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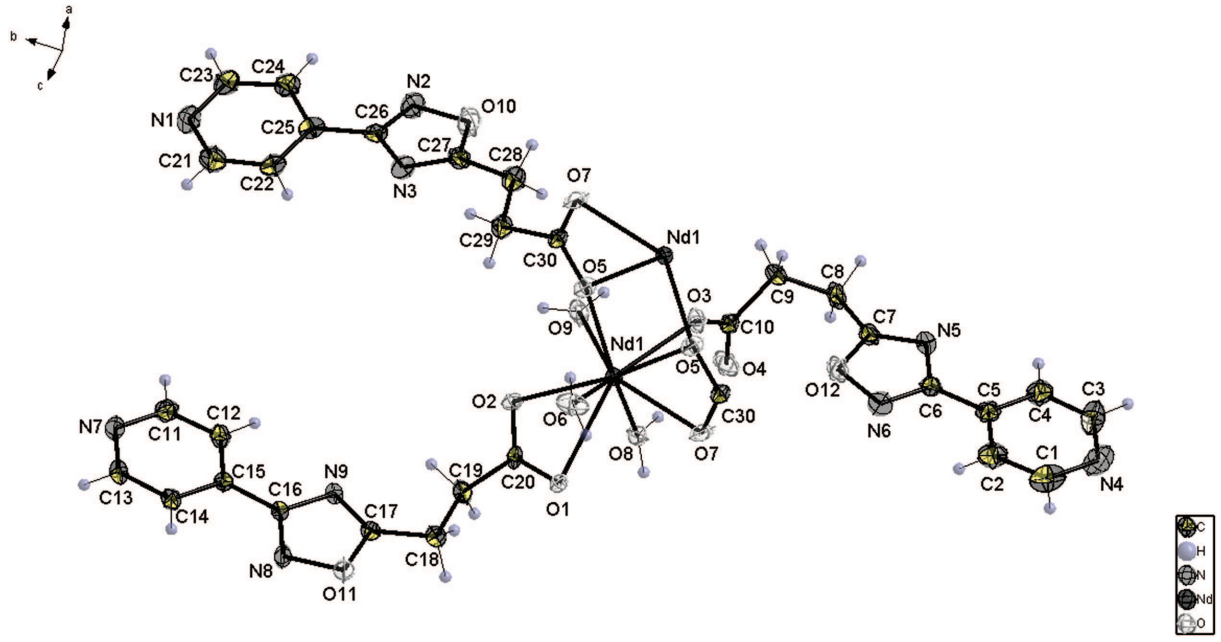
Crystal structure of hexaaqua-bis(3-(3-pyridin-4-yl-[1,2,4]oxadiazol-5-yl) propionato-κ^3O,O′:O′)-bis(3-(3-pyridin-4-yl-[1,2,4]oxadiazol-5-yl)propionato-κO)-bis(3-(3-pyridin-4-yl-[1,2,4]oxadiazol-5-yl)propionato-κ^2O,O′) dineodymium(III) octahydrate, $C_{60}H_{76}N_{18}O_{32}Nd_2$



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Abstract

$C_{60}H_{76}N_{18}O_{32}Nd_2$, monoclinic, $P2_1/c$ (no. 14), $a = 10.609(5)$ Å, $b = 31.215(5)$ Å, $c = 12.407(4)$ Å, $\beta = 116.54(3)°$, $V = 3676(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0450$, $wR_{ref}(F^2) = 0.0910$, $T = 293(2)$ K.

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A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

3-(3-Pyridin-4-yl-[1,2,4]oxadiazol-5-yl)-propionic acid (*HL*) is easily available by a literature known synthesis [1]. A solution

Table 1: Data collection and handling.

Crystal:	Black strip
	Size 0.30 × 0.21 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	15.0 cm^{-1}
Diffractometer, scan mode:	Bruker FRAMBO, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13629, 6487, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5973
$N(param)_{refined}$:	485
Programs:	Bruker programs [8], SHELX [9]

of ammonia (0.5 M) was added dropwise to a methanol (15 mL) solution of HL (3 mmol), resulting in a transparent solution. A methanol (15 mL) solution of acetate dihydrate (1.5 mmol) was added into the resulting solution and stirred for 3 h, resulting in a suspension. A methanol (15 mL) solution of neodymium chloride hexahydrate (1 mmol) was added into the resulting suspension and stirred for 8 h. The suspension was filtered and diethyl ether was allowed to diffuse slowly into the solution of the filtrate. Black crystals were obtained in about 3 weeks.

Experimental details

H atoms bound to C atoms were placed in calculated positions and treated as mixed on their parent atoms, with C—H = 0.93 or 0.97 Å and $U_{iso}(H) = 1.2$ or 1.5 $U_{eq}(C)$. H atoms of water molecule were initially found in the difference Fourier map and were refined with restraint as O—H = 0.85 Å.

Discussion

Lanthanide-based carboxylate complexes have been found to exhibit anticancer, unusual coordination characteristics, exceptional optical, magnetic properties [2–4]. They are used as precursors for oxides [5] and may possess fungicidal properties [6]. Lanthanide nitrates have been previously used in the synthethic protocol of such complexes, while little attention has been paid to the use of lanthanide chloride [7]. As a part of our ongoing investigations we report the preparation and crystal structure of the title compound. In the asymmetric unit of title structure, there are four lattice water molecules, and three organic ligands adopting a monodentate mode a chelating mode and a chelating bidentate coordination mode, respectively. Two neighboring Nd^{III} ions form a dimeric structural unit with the Nd1—Nd1 distance of 4.2658(21) Å bridged by two oxygen atoms of two carboxylates. Each Nd(III) is coordinated by three water ligands and six oxygen atoms of four carboxylate ligands, exhibiting a tricapped trigonal prism geometry (see the figure, lattice water molecules are removed for clarity). The Nd—O bond lengths, depending on the nature of the O-atoms, vary from 2.4152(4) to 2.6839(4) Å. The

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.6989(6)	−0.3015(2)	0.8046(6)	0.0537(17)
H1	0.7121	−0.3142	0.8766	0.064*
C2	0.7100(6)	−0.25793(19)	0.8025(5)	0.0420(14)
H2	0.7324	−0.2418	0.8717	0.050*
C3	0.6514(6)	−0.30765(19)	0.6084(6)	0.0500(15)
H3	0.6330	−0.3246	0.5413	0.060*
C4	0.6572(5)	−0.26392(18)	0.5964(5)	0.0378(13)
H4	0.6410	−0.2519	0.5228	0.045*
C5	0.6872(5)	−0.23834(17)	0.6953(4)	0.0295(11)
C6	0.6912(5)	−0.19162(16)	0.6839(4)	0.0261(11)
C7	0.6769(5)	−0.13177(17)	0.6036(4)	0.0279(11)
C8	0.6543(6)	−0.09452(16)	0.5250(4)	0.0335(12)
H8A	0.5542	−0.0884	0.4843	0.040*
H8B	0.6837	−0.1020	0.4637	0.040*
C9	0.7319(6)	−0.05405(17)	0.5889(4)	0.0336(12)
H9A	0.7222	−0.0329	0.5285	0.040*
H9B	0.8313	−0.0607	0.6337	0.040*
C10	0.6811(5)	−0.03434(15)	0.6752(4)	0.0249(10)
C11	0.6490(5)	0.31857(17)	1.1899(4)	0.0315(12)
H11	0.6382	0.3305	1.1177	0.038*
C12	0.6351(5)	0.27499(16)	1.1953(4)	0.0287(11)
H12	0.6147	0.2582	1.1276	0.034*
C13	0.6945(5)	0.32589(16)	1.3869(4)	0.0319(12)
H13	0.7151	0.3433	1.4536	0.038*
C14	0.6836(5)	0.28278(16)	1.3997(4)	0.0283(11)
H14	0.6975	0.2714	1.4735	0.034*
C15	0.6517(5)	0.25618(15)	1.3013(4)	0.0233(10)
C16	0.6345(5)	0.21016(15)	1.3103(4)	0.0233(10)
C17	0.6016(5)	0.14545(15)	1.2637(4)	0.0257(10)
C18	0.5777(6)	0.10179(17)	1.2121(5)	0.0372(13)
H18A	0.4968	0.0894	1.2173	0.045*
H18B	0.6590	0.0842	1.2597	0.045*
C19	0.5524(5)	0.10103(16)	1.0812(4)	0.0309(11)
H19A	0.4539	0.0946	1.0307	0.037*
H19B	0.5706	0.1294	1.0597	0.037*
C20	0.6406(5)	0.06936(14)	1.0536(4)	0.0223(10)
C21	0.9737(6)	0.34149(19)	0.9283(5)	0.0437(14)
H21	0.9798	0.3498	1.0025	0.052*
C22	0.9799(5)	0.29829(17)	0.9076(4)	0.0340(12)
H22	0.9888	0.2782	0.9660	0.041*
C23	0.9544(6)	0.35950(18)	0.7443(5)	0.0379(13)
H23	0.9465	0.3803	0.6879	0.046*
C24	0.9606(5)	0.31704(17)	0.7158(4)	0.0325(12)
H24	0.9567	0.3097	0.6417	0.039*
C25	0.9728(5)	0.28547(16)	0.7988(4)	0.0270(11)
C26	0.9764(5)	0.23998(15)	0.7714(4)	0.0251(10)
C27	1.0004(5)	0.17382(17)	0.7904(4)	0.0310(11)
C28	1.0293(6)	0.12750(17)	0.8235(5)	0.0391(13)
H28A	1.1126	0.1189	0.8153	0.047*
H28B	0.9508	0.1106	0.7671	0.047*
C29	1.0518(5)	0.11743(16)	0.9495(4)	0.0316(11)
H29A	0.9647	0.1218	0.9556	0.038*
H29B	1.1226	0.1364	1.0062	0.038*
C30	1.0989(5)	0.07160(15)	0.9794(4)	0.0237(10)
N1	0.9594(5)	0.37206(15)	0.8487(4)	0.0417(11)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
N2	0.9447(5)	0.22736(14)	0.6624(4)	0.0401(11)
N3	1.0112(4)	0.20736(14)	0.8551(4)	0.0325(10)
N4	0.6704(5)	−0.32720(17)	0.7097(5)	0.0527(13)
N5	0.6687(4)	−0.17186(13)	0.5772(3)	0.0287(9)
N6	0.7137(5)	−0.16545(15)	0.7718(4)	0.0372(11)
N7	0.6772(4)	0.34472(13)	1.2838(4)	0.0325(10)
N8	0.6414(4)	0.19246(13)	1.4078(4)	0.0314(10)
N9	0.6092(4)	0.18162(13)	1.2172(3)	0.0275(9)
Nd1	0.78621(2)	0.016123(7)	0.95239(2)	0.01792(9)
O1	0.6433(3)	0.03073(10)	1.0808(3)	0.0235(7)
O2	0.7080(4)	0.08311(11)	0.9994(3)	0.0337(8)
O3	0.7747(3)	−0.01963(11)	0.7726(3)	0.0268(7)
O4	0.5524(3)	−0.03387(12)	0.6455(3)	0.0358(9)
O5	1.0151(3)	0.04437(10)	0.9874(3)	0.0258(7)
O6	0.9420(3)	0.02067(12)	1.1728(3)	0.0359(9)
H602	0.9292	0.0025	1.2169	0.043*
H601	1.0310	0.0230	1.1986	0.043*
O7	0.7809(3)	−0.06107(11)	1.0058(3)	0.0322(8)
O8	0.5322(3)	0.00401(10)	0.8326(3)	0.0269(7)
H801	0.5052	−0.0014	0.7580	0.032*
H802	0.4721	−0.0071	0.8522	0.032*
O9	0.7208(4)	0.06714(11)	0.7830(3)	0.0334(8)
H901	0.7079	0.0937	0.7867	0.040*
H902	0.7140	0.0576	0.7207	0.040*
O10	0.9605(4)	0.18269(12)	0.6738(3)	0.0443(10)
O11	0.6200(4)	0.14871(11)	1.3781(3)	0.0330(8)
O12	0.7056(4)	−0.12473(11)	0.7207(3)	0.0359(8)
O2W	0.2986(4)	−0.05661(14)	0.4456(3)	0.0492(10)*
H201	0.3658	−0.0503	0.5136	0.059*
H202	0.2824	−0.0833	0.4458	0.059*
O1W	0.6578(4)	−0.08209(15)	0.2532(4)	0.0589(11)*
H101	0.5918	−0.0652	0.2097	0.071*
H102	0.6350	−0.1071	0.2243	0.071*
O4W	1.1023(4)	−0.05369(14)	1.5509(3)	0.0519(10)*
H401	1.1069	−0.0311	1.6048	0.062*
H402	1.0675	−0.0723	1.5899	0.062*
O3W	1.1177(4)	0.02561(14)	1.6642(4)	0.0571(11)*
H302	1.0697	0.0384	1.6663	0.069*
H301	1.2036	0.0445	1.7044	0.069*

dimeric structural unit is further connected by intermolecular hydrogen bonds.

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References

1. Zhang, F.; Liu, F.; Chen, Q. F.; Dong, M. D.: 3-[3-(Pyridin-3-yl)-1, 2, 4-oxadiazol-5-yl]propanoic acid. Acta Crystallogr. **E67** (2011) o193.
2. Kido, J.; Okamoto, Y.: Organo lanthanide metalcomplexes for electroluminescent materials. Chem. Rev. **102** (2002) 2357–2368.
3. Marques, N.; Sella, A.; Takats, J.: Chemistry of the lanthanides using pyrazolylborate ligands. Chem. Rev. **102** (2002) 2137–2159.
4. Zaleski, C. M.; Depperman, E. C.; Kampf, J. W.; Kirk, M. L.; Pecoraro, V. L.: Synthesis, structure, and magnetic properties of a large lanthanide-transition metal single-molecule magnet. Angew. Chem. Int. Ed. **43** (2004) 3912–3914.
5. Muro, I. G.; Mautner, F. A.; Insausti, M.; Lezama, L.; Arriortua, M. I.; Rojo, T.: Study of the two-dimensional $[MM'(C_3H_2O_4)_2(H_2O)_4]$ (M = Ba, Sr and M′ = Cu, Mn) systems: synthesis, structure, magnetic properties, and thermal decomposition. Inorg. Chem. **37** (1998) 3243–3251.
6. Wang, L. N.; Liu, X. R.; Wang, C. X.; Yan, L. Y.; Huang, J.: Preparation and thermal analysis of $C_{14}H_{10}N_4O_6$ and its complexes with rare earth. Synth. React. Inorg. Met.-Org. Chem. **39** (2009) 619–624.
7. Liu, F.; Zhang, Q.; Hou, G. F.; Chen, Q. F.; Chen, P.; Li, G. M.: Synthesis, structures of salen-type 3d-4f heteronuclear metal complexes. J. Inorg. Organomet. Polym. **23** (2013) 1097–1103.
8. Bruker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, Wisconsin, USA, 2009.
9. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.