color version).

than the respective Re–O–Cu angle. However, the relatively low Re–O–Cu angle can be easily understood from the full coordination scheme of the Cu atom, which stays close to the cluster core due to the additional Cu···S interaction (~3 Å) with one of the μ_3-S atoms. It is probably not so favorable for the cadmium complex, owing to the lower affinity of Cd^{2+} ion towards sulfur binding sites. Similar to **1**, the structure of **2** is stabilized in all directions by multiple hydrogen bonds between the complex ions and lattice water molecules (figure 4).

4. Conclusions

In the present work, two new octahedral rhenium cluster anions, $[Re_6S_8(CN)_4(NH_3)_2]^{2-}$ and $[Re_6S_8(CN)_4(OH)(NH_3)]^{3-}$, have been isolated and characterized as their Ni(II) and Cd(II) ammine salts, respectively. The structures of the compounds are ionic in which the metal ammine cations exist in discrete (**1**) or both in discrete and coordinated to the cluster anion forms (**2**). In the field of rhenium cluster complexes, the compounds represent the second examples both in terms of the successful introduction of ammonia into the outer ligand shell of the cluster and the formation of Re–OH–M bridges in **2** through interaction with transition metal ammine ions. These findings suggest that rhenium cluster anions bearing CN^-, OH^-, and/or NH_3 apical ligands can be viewed as building blocks that will offer some new perspectives for construction of Re-containing coordination polymers and materials. In this way, by combining bridging and hindering functions of the anion's outer ligand shell, new low-dimensional compounds can be rationally designed and synthesized.

Supplementary material

The complete crystallographic data for the title complexes have been deposited at the Inorganic Crystal Structure Database under reference CSD-numbers 426457 (**1**) and 426458 (**2**). These data can be obtained free of charge via http://www.fiz-karlsruhe.de/obtaining_crystal_structure_data.html (or from the ICSD, Fachinformationszentrum Karlsruhe, 76,344 Eggenstein-Leopoldshafen, Germany).

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