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Poly[(μ_3-*n*-hexane-1,6-dicarboxylato)dysprosium(III)]

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.029
wR factor = 0.079
Data-to-parameter ratio = 18.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title dysprosium coordination polymer, $[Dy(C_9H_{16}O_8)]_n$, consists of Dy_2O_{16} dimers linked by adipate bridges to form a two-dimensional layered structure. Furthermore, intermolecular O—H···O hydrogen bonds link these layers into a three-dimensional network.

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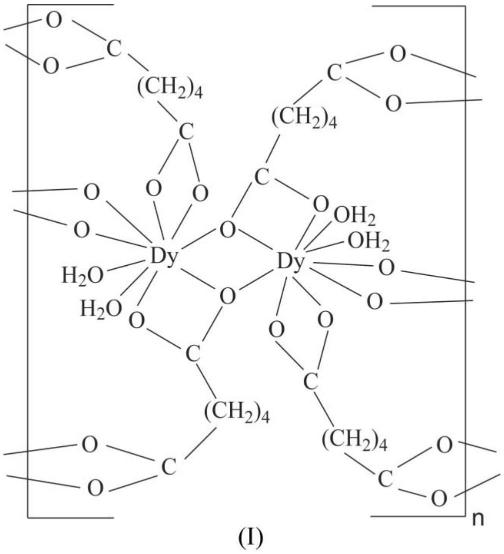
Comment

The title compound, (I), was synthesized as a means to further understand the formation of lanthanide–organic framework materials. The compound consists of DyO_9 polydedra that are edge-shared to produce dimers. These dimers are linked together through adipate anions to form a two-dimensional layer perpendicular to (010). Around each Dy metal center there are nine O atoms, two of which are bound water molecules (O7 and O8). The metal centers dimerize through O5 and its symmetry equivalent in a bridging tridentate fashion. The remaining O atoms (O1–O4 and O6) serve to link the dimers into a layered structure. One of the organic ligands is modeled as disordered over two positions (C2 and C3).



(I)

Experimental

Crystals of the title compound were synthesized using hydrothermal methods in a 23 ml Teflon-lined Parr bomb, which was then sealed. Dysprosium(III) chloride hexahydrate (0.137 g), adipic acid (0.071 g), concentrated aqueous ammonium hydroxide (0.077 g), and distilled water (1.36 g) were placed into the bomb and sealed. The bomb was then heated under autogenous pressure for 6 d at 450 K and allowed to cool at room temperature for 24 h. Upon opening the bomb, a clear colorless solution was decanted from small colorless crystals. These crystals were washed with distilled water followed by ethanol, and allowed to air-dry at room temperature. Powder X-ray diffraction was conducted on the sample. The experimental pattern was compared to that calculated from single-crystal analysis and the purity of the sample was confirmed.

Crystal data

$[Dy(C_9H_{16}O_8)]$
$M_r = 414.72$
Monoclinic, $P2_1/c$
$a = 11.651$ (2) Å
$b = 13.942$ (2) Å
$c = 8.981$ (2) Å
$\beta = 110.837$ (3)°
$V = 1363.4$ (4) Å^3
$Z = 4$

$D_x = 2.020$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1890 reflections
$\theta = 1.9$–27.5°
$\mu = 5.51$ mm^{-1}
$T = 173$ (2) K
Thick plate, colorless
0.26 × 0.20 × 0.10 mm

Data collection

Bruker SMART Platform CCD diffractometer
ω scans
Absorption correction: multi-scan (*TWINABS*; Blessing, 1995; Sheldrick, 2002)
$T_{min} = 0.277$, $T_{max} = 0.576$
3106 measured reflections

3139 independent reflections
2657 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.5°$
$h = -15 \rightarrow 14$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.080$
$S = 1.02$
3106 reflections
168 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.5P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.53$ e Å^{-3}
$\Delta\rho_{min} = -1.40$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Dy1—O8	2.380 (3)	Dy1—O1	2.502 (3)
Dy1—O7	2.380 (3)	Dy1—O6	2.503 (3)
Dy1—O5^{i}	2.400 (3)	Dy1—O3	2.510 (3)
Dy1—O4	2.423 (3)	Dy1—O5	2.567 (3)
Dy1—O2	2.471 (3)	O5—Dy1^{i}	2.400 (3)
O8—Dy1—O7	83.15 (11)	Dy1^{i}—O5—Dy1	113.22 (10)

Symmetry codes: (i) $-x, -y, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O6^{ii}	0.85	2.16	2.645 (4)	116
O7—H7B···O2^{i}	0.85	1.84	2.689 (4)	180
O8—H8C···O1^{ii}	0.85	2.22	2.740 (4)	120
O8—H8D···O3^{iii}	0.85	1.86	2.687 (4)	166

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

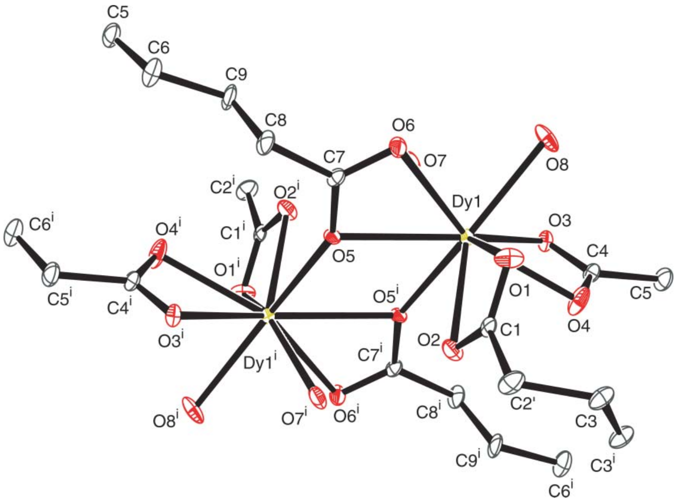


Figure 1
View of part of the title structure. Ellipsoids are shown at the 50% probability level. Apostrophe indicates the second position of a disordered atom. [Symmetry code: (i) $-x, -y, -z$.]

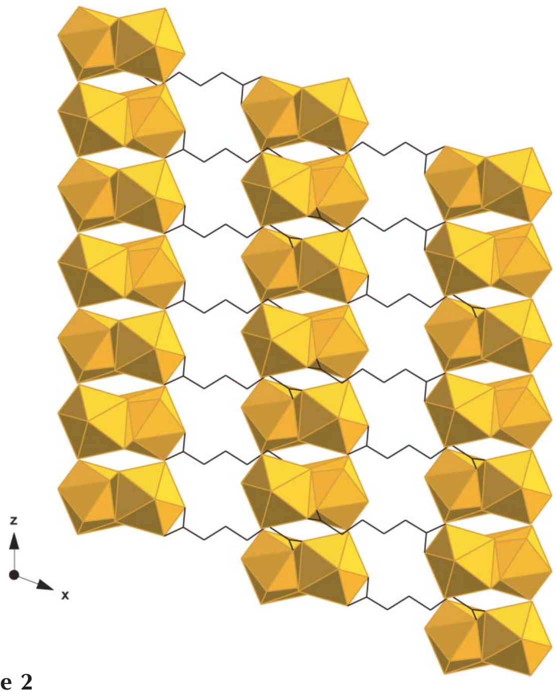


Figure 2
View along [010] of the title compound. Orange–yellow polyhedra represent DyO_9 polyhedra and black lines represent the adipate ligand.

H atoms were placed in ideal positions (C—H = 0.99 Å and O—H = 0.85 Å) and refined in a riding-model approximation with $U_{iso} = 1.2U_{eq}(C)$. The refinement stalled at $R1 = 0.0463$ and signs of twinning were noticed. The data were re-integrated and the absorption correction was reapplied. The non-merohedral twin law, [0.9736 −0.0033−0.0854 / −0.0001 0.9987 −0.0151 / 0.0504 0.0062 1.0204], a 176.5° rotation about the direct space vector [010], was determined from the program *GEMINI* (Bruker, 2000). Redundant reflections produced from *TWINABS* (Sheldrick, 2002) were removed using the *STRIP-REDUNDANT* (Brennessel & Young, 2002) program. Additionally, *SYSABSFILTER* (Brennessel & Young, 2002) was used to remove systematic absence violations. There were 14636 (3236 unique) reflections associated solely with the major (62%) twin component, 14549 (3225 unique) reflections associated solely with the minor (38%) twin component, and 1435 (683 unique) overlapping reflections. A void space of approximately 40 Å^3 per unit cell is present, which is typical for these types of materials. Residual electron density of 1.53 e Å^{-3} was found at 0.91 Å from the Dy atom, and the deepest hole, −1.40 e Å^{-3}, was also located near the Dy atom, at a distance of 0.80 Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve

m1344 Lill et al. • $[Dy(C_9H_{16}O_8)]$ Acta Cryst. (2005). E61, m1343–m1345

structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL* (Bruker, 2000); molecular graphics: *CRYSTALMAKER* (CrystalMaker, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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