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GADOLINIUM BREAK IN A SERIES OF THREE-DIMENSIONAL *TRANS*-1,4-CYCLOHEXANE DICARBOXYLATES OF RARE EARTH ELEMENTS

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By a reaction of lanthanide nitrates with *trans*-1,4-cyclohexane dicarboxylic acid H_2chdc in an aqueous medium new coordination polymers with the composition [$Ln_2(H_2O)_4(chdc)_3$] (Ln = Ce (**1**), Sm (**2**), Eu (**3**), Gd (**4**)) are obtained. Crystallization conditions are optimized with the use of a hexamine-based pH buffer. Compound **1** is additionally characterized by powder X-ray diffraction, elemental and thermogravimetric analyses, and IR spectroscopy. The structure of the obtained compounds is determined by single crystal X-ray diffraction. In a series of isostructural three-dimensional frameworks [$Ln_2(H_2O)_4(chdc)_3$] (Ln = La–Eu, except Pm) a regular change in the structural features is analyzed. The [$Gd_2(H_2O)_4(chdc)_3$] compound has a similar composition but the topology of the three-dimensional network differs from that of the previous ones due to a decrease in the coordination number of metal centers. Heavier lanthanides, starting from Tb^{3+}, and also yttrium(III) do not form compounds with the mentioned composition under similar reaction conditions.

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INTRODUCTION

The chemistry of metal-organic frameworks (MOFs) has been extensively developed for the last 20 years. Significant progress in the synthesis of MOFs, which has been reached at presence, enables the preparation of various crystalline products, including polymorphs, for a wide range of metal–ligand combinations [1-5]. Up to date, it is of interest to obtain a series of isostructural MOFs with a variable composition and crystal parameters, which enable the fine tuning of the physicochemical properties of compounds for a particular application. *Trans*-1,4-cyclohexane dicarboxylic acid (H_2chdc) is a close structural analogue of terephthalic acid but has an alicyclic (nonaromatic) nature. Owing to this, MOFs based on $chdc^{2-}$ bridges are UV transparent [6] and exhibit a minimum luminescent activity, which, in particular, allows the study of the photophysical properties of lanthanide (Ln) cations contained in these coordination frameworks. Note that less than ten structurally characterized *trans*-1,4-cyclohexane dicarboxylates have been known so far for the entire series of lanthanides

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[7-12], with none sufficiently representative series of isostructural compound being obtained. Thus, the synthesis of isostructural MOFs in Ln–H_2chdc systems, the crystal structure study, and the analysis of systematic structural regularities in these compounds is a topical problem.

The synthesis and structure of three isostructural coordination polymers [$Ln_2(H_2O)_4$(chdc)$_3$] (Ln = La^{3+} [11], Pr^{3+} [7], Nd^{3+} [12]) were discussed in the literature. We aimed at expanding this series of compounds and characterizing in detail their structural distinctions. In this work, we report the synthesis and single crystal X-ray diffraction study of four novel [$Ln_2(H_2O)_4$(chdc)$_3$] compounds for Ln = Ce^{3+}, Sm^{3+}, Eu^{3+}, Gd^{3+} and analyze the structural features of this family of compounds.

EXPERIMENTAL

All starting compounds were used as commercially available reagents without additional purification.

IR spectra were measured in the range 4000-400 cm^{-1} in pellets with KBr on a Scimitar FTS 2000 Fourier spectrometer. The thermogravimetric analysis (TGA) was performed on a NETZSCH TG 209 F1 thermoanalyzer during the linear heating of the samples in the He atmosphere with a rate of 10 deg/min. The elemental analysis was conducted on a VarioMICROcube CHNS-analyzer. Powder X-ray diffraction data were obtained on a Shimadzu XRD 7000S powder diffractometer (CuK_α radiation, λ = 1.54178 Å).

Synthesis of *catena*-[(μ$_2$-*trans*-cyclohexane-1,4-dicarboxylato)-bis-((μ$_4$-*trans*-cyclohexane-1,4-dicarboxylato) tetraqua dicerium(III)] [$Ce_2(H_2O)_4$(chdc)$_3$] (1). Single crystals of **1** were obtained by heating a mixture of 10.0 mg (0.023 mmol) of cerium(III) nitrate hexahydrate, 8.0 mg (0.047 mmol) of *trans*-1,4-cyclohexane dicarboxylic acid, 14.0 mg (0.10 mmol) of hexamine, 20 μl (0.21 mmol) of perchloric acid, and 2 ml of water in a sealed glass tube at 130 °C for 12 h. The product structure was determined by single crystal X-ray diffraction. The representative series of **1** were synthesized by scaling the above reaction conditions in a stainless steel bomb with a Teflon liner and a screw cap with a 20-fold increase in the amount of all reagents. The colorless fine precipitate obtained was filtered on a porous paper filter, washed with water (2×20 ml), then acetone (5 ml), and dried in air for 5 min. The yield was 119 mg (60%). IR spectrum, ν, cm^{-1}: 3591 (w), 3350 (m, broad), 2935 (s), 2860 (m), 1529 (s), 1428 (s), 1360 (m), 1329 (m), 1283 (s), 1244 (w), 1214 (m), 1135 (w), 1107 (w), 1055 (w), 1039 (w), 926 (m), 901 (w), 886 (w), 801 (m), 781 (m), 766 (m), 742 (m), 624 (m), 541 (m), 522 (m), 473 (s). Found (%): C 33.3, H 4.2. [$Ce_2(H_2O)_4$(chdc)$_3$] ($C_{24}H_{38}O_{16}Ce_2$). Calculated (%): C 33.4, H 4.4. TGA: weight loss in the range 100-180 °C Δm = 8.4% (calculation per $4H_2O$ = 8.3%).

Synthesis of *catena*-[(μ$_2$-*trans*-cyclohexane-1,4-dicarboxylato)-bis-((μ$_4$-*trans*-cyclohexane-1,4-dicarboxylato) tetraqua disamarium(III)] [$Sm_2(H_2O)_4$(chdc)$_3$] (2), *catena*-[(μ$_2$-*trans*-cyclohexane-1,4-dicarboxylato)-bis-((μ$_4$-*trans*-cyclohexane-1,4-dicarboxylato)tetraqua dieuropium(III)] [$Eu_2(H_2O)_4$(chdc)$_3$] (3), and *catena*-[(μ$_2$-*trans*-cyclohexane-1,4-dicarboxylato)-bis-((μ$_4$-*trans*-cyclohexane-1,4-dicarboxylato)tetraqua digadolinium(III)] [$Gd_2(H_2O)_4$(chdc)$_3$] (4). The ampoule synthesis of single crystals of compounds **2-4** was performed similarly to the synthesis of **1**, with cerium nitrate being replaced by ~11 mg (0.023 mmol) of hexahydrates of respective metals. The structure and composition of the products were established by X-ray crystallography.

The crystals of [$La_2(H_2O)_4$(chdc)$_3$], [$Pr_2(H_2O)_4$(chdc)$_3$], [$Nd_2(H_2O)_4$(chdc)$_3$] compounds whose structure was described in the literature [7, 11, 12] can also be obtained by a similar procedure with the use of 10-11 mg (0.023-0.025 mmol) of respective hexahydrates of lanthanide nitrates.

X-ray crystallographic study. Single crystal diffraction data for compounds **1-4** were obtained at 130 K on an automated Agilent Xcalibur diffractometer equipped with an AtlasS2 area detector (graphite monochromator, λ(MoK_α) = 0.71073 Å, ω-scanning). Integration, absorption correction, and determination of unit cell parameters were performed using the CrysAlisPro program package [13]. Crystal structures were solved using the SHELXT program [14] and refined by the full-matrix least-squares technique in the anisotropic (except hydrogen atoms) approximation using the

TABLE 1. Crystallographic Parameters and Details of the Single Crystal X-ray Diffraction Experiment

Parameter	**1**	**2**	**3**	**4**
Chemical formula	$C_{24}H_{38}Ce_2O_{16}$	$C_{24}H_{38}Sm_2O_{16}$	$C_{24}H_{38}Eu_2O_{16}$	$C_{24}H_{38}Gd_2O_{16}$
M, g/mol	862.78	883.24	886.46	897.04
Crystal system	Triclinic	Triclinic	Triclinic	Triclinic
Space group	P–1	P–1	P–1	P–1
a, Å	11.4381(5)	11.3215(8)	11.3175(4)	11.2799(6)
b, Å	11.9870(3)	11.8611(6)	11.7594(5)	11.8431(7)
c, Å	12.2501(6)	12.1359(7)	12.2254(6)	12.5654(8)
α, deg	95.000(3)	94.847(4)	93.917(4)	84.384(5)
β, deg	115.359(4)	115.309(6)	115.627(4)	118.227(6)
γ, deg	101.371(3)	101.100(5)	101.033(3)	98.716(5)
V, Å^3	1459.78(11)	1419.90(16)	1418.73(11)	1461.24(17)
Z	2	2	2	2
D_{calc}, g/cm^3	1.963	2.066	2.075	2.039
μ, mm^{-1}	3.154	4.172	4.457	4.574
$F(000)$	852	868	872	876
Crystal dimensions, mm	0.15×0.05×0.05	0.35×0.07×0.06	0.13×0.12×0.05	0.09×0.06×0.05
θ scanning range, deg	3.34-29.56	3.39-29.51	3.36-29.48	3.28-29.39
hkl index range	$-16 < h < 15$, $-13 < k < 16$, $-15 < l < 16$	$-15 < h < 16$, $-17 < k < 17$, $-17 < l < 14$	$-16 < h < 13$, $-16 < k < 14$, $-17 < l < 17$	$-11 < h < 16$, $-17 < k < 16$, $-18 < l < 14$
N_{hkl} meas. / indep.	12168 / 6834	11683 / 6620	12980 / 6633	12545 / 6718
R_{int}	0.0230	0.0272	0.0293	0.0262
N_{hkl} c $I > 2\sigma(I)$	5765	5781	5687	5682
GOOF on F^2	1.025	1.065	1.197	1.009
R-factor $[I > 2\sigma(I)]$	$R_1 = 0.0250$, $wR_2 = 0.0448$	$R_1 = 0.0281$, $wR_2 = 0.0624$	$R_1 = 0.0385$, $wR_2 = 0.0633$	$R_1 = 0.0253$, $wR_2 = 0.0403$
R-factor (over all reflections)	$R_1 = 0.0351$, $wR_2 = 0.0470$	$R_1 = 0.0346$, $wR_2 = 0.0650$	$R_1 = 0.0490$, $wR_2 = 0.0656$	$R_1 = 0.0355$, $wR_2 = 0.0424$
Residual electron density (max / min), e/Å^3	0.761 / –0.633	1.719 / –0.924	1.427 / –1.555	0.731 / –0.877

SHELXL program [15]. Positions of hydrogen atoms of organic ligands were calculated geometrically and refined in the riding model. Crystallographic data and details of the diffraction experiments are summarized in Table 1. Complete tables of interatomic distances and bond angles, atomic coordinates and displacement parameters have been deposited with the Cambridge Crystallographic Data Center (CCDC 1876555-1876558) and can also be received from the authors.

RESULTS AND DISCUSSION

The hydrothermal synthesis of metal-organic coordination polymers is often used for metal cations forming carboxylate complexes that are stable in an aqueous solution. To enhance the product crystallinity modulators such as HF [16, 17], formic and acetic acids [18-20] have often been used. The role of a modulator can be both change in the medium acidity and specific interactions slowing down the major reaction by, e.g., partial reversible coordination of metal centers. The Ln^{3+} ions having a pronounced oxophilic nature are not expected to form stable complexes with nonchelating N-donor ligands in an aqueous solution, therefore we applied a mixture of hexamine and noncoordinating perchloric acid, in which hexamine is in protonated forms, as the acidity modulator. This partially protonated hexamine represents a buffer system maintaining an almost constant acidity in the reaction solution during the MOF formation. It is worth noting that at high temperatures hexamine can undergo hydrolysis resulting in contaminating the crystalline product with ammonium perchlorate. Thus, the temperature and time of the synthesis of MOFs **1-4** were optimized with regard to the parallel hydrolysis reaction.

According to the X-ray crystallography data, compound **1** crystallizes in the triclinic crystal system with the *P*–1 space group. The asymmetric unit of the structure of **1** contains two Ce^{3+} cations, three cyclohexane dicarboxylic acid anions, and four water molecules. The Ce(1) atom is in the coordination environment composed of six oxygen atoms of three carboxyl groups chelating Ce(1), one bridging O atom of the carboxyl group chelating Ce(2), one oxygen atom of the Ce(1)–Ce(1) bridge, and two oxygen atoms of aqua ligands. The Ce(1)–O bond lengths are within 2.4816(18)–2.7641(18) Å. Coordination number of Ce(1) is 10. The coordination environment of Ce(2) consists of four oxygen atoms of two carboxyl groups chelating Ce(2), two bridging O atoms of the carboxyl groups chelating Ce(1), one bridging O atom of the monodentately coordinated carboxyl group, and two oxygen atoms of aqua ligands. The Ce(2)–O bond lengths are within 2.4846(19)-2.6838(17) Å. Coordination number of Ce(2) atoms is 9. Thus, in the structure of **1** it is possible to reveal the secondary building block $\{Ce_2(\mu\text{-}RCOO\text{-}\kappa^1,\kappa^2)_2(H_2O)_4(RCOO\text{-}\kappa^1)_3(RCOO\text{-}\kappa^2)_3\}$ (Fig. 1*a*). These blocks are linked with the neighboring ones through additional bridging interactions Ce–O(μ-RCOO) with the formation of infinite one-dimensional chains $\{–O–Ce(1)–O–Ce(1)–O–Ce(2)–O–Ce(2)–O–\}_\infty$ (Fig. 2), which are very typical of lanthanides. Through the carboxyl groups of the bridging aliphatic dicarboxylate ligand $chdc^{2-}$ the chains are organized into a three-dimensional framework (Fig. 3). Compound **1** is the first described cerium(III) 1,4-cyclohexane dicarboxylate.

Powder X-ray diffraction (PXRD) data for powder **1** completely correspond to the theoretically calculated diffraction pattern and demonstrate the phase purity of the obtained compound. TGA and elemental (C, H, N) analysis results are consistent with the chemical formula $[Ce_2(H_2O)_4(chdc)_3]$ determined by X-ray crystallography. The IR spectrum of compound **1** contains the characteristic absorption bands for CO bonds of the ligand carboxyl group at 1529 cm^{-1} and 1283 cm^{-1} (antisymmetric and symmetric stretching vibrations respectively), O–H bonds of water molecules at 3350 cm^{-1}, and C(sp^3)–H bonds of aliphatic fragments at 2935 cm^{-1} and 2860 cm^{-1}.

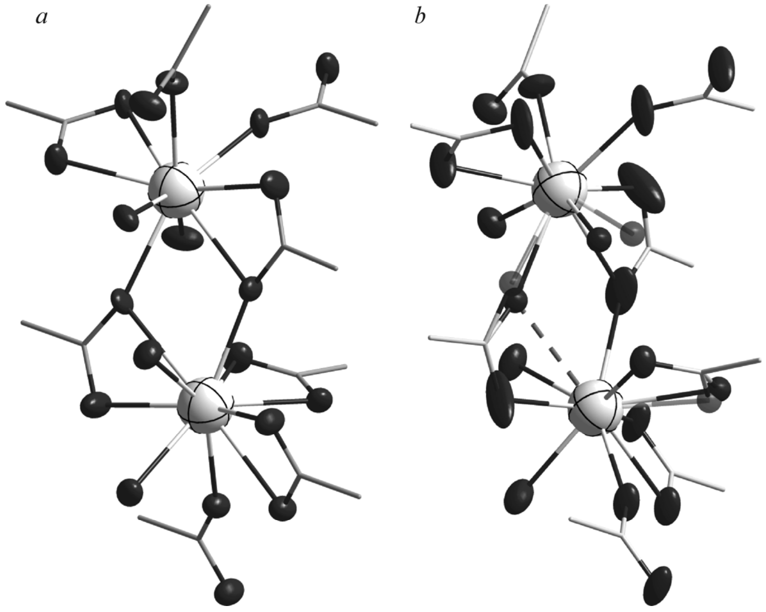

Fig. 1. Secondary building block in **1-3** (*a*) and bonding type between the secondary building blocks in **3** (*b*). Oxygen atoms are shown by 90% probability ellipsoids. Hydrogen atoms of water molecules are not shown. The dashed line shows an increased distance Eu (CN = 9+1) – O_{COO} (2.918(11) Å). Disordered oxygen atoms are depicted as spheres; the second positions are semitransparent.

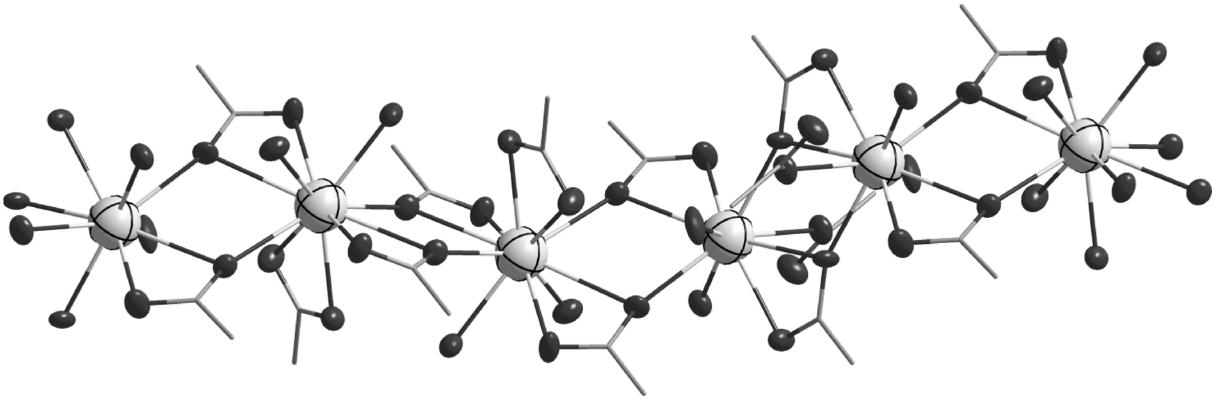

Fig. 2. Metal-carboxylate chain in **1** and **2**. Oxygen atoms are shown by 90% probability ellipsoids. Hydrogen atoms of water molecules are not shown.

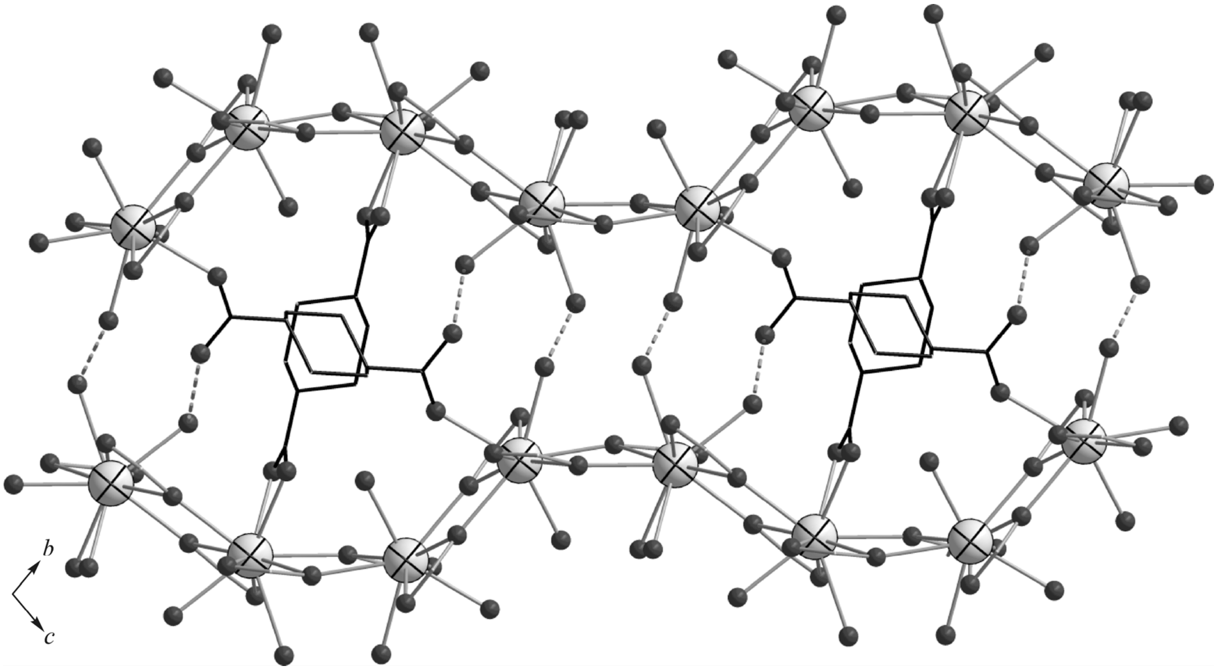

Fig. 3. Association of metal-carboxylate chains in **1-3**. Black color marks interchain ligands chdc^{2-}, dashed lines show hydrogen bonds between O atoms. Hydrogen atoms are omitted for clarity.

Compounds **2** and **3** are isostructural to **1**. The Sm(1)–O distances are within 2.408(2)-2.749(3) Å, Sm(2)–O are within 2.421(2)-2.649(3) Å. The Eu(1)–O bonds are within 2.281(6)-3.380(7) Å, Eu(2)–O are within 2.288(5)-2.744(4) Å. It should be noted that in the analysis of the crystal structure of **3** the disorder of oxygen atoms of one of the aqua ligands and two bridging COO groups is revealed with an elongation of one of the Eu(1)–O(μ-RCOO) distances to 2.918(11) Å and 3.380(7) Å, which allows Eu(1) CN to be taken as 9+1 or 9 (Fig. 1*b*). Thus, two types of bonding between the secondary building blocks compete in compound **3**.

Compound $[Gd_2(H_2O)_4(chdc)_3]$ (**4**) contains two types of secondary building blocks that are similar in the composition to those in above described compounds **1-3**: $\{Gd_2(\mu\text{-}RCOO\text{-}\kappa^1,\kappa^2)_2(H_2O)_4(RCOO\text{-}\kappa^2)_2(RCOO\text{-}\kappa^1)_4\}$. The first type is based on Gd(1) cations (Fig. 4*a*), the second is based on Gd(2) cations (Fig. 4*b*). The Gd(1)–O distances are within 2.325(2)-2.633(2) Å, the Gd(2)–O distances are within 2.300(2)-2.633(2) Å. A difference between these two types of the building blocks consists in the coordination mode of the bridging carboxylate ligands (μ-RCOO-κ^1,κ^2). For Gd(2) the Gd(2)–μ-O(RCOO) distances are 2.423(2) Å and 2.633(2) Å, whereas for Gd(1) one of the distances is much longer. The Gd(1)–μ-O(RCOO) distances are 2.325(2) Å and 3.216(2) Å. Thus, although the general structural motif of the three-dimensional

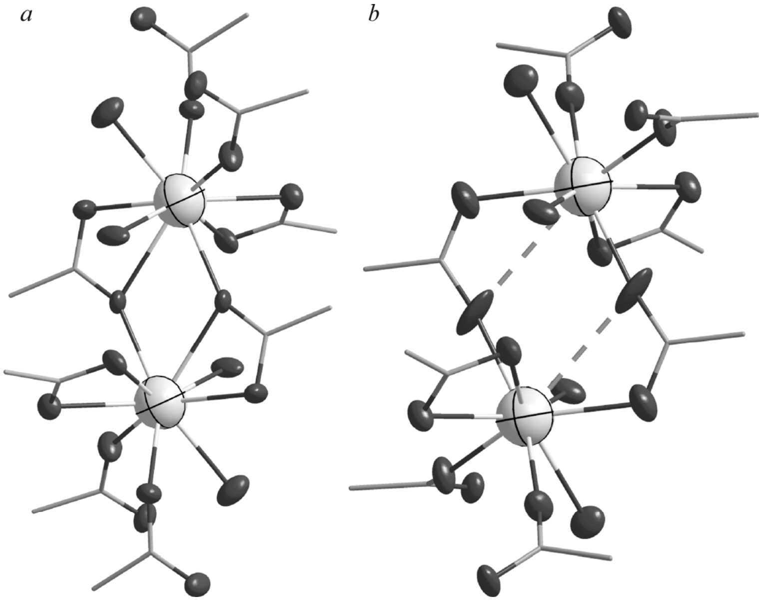

Fig. 4. Two secondary building blocks in **4** Gd (CN = 9) (*a*) and Gd (CN = 8) (*b*). Oxygen atoms are shown by 85% probability ellipsoids. Hydrogen atoms of water molecules are not shown. The dashed line depicts an increased distance Gd (CN = 8) – O_{COO} (3.216(2) Å).

framework is retained (Fig. 5) compound **4** is not isostructural to the other compounds of the series and contains metal centers with CN = 9 (Gd(2)) and 8+1 (Gd(1)). The structural features of compounds **1-4** and the similar compounds with La, Pr, and Nd described previously are combined in Table 2.

The analysis of the listed parameters shows a regular decrease in the unit cell parameters and volume with increasing atomic number of lanthanide in the series La–Eu. The Ln^{III}–O_{H_2O} bond lengths in the coordination sphere of metal centers

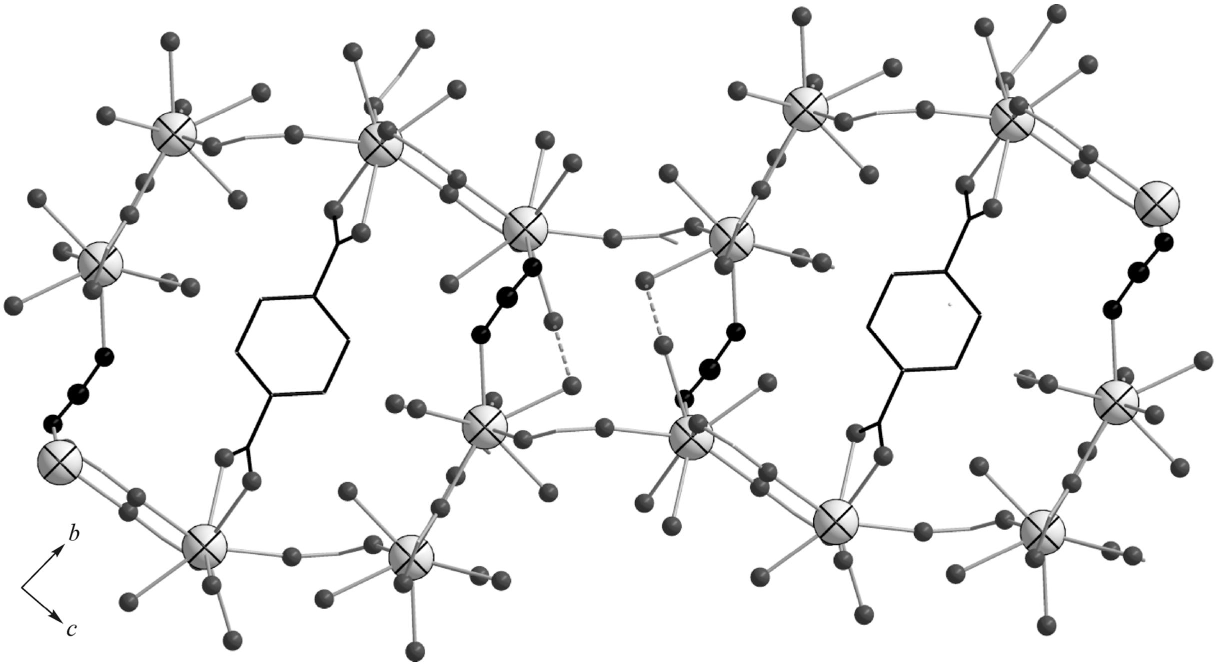

Fig. 5. Association of metal-carboxylate chains in **4**. Black color marks interchain ligands chdc^{2-} and carboxyl groups; dashed lines show hydrogen bonds between coordinated water molecules. Hydrogen atoms are omitted for clarity.

TABLE 2. Selected Interatomic Distances in $[Ln_2(H_2O)_4(chdc)_3]$ Compounds

Ln^{3+}	La	Ce (**1**)	Pr	Nd (273 K)	Sm (**2**)	Eu (**3**)	Gd (**4**)
Distance M(CN = 9)–O_{H_2O}, Å	2.5342(34) 2.5883(37)	2.499(2) 2.5519(19)	2.4725(25) 2.4804(23)	2.4958(49) 2.5499(52)	2.422(3) 2.478(2)	2.411(10) 2.442(7) 2.466(3)	2.4330(19) 2.510(2)
Distance Ln–Ln, Å	4.4090(4) 4.4797(4) 4.5445(4)	4.3339(4) 4.4093(4) 4.4999(4)	4.3204(8) 4.4545(11) 4.5113(12)	4.3451(6) 4.4641(6) 4.5204(6)	4.2643(4) 4.3820(4) 4.4827(4)	4.1622(4) 4.4032(4) 4.5518(4)	4.3129(4) 4.6132(5)
Reference	[11]	[This work]	[7]	[12]	[This work]	[This work]	[This work]

also decrease as expected as well as the Ln–Ln distances inside the secondary building blocks $\{Ln_2(H_2O)_4(OOC)_6\}$. These changes seem to be caused by a decrease in the radius of Ln^{3+} ions (the so-called lanthanide contraction) and the concomitant increase in their Lewis acidity. For the Gd-based compound the monotonic change in the unit cell parameters is broken due to a change in the topology of the three-dimensional framework, which is caused by a decrease in CN of metal centers and a change in the coordination mode of carboxyl groups.

The preparation of a series of isostructural coordination polymers based on lanthanides made it possible to reveal a relationship between their stability and Ln^{3+} ionic radii. Thus, for europium a significant weakening (elongation) of the coordination bond Ln (CN = 10)–O_{COO} with a small lattice extension along the respective crystallographic direction are observed. For gadolinium the non-stability of CN 10 results in the formation of a non-equivalent structure containing metal centers with CNs 8 and 9 and similar parameters. The attempt to obtain similar $[Ln_2(H_2O)_4(chdc)_3]$ compounds for heavier lanthanides (starting from Tb^{3+}) caused only the crystallization of ammonium perchlorate from the hydrothermal decomposition of hexamine. This is most likely to be associated with the so-called gadolinium break, i.e. a decrease in CN of the Ln^{3+} cation for heavy lanthanides to 8, and consequently, the impossibility of the formation of the polymer coordination structure $[Ln_2(H_2O)_4(chdc)_3]$ containing Ln^{3+} ions with CNs 9 and 10. For the same reasons the attempt to synthesize a similar compound with yttrium(III) $[Y_2(H_2O)_4(chdc)_3]$ failed.

CONCLUSIONS

Thus, we synthesized and structurally characterized four novel coordination polymers based on Ln^{3+} ions and alicyclic *trans*-1,4-cyclohexane dicarboxylic acid. For the obtained series of isostructural coordination polymers $[Ln_2(H_2O)_4(chdc)_3]$ (Ln = La–Nd, Sm, Eu) regular changes in Ln(CN = 9)–O_{H_2O} bond lengths, Ln–Ln distances, and crystal cell parameters are analyzed. The stability boundaries of the $[Ln_2(H_2O)_4(chdc)_3]$ structure type are determined. A significant distinction between the $[Gd_2(H_2O)_4(chdc)_3]$ structure and analogues based of lighter lanthanides and the impossibility to crystallize a product for rare earth elements with a smaller ionic radius and characteristic CN = 8: Ln ≥ Tb^{3+} and Y^{3+} are shown.

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CONFLICT OF INTERESTS

The authors declare that they have no conflict of interests.

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