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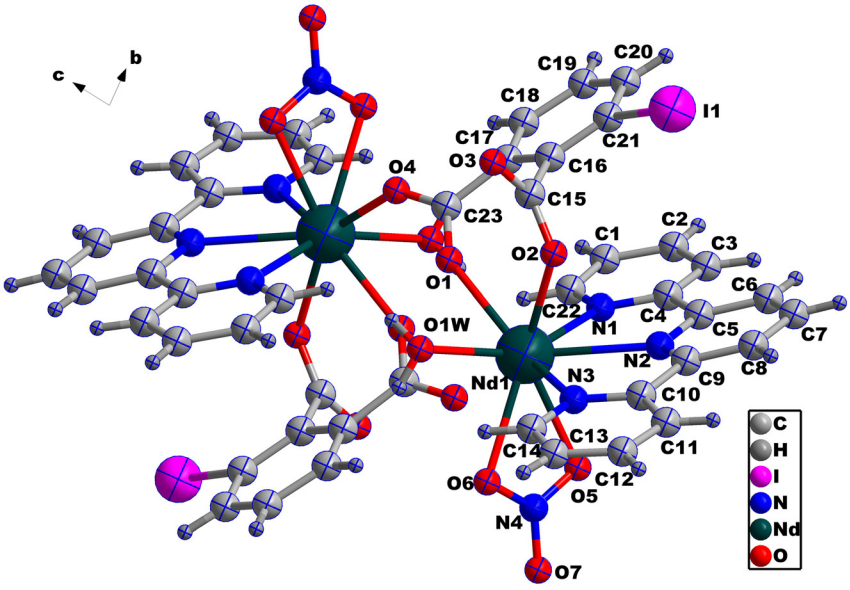
The crystal structure of bis(μ_2-biphenyl-2,2′-dicarboxylato)-diaqua-bis(nitrato)-bis(2,2′:6′,2″-terpyridine)dineodymium(III), $C_{46}H_{32}I_2N_8Nd_2O_{16}$



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Abstract

$C_{46}H_{32}I_2N_8Nd_2O_{16}$, triclinic, $P\bar{1}$ (no. 2), a = 8.6591(17) Å, b = 11.655(2) Å, c = 12.711(3) Å, α = 81.55(3)°, β = 86.62(3)°, γ = 68.50(3)°, V = 1180.5(5) Å^3, Z = 1, $R_{gt}(F)$ = 0.0298, $wR_{ref}(F^2)$ = 0.1211, T = 293(2) K.

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Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.26 × 0.18 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.57 mm^{-1}
Diffractometer, scan mode:	Bruker P4, φ and ω scans
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11663, 5383, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4,758
$N(param)_{refined}$:	335
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

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A part of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

All raw materials are analytical grade, purchased without further purification, and used directly. The titled

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1522 (6)	0.7273 (6)	0.2679 (5)	0.0385 (12)
H1	0.060136	0.723509	0.307787	0.046*
C2	0.1351 (7)	0.8124 (6)	0.1785 (5)	0.0476 (15)
H2	0.030477	0.868820	0.157430	0.057*
C3	0.2746 (8)	0.8140 (6)	0.1196 (5)	0.0469 (15)
H3	0.264559	0.868815	0.057101	0.056*
C4	0.4293 (6)	0.7321 (5)	0.1557 (4)	0.0299 (10)
C5	0.5865 (6)	0.7332 (5)	0.0990 (4)	0.0284 (10)
C6	0.5780 (8)	0.8238 (6)	0.0114 (4)	0.0430 (14)
H6	0.476427	0.880807	−0.014507	0.052*
C7	0.7252 (8)	0.8254 (6)	−0.0351 (5)	0.0472 (15)
H7	0.724171	0.885840	−0.091904	0.057*
C8	0.8733 (8)	0.7378 (6)	0.0025 (4)	0.0401 (13)
H8	0.973108	0.737889	−0.028813	0.048*
C9	0.8716 (6)	0.6491 (5)	0.0880 (4)	0.0294 (10)
C10	1.0269 (6)	0.5517 (5)	0.1296 (4)	0.0277 (10)
C11	1.1796 (6)	0.5348 (6)	0.0769 (4)	0.0347 (11)
H11	1.182709	0.583081	0.012293	0.042*
C12	1.3241 (6)	0.4477 (6)	0.1202 (4)	0.0370 (12)
H12	1.425451	0.436234	0.085806	0.044*
C13	1.3148 (6)	0.3782 (5)	0.2153 (4)	0.0345 (11)
H13	1.410887	0.319449	0.246977	0.041*
C14	1.1648 (6)	0.3951 (5)	0.2635 (4)	0.0334 (11)
H14	1.160694	0.345685	0.327299	0.040*
C15	0.7237 (5)	0.7556 (4)	0.4158 (4)	0.0217 (9)
C16	0.5406 (5)	0.8311 (4)	0.4110 (3)	0.0221 (9)
C17	0.4268 (6)	0.7864 (4)	0.4696 (3)	0.0246 (9)
C18	0.2612 (6)	0.8599 (5)	0.4703 (4)	0.0357 (11)
H18	0.186641	0.831046	0.510993	0.043*
C19	0.2049 (7)	0.9768 (5)	0.4107 (5)	0.0440 (14)
H19	0.092439	1.025317	0.410664	0.053*
C20	0.3124 (7)	1.0210 (5)	0.3526 (5)	0.0400 (13)
H20	0.273548	1.098887	0.311750	0.048*
C21	0.4814 (6)	0.9491 (5)	0.3541 (4)	0.0297 (10)
C22	0.3124 (6)	0.6464 (5)	0.2972 (4)	0.0303 (10)
H22	0.324820	0.587831	0.357412	0.036*
C23	0.4847 (6)	0.6558 (4)	0.5262 (4)	0.0247 (9)
I1	0.64666 (5)	1.02360 (3)	0.26854 (3)	0.04201 (13)
N1	0.4490 (5)	0.6485 (4)	0.2433 (3)	0.0271 (8)
N2	0.7291 (5)	0.6468 (4)	0.1349 (3)	0.0245 (8)
N3	1.0221 (5)	0.4809 (4)	0.2222 (3)	0.0286 (9)
N4	0.8356 (6)	0.2750 (5)	0.2096 (4)	0.0405 (11)
Nd1	0.73807 (3)	0.50312 (2)	0.32045 (2)	0.01917 (10)
O1	0.6093 (4)	0.5762 (3)	0.4882 (2)	0.0251 (7)
O2	0.7864 (4)	0.6866 (3)	0.3442 (3)	0.0267 (7)
O3	0.8025 (5)	0.7685 (3)	0.4886 (3)	0.0341 (8)
O4	0.4060 (4)	0.6319 (3)	0.6074 (3)	0.0293 (7)
O5	0.7275 (5)	0.3800 (4)	0.1732 (3)	0.0415 (9)
O6	0.9097 (5)	0.2703 (4)	0.2926 (4)	0.0448 (10)
O7	0.8633 (8)	0.1839 (5)	0.1639 (5)	0.0746 (18)
O1W	0.9263 (5)	0.4108(4)	0.4721(3)	0.0368 (8)
H1WA	0.961452	0.330874	0.477517	0.055*
H1WB	0.872213	0.427544	0.530396	0.055*

neodymium(III) complex was hydrothermally synthesized as follows: 0.0433 g neodymium nitrate hexahydrate (0.1 mmol), 0.0292 g (0.1 mmol) 3-iodobenzene-1,2-dicarboxylic acid, 0.0233 g 2,2′:6′,2″-terpyridine (0.1 mmol), and 0.008 g NaOH (0.2 mmol) were mixed with 5 mL H_2O, then added to a 20 mL Teflon-lined stainless steel autoclave at 393 K for 72 h. The pink block crystals were harvested with a yield 56.4 % (based on 3-iodobenzene-1,2-dicarboxylic acid).

2 Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C and O atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2U_{eq}(C) and U_{iso}(H) = 1.5U_{eq}(O)) using a riding model with C–H = 0.930 Å and O–H = 0.861 Å.

3 Comment

The crystal structures of four-components of lanthanide complexes base on lanthanide(III), benzenecarboxylic acid or its direvatives, 2,2′:6′,2″-terpyridine, and nitrate have been reported elsewhere, including mono-nuclear Ln(III),[5,6] di-nuclear Ln(III)[7–11] and 1D chain.[12] Besides, the crystal structure of 3-iodophthalic acid[13] and its mono-nuclear 1D chainlike structure[12] have been reported, the crystal structure of di-nuclear Ln(III) complex based on the four-components mentioned above has not been published anywhere. Thus, we reported one crystal structure of this type of neodymium(III) complex. The asymmetric unit consists of one neodymium(III) cation, one full-deprotonated 3-iodobenzene-1,2-dicarboxylate anion, one neutral 2,2′:6′2″-tripyridine, one nitrate, and one coordinated water molucule. The neodymium(III) is nine-coordinated with three nitrogen atoms from one 2,2′:6′2″-tripyridine, three oxygen atoms from three carboxyl groups of two 3-iodobenzene-1,2-dicarboxylate anions, two oxygen atoms from one nitrate, and one oxygen atom from one coordinated water molecule. 3-Iodobenzene-1,2-dicarboxylates were bridged by two carboxylates to generate one di-nuclear neodymium(III) complex. A three dimensional supramolecular structure is obtained by two kind of hydrogen bonds C–H···O (C1–H1···O2, C8–H8···O7, C11–H11···O5, C12–H12···O5, and C14–H14···O3) and O–H···O (O1–H1WA···O3), and π···π weak interactions.

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