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Syntheses and Structures of 1D Coordination Polymers Based on Cluster Anions $[Re_4Te_4(CN)_{12}]^{4-}$ and Cationic Ln^{3+} (Ln = La, Gd) Complexes with 1,10-Phenanthroline

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Abstract—Two new porous coordination polymers based on cluster anions $[Re_4Te_4(CN)_{12}]^{4-}$ and cationic Ln^{3+} (Ln = La, Gd) complexes with 1,10-phenanthroline (Phen) are synthesized under hydrothermal conditions. The structures of the compounds are determined by X-ray diffraction analysis (CIF files CCDC 1437445 (**I**) and 1437446 (**II**)). Compound $(PhenH)[\{La(H_2O)_3(Phen)_2\}\{Re_4Te_4(CN)_{12}\}] \cdot 1.5Phen \cdot 6H_2O$ (**I**) crystallizes in the space group $P\bar{1}$ (triclinic system): $a = 13.322(3)$, $b = 15.977(3)$, $c = 18.576(4)$ Å, $\alpha = 71.34(3)°$, $\beta = 85.56(3)°$, $\gamma = 88.27(3)°$, $V = 3734.8(13)$ Å^3. Compound $(PhenH)[\{Gd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}] \cdot 2Phen \cdot 0.5H_2O$ (**II**) crystallizes in the space group $C2/c$ (monoclinic crystal system): $a = 18.146(1)$, $b = 30.245(2)$, $c = 13.455(2)$ Å, $\beta = 97.858(2)°$, $V = 7315.4(1)$ Å^3. Structures **I** and **II** are based on polymer chains consisting of alternating fragments $[Re_4Te_4(CN)_{12}]^{4-}$ and $\{Ln(H_2O)_n(Phen)_2\}^{3+}$ (Ln = La, $n = 3$; Ln = Gd, $n = 2$) linked by the bridging CN ligands. The packings of the polymers contain extended channels due to the developed network of noncovalent interactions. The walls of the channels are formed by both hydrophilic (CN^-) and hydrophobic (Phen) groups. The channels, whose volume is 25 and 15% for compounds **I** and **II**, respectively, are filled by disordered Phen molecules and $PhenH^+$ cations, as well as by H_2O molecules.

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INTRODUCTION

The synthesis and study of coordination polymers represent a field of experimental inorganic chemistry intensively developed during three recent decades [1–3]. Interest in these compounds is related to wide possibilities of their use, for example, as molecular sieves and safe containers for gas storage [4–6]. The crystalline lattice of the coordination polymers consists of fragments of different nature named building blocks. The latter can be metal ions, metal clusters and polyoxometallates, and organic linking molecules [7]. In particular, the rhenium cyanoclusters are successfully used as building blocks for the synthesis of coordination polymers with *d*- and *f*-metal ions due to the rigidly fixed geometry and ambidentate terminal CN groups [8–10].

The search for synthesis methods and the study of the properties of the coordination polymers based on cluster anions $[Re_4Q_4(CN)_{12}]^{4-}$ and rare-earth metal cations are being continued for about ten years. The tetrahedral cluster anions $[Re_4Q_4(CN)_{12}]^{4-}$ (Q = S, Se, Te) [11–13] consist of the metal center $\{Re_4\}$, each face of which is coordinated by the μ_3-chalcogen atoms to form a pseudo-cubic core $\{Re_4Q_4\}^{8+}$, and 12 terminal CN ligands coordinated to the Re atoms. The differently directed sterically available CN ligands in combination with high coordination numbers characteristic for Ln^{3+} cations create prerequisites for the formation of numerous types of structures with different geometries and dimensionalities. In addition, the inclusion of rare-earth metal cations into the structure of coordination polymers makes it possible to impart unique combinations of properties to materials based on these compounds, for example, luminescence and selective adsorption of gases [14]. Several tens of compounds containing cluster anions $[Re_4Q_4(CN)_{12}]^{4-}$ and aqua complexes of rare-earth metal cations $\{Ln(H_2O)_n\}^{3+}$ as building blocks have been synthesized previously [15]. The crystal structures and magnetic properties of these compounds were studied, and proton conductivity was observed for acid hydrate $H[Lu(H_2O)_6\{Re_4Te_4(CN)_{12}\}] \cdot 6H_2O$ [16]. A common feature of the structures of all listed polymers is the presence of aqua complexes of rare-earth metal cations as secondary building blocks. No examples for coordination polymers containing simultaneously the cluster anion $[Re_4Q_4(CN)_{12}]^{4-}$ and cation Ln^{3+} coordinated by the organic ligand were met in the literature up to recently. The coordination of aromatic organic ligands to rare-earth metal cations in polymer compounds is interesting from the viewpoint of packing design and is a popular method to increase the photoluminescence intensity [17]. The first example for the synthesis and structural study of the coordination polymer $(PhenH)[\{Nd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}] \cdot 11H_2O$ in which the Nd^{3+} cation is coordinated by two 1,10-phenanthroline (Phen) molecules has been described several years ago [18]. In this work, we describe the syntheses and structures of two new 1D chain coordination polymers based on the cluster anion $[Re_4Te_4(CN)_{12}]^{4-}$ and La^{3+} and Gd^{3+} cationic complexes with Phen. The key feature of the structures of compounds $(PhenH)[\{La(H_2O)_3(Phen)_2\}\{Re_4Te_4(CN)_{12}\}] \cdot 1.5Phen \cdot 6H_2O$ (**I**) and $(PhenH)[\{Gd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}] \cdot 2Phen \cdot 0.5H_2O$ (**II**) is the channel type packing formed by noncovalent interactions between the Phen molecules of the adjacent polymer chains.

EXPERIMENTAL

Salt $K_4[Re_4Te_4(CN)_{12}] \cdot 5H_2O$ (**III**) obtained by the interaction of $[Re_4Te_4(TeBr_2)_4Br_8]$ with an aqueous solution of KCN [12] was used. Other reagents (Phen, rare-earth metal salts) were obtained from commercial sources and used as received. The ratio of heavy elements in the obtained crystals was determined by energy dispersive X-ray spectroscopy (EDS) on a Hitachi TM-3000 electron microscope with a Bruker Nano EDS analyzer. IR absorption spectra in a range of 4000–375 cm^{-1} were recorded on a Scimitar FTS2000 spectrometer.

Synthesis of complex I. Solutions of $K_4[Re_4Te_4(CN)_{12}] \cdot 5H_2O$ (10 mg, 0.006 mmol) in H_2O (0.5 mL) and $LaCl_3 \cdot 5H_2O$ (25 mg, 0.071 mmol) and Phen (15 mg, 0.083 mmol) in H_2O (0.5 mL) were loaded into a glass tube. The solutions were mixed together to form a colored precipitate and a colorless solution. The resulting solution was acidified with an aqueous solution of HCl to pH 2.5. The tube was sealed, kept for 48 h at 120°C, and cooled to room temperature. Needle-like red-brown crystals along with a brown amorphous substance were formed during the reaction time on the bottom and walls of the tube. Crystals for the studies were sampled manually, washed with water, and dried in air. The yield was 4.5 mg (38%). EDS: found La : Re : Te = 1.1 : 4.0 : 4.0.

IR (KBr), ν, cm^{-1}: 3464 ν(OH), 2143 ν(CN), 1615 δ(HOH). All absorption bands characteristic of phenanthroline molecules are observed in the IR spectrum.

Synthesis of complex II was similar to that of complex **I** using $GdCl_3 \cdot 6H_2O$ (25 mg, 0.067 mmol) instead of $LaCl_3 \cdot 5H_2O$. The yield was 5.0 mg (42%). EDS: found Gd : Re : Te = 1.0 : 4.0 : 4.2.

IR (KBr), ν, cm^{-1}: 3445 ν(OH), 2149 ν(CN), 1616 δ(HOH). All absorption bands characteristic of Phen molecules are observed in the IR spectrum.

X-ray diffraction analysis. Diffraction data for a single crystal of compound **I** were obtained at 150 K using a standard procedure on a Bruker Apex II Duo automated diffractometer (two-coordinate CCD detector, 150 K, MoK_α radiation, $\lambda = 0.71073$ Å, graphite monochromator). Diffraction data for a single crystal of compound **II** were obtained on a Bruker Nonius X8 Apex automated diffractometer (two-coordinate CCD detector, 293 K, MoK_α radiation, $\lambda = 0.71073$ Å, graphite monochromator). An absorption correction was applied empirically starting from intensities of equivalent reflections (SADABS) [19]. The structures of compounds **I** and **II** were solved by a direct method and refined by full-matrix least squares in the anisotropic approximation for all non-hydrogen atoms (SHELX-2007) [19]. The hydrogen atoms of the phenanthroline molecules were specified geometrically and refined by the riding model. The hydrogen atoms of the molecules of water of crystallization were not localized. Experimental details and the main crystallographic data are presented in Table 1. Selected bond lengths are listed in Table 2.

The crystallographic data for compounds **I** and **II** were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC 1437445 and 1437446, respectively; www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The reactions of the cluster anions $[Re_4Q_4(CN)_{12}]^{4-}$ (Q = S, Se, Te) with rare-earth metal cations in aqueous solutions have been studied for several years. The interaction of aqueous solutions of salts of these ions was shown to afford coordination polymers with cations of almost all rare-earth metals [15]. The addition of chelating organic ligands, such as Phen or 2,2'-bipyridine (Bipy), to the reaction mixture sharply changes the reaction course. As a rule, the mixing of solutions containing $[Re_4Q_4(CN)_{12}]^{4-}$, Ln^{3+}, and organic ligand L results in the fast formation of poorly soluble precipitates amorphous to X-rays and containing, according to the elemental analysis data, all components of the mixture in a variable ratio. Different methods are used to obtain crystalline products. We have previously shown that the counter diffusion of aqueous solutions containing salts $K_4[Re_4Te_4(CN)_{12}] \cdot 5H_2O$ and $NdCl_3 \cdot 6H_2O$ and Phen is an efficient method for the preparation of single crystals of the chain coordination polymer $(PhenH)[\{Nd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}] \cdot$

Table 1. Experimental details and the main crystallographic data for compounds **I** and **II**

Parameter	**I**	**II**
Empirical formula	$C_{66}H_{38}LaN_{21}O_6Re_4Te_4$	$C_{72}H_{40}GdN_{22}O_{2.50}Re_4Te_4$
FW	2615.28	2665.71
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$
a, Å	13.322(3)	18.147(1)
b, Å	15.977(3)	30.245(2)
c, Å	18.576(4)	13.455(2)
α, deg	71.34(3)	90.00
β, deg	85.56(3)	97.858(2)
γ, deg	88.27(3)	90.00
V, Å^3	3734.8(13)	7315.4(13)
Z	2	4
ρ_{calcd}, g cm^{-3}	2.326	2.420
μ, mm^{-1}	8.613	9.115
F(000)	2388	4872
Crystal size, mm	0.18 × 0.05 × 0.02	0.40 × 0.03 × 0.02
2θ Range, deg	1.86–27.59	1.32–28.31
Range of indices	$-17 \leqslant h \leqslant 17$, $-18 \leqslant k \leqslant 20$, $-24 \leqslant l \leqslant 22$	$-24 \leqslant h \leqslant 22$, $-40 \leqslant k \leqslant 40$, $-17 \leqslant l \leqslant 16$
Measured reflections	33283	34411
Independent reflections	17203	9050
R_{int}	0.0301	0.0450
Reflections with $I > 2\sigma(I)$	13682	7317
GOOF for F^2	1.061	1.080
R, $I > 2\sigma(I)$	$R_1 = 0.0359$, $wR_2 = 0.0878$	$R_1 = 0.0333$, $wR_2 = 0.0812$
R, all reflections	$R_1 = 0.0524$, $wR_2 = 0.0926$	$R_1 = 0.0515$, $wR_2 = 0.0905$
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	1.895/–1.939	1.305/–1.557

Table 2. Selected bond lengths in compounds **I** and **II**

Bond	*d*, Å			
	I (Ln = La)		**II** (Ln = Gd)	
	range	average	range	average
Re–Re	2.864(1)–2.877(1)	2.871(5)	2.8598(4)–2.8987(6)	2.88(2)
Re–Te	2.618(1)–2.647(1)	2.636(8)	2.6290(6)–2.6468(5)	2.639(6)
Ln–N(CN)	2.561(9)–2.641(9)	2.60(6)	2.465(5)	
Ln–N(Phen)	2.695(9)–2.800(9)	2.73(5)	2.563(7)–2.568(6)	2.566(4)
Ln–O(H_2O)	2.509(7)–2.552(7)	2.53(2)	2.377(5)	

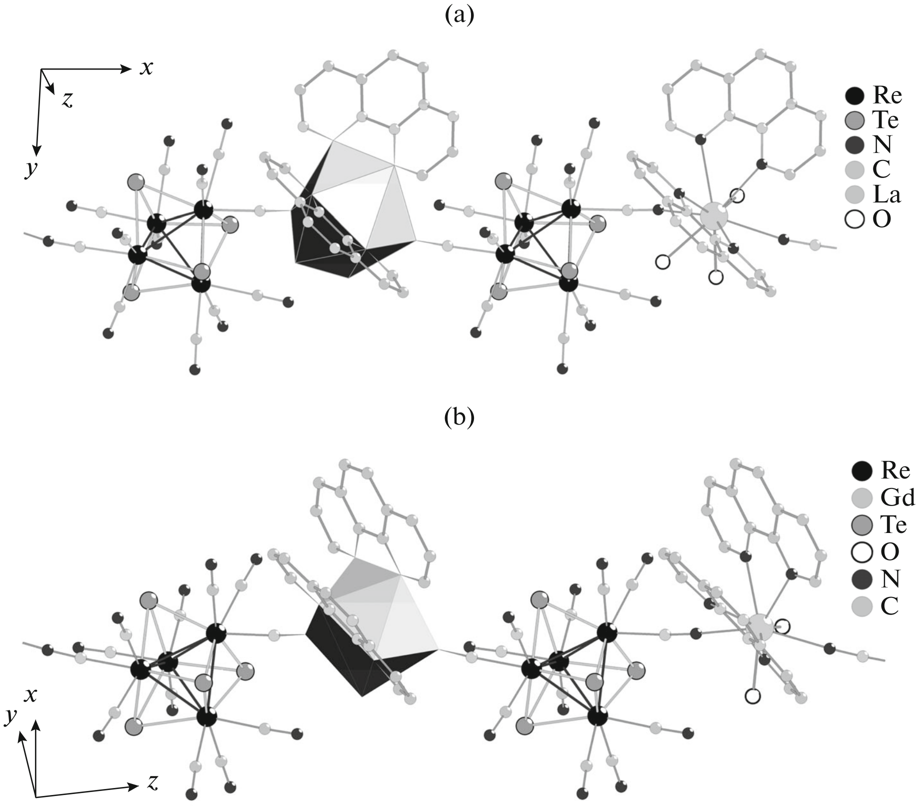

Fig. 1. Polymer chains $[\{La(H_2O)_3(Phen)_2\}\{Re_4Te_4(CN)_{12}\}]^-$ and $[\{Gd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}]^-$ in the structures of compounds (a) **I** and (b) **II**, respectively. Hydrogen atoms are omitted; one of the cations (a) La^{3+} and (b) Gd^{3+} is shown as a polyhedron.

$11H_2O$ [18], which is the first representative of coordination compounds based on $[Re_4Q_4(CN)_{12}]^{4-}$, Ln^{3+}, and organic ligand L. The applicability of the hydrothermal method for the preparation of crystalline phases of similar compounds was studied in this work. Compounds **I** and **II** were obtained by the storage of an X-ray amorphous precipitate formed by the pouring together of aqueous solutions of the corresponding salts and Phen in an acidified mother liquor at 120°C. It is most likely that the elevated temperature and an acidic medium favored an increase in the solubility of the precipitate, which resulted in its partial recrystallization. The mother liquor remained colorless after cooling, indicating the quantitative binding of cluster anions in the solid products. The yields of the reactions are relatively low, but a change in the pH, temperature, or storage time does not increase the yields. Similar reactions involving other rare-earth metal cations did not result in the formation of polymeric crystalline products, which requires, most likely, a more thorough selection of the synthesis conditions.

The spatial structures of the cluster anions $[Re_4Te_4(CN)_{12}]^{4-}$ in compounds **I** and **II** are similar to the structure of the anion in the initial salt. The main bond lengths are well consistent with the corresponding interatomic distances in other compounds containing this cluster anion [13, 18].

The independent part of the structure of compound **I** contains the cluster anion $[Re_4Te_4(CN)_{12}]^{4-}$, cationic fragment $\{La(H_2O)_3(Phen)_2\}^{3+}$, three solvate Phen molecules, and six partially occupated positions of solvate H_2O molecules. All atoms are in the general positions. The compound has a polymer structure based on 1D anionic chains $[\{La(H_2O)_3(Phen)_2\}\{Re_4Te_4(CN)_{12}\}]^-$ (Fig. 1a). The chains consist of alternating fragments $[Re_4Te_4(CN)_{12}]^{4-}$ and $\{La(H_2O)_3(Phen)_2\}^{3+}$ linked by the bridging CN ligands. The cationic fragments do not compensate all the negative charge of the cluster anions. In addition, according to the EDS data, compound **I** contains no potassium cations in the structure. Taking into account that an acidic medium was formed for the synthesis of the compounds, we assumed that one or several solvate Phen molecules were protonated at the nitrogen atoms. The analysis of hydrogen bonds revealed that only one solvate Phen molecule formed a short contact (N···O 2.48 Å) between one of the N atoms and the adjacent water molecule, indicating the presence of an acidic proton on this nitrogen atom.

Each La^{3+} cation is coordinated by two N(CN) atoms, three O atoms of the H_2O molecules, and four N atoms of two Phen molecules. Thus, the coordination number of the La^{3+} cation is 9, and the coordination polyhedron is a one-capped square antiprism. Three H_2O molecules and two Phen molecules form opposite faces of the polyhedron. This coordination mode results in the formation of surfaces with hydrophobic and hydrophilic characters on the opposite sides of the polymer chain. The hydrophilic surface is formed by the coordinated H_2O molecules and CN ligands of the cluster anions. The hydrophobic surface is formed by the coordinated Phen molecules, which overlap closely arranged CN groups due to the volume of the Phen molecules and chain geometry. Owing to this effect, the polymer chains are packed in the structure along the *x* axis in pairs and parallel, orienting to each other by hydrophilic sides and forming hydrogen bonds N(CN)···H–O(La) between the cyano ligands and water molecules coordinated to the La^{3+} cations. A high number of these hydrogen bonds (two bonds per cluster anion) and their strength (N···O 2.73–2.78 Å) result in the binding of the polymer chains to each other.

The Phen molecules are coordinated to the La^{3+} cations in such a way that π–π interactions cannot be formed between their π systems. This is compensated by the fact that each of the coordinated Phen molecules participates in the π–π-stacking interaction with the closely arranged solvate Phen molecules remote almost parallel at a distance of 3.4–3.7 Å and in the C–H···π interactions with the coordinated Phen molecules of the adjacent perpendicular chains. Thus, the developed network of π–π and C–H···π interactions plays a substantial role in the stabilization of the packing.

The packing of compound **I** (Fig. 2) contains cavities of the channel type arranged along the crystallographic *x* axis. The center of the cavity coincides with the vector (*x*, 1/2, 1/2). The channels have a rectangular cross section with sizes of 10.8 × 5.4 Å (with allowance for ionic radii of the surrounding atoms) and a volume of 650 $Å^3$, which is about 25% of the total cell volume. A characteristic feature of the channels is the presence of hydrophobic and hydrophilic surfaces of the internal walls. The hydrophobic surfaces are formed by the coordinated and solvate Phen molecules, and the hydrophilic surfaces are formed by the CN groups of the cluster anions. This property of the internal surfaces of the channels makes them suitable for the inclusion of amphiphilic molecules (ROH, RNH_2). In the structure of compound **I**, the channels are filled by solvate Phen and the molecules packed in piles of three molecules and by the disordered H_2O molecules forming hydrogen bonds with the CN groups of the cluster anions with the $PhenH^+$ cations.

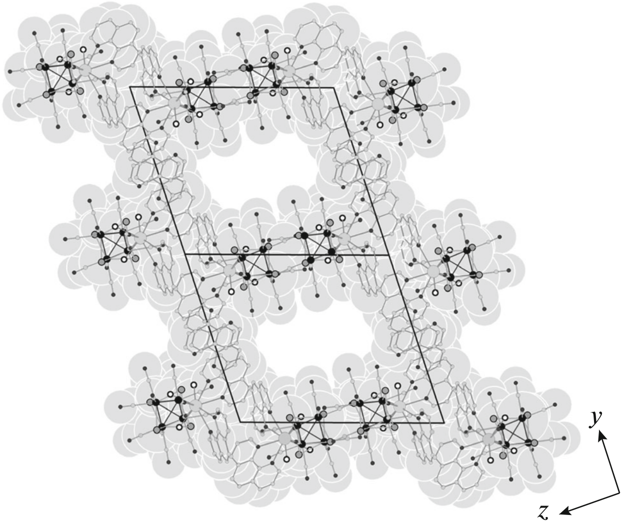

Fig. 2. Packing of the anionic chains $[\{La(H_2O)_3(Phen)_2\}\{Re_4Te_4(CN)_{12}\}]^-$ in the structure of compound **I** in van der Waals radii (projection onto the *yz* plane). The solvate molecules Phen and H_2O filling the cavities of the structure and the $PhenH^+$ cations are omitted.

The cluster anions and cationic fragment $\{Gd(H_2O)_2(Phen)_2\}^{3+}$ in structure **II** have the point symmetry 2. Compound **II** has a polymer structure based on the anionic chains $[\{Gd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}]^-$ (Fig. 1b) organized by the alternation of the cluster anions $[Re_4Te_4(CN)_{12}]^{4-}$ and cationic fragments similarly to the chains in the structure of compound **I** (Fig. 1a). The Gd^{3+} cation has a coordination number of 8 due to a smaller ionic radius compared to that of La^{3+}. Its coordination environment consists of two N(CN) atoms, two O atoms of the H_2O molecules, and four N atoms of two Phen molecules. The coordination polyhedron of the Gd^{3+} ion is a square antiprism. Two H_2O molecules and two Phen molecules lie on the opposite faces of the polyhedron, resulting in the formation of the surfaces with the hydrophobic and hydrophilic characters on the opposite sites of the polymer chain. As in compound **I**, in compound **II** the polymer chains are packed in pairs and parallel, orienting to each other by the hydrophilic sides and forming hydrogen bonds N(CN)···H–O(Cd) (N···O 2.73 Å between the cyano ligands and water molecules coordinated to the Gd^{3+} cations.

A smaller number of substituents and a higher symmetry of the cationic fragment $\{Gd(H_2O)_2(Phen)_2\}^{3+}$ compared to $\{La(H_2O)_3(Phen)_2\}^{3+}$ result in some differences between the structures of compounds **II** and

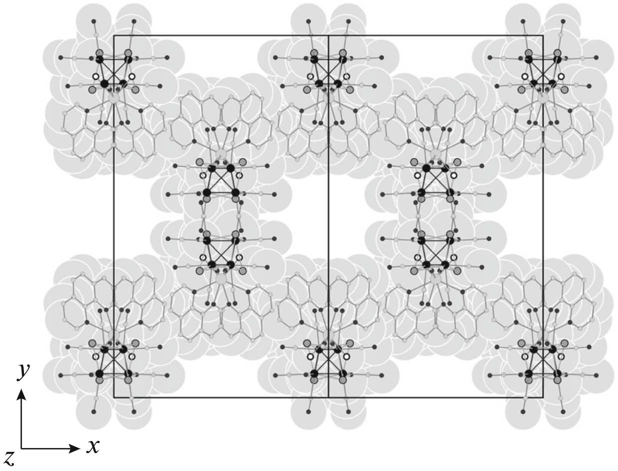

Fig. 3. Packing of the anionic chains $[\{Gd(H_2O)_2(Phen)_2\}\{Re_4Te_4(CN)_{12}\}]^-$ in the structure of compound **II** in van der Waals radii (projection onto the *xy* plane). The solvate molecules Phen and H_2O filling the cavities of the structure and the $PhenH^+$ cations are omitted.

I, respectively. The most important difference is a change in the relative orientation of two Phen molecules coordinated to the Ln^{3+} cations. In compound **I**, these molecules are almost perpendicular to each other, preventing the π–π interaction between them. At the same time, in compound **II**, the dihedral angle between the planes of the Phen molecules is only 25°, which leads to a possibility of the π–π-stacking interaction taking into account their partial overlap and a distance of 3.7 Å between the centroids of the rings. As a result, in the packing of compound **I** the dimers of two polymer chains were linked through the C–H···π interactions between the perpendicular Phen molecules of the adjacent chains, whereas in compound **II** these dimers are packed due to the π–π-stacking interactions between the parallel Phen molecules of the adjacent chains and the solvate Phen molecules.

The packing of compound **II** (Fig. 3) contains cavities of the channel type arranged along the crystallographic *z* axis. The center of the cavity coincides with the vector (0, 1/2, *z*). The channels have a rectangular cross section with sizes of 7.4 × 7.1 Å (with allowance for ionic radii of the surrounding atoms) and a volume of approximately 1060 $Å^3$, being ~15% of the total cell volume. As in the structure of compound **I**, the characteristic feature of the channels in compound **II** is the presence of the hydrophobic and hydrophilic surfaces of the internal walls. The hydrophobic surfaces are formed by the coordinated and solvate Phen molecules, and the hydrophilic surfaces are formed by the CN groups of the cluster anions. In the structure of compound **II**, the channels are completely filled by the disordered solvate phenanthroline molecules and $PhenH^+$ cations packed into piles of two molecules.

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