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Poly[[diaqua-μ_6-succinato-di-μ_5-succinato-didysprosium(III)] monohydrate]

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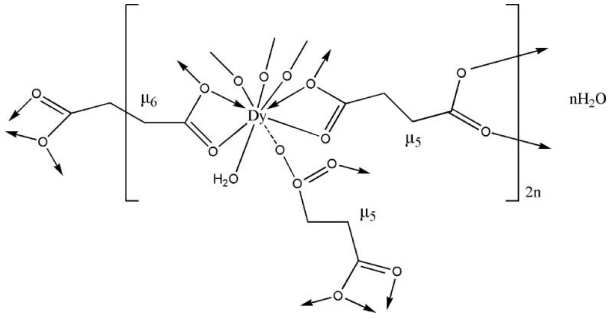
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.016; wR factor = 0.038; data-to-parameter ratio = 15.7.

The title compound, $\{[Dy_2(C_4H_4O_4)_3(H_2O)_2]\cdot H_2O\}_n$, is isostructural with other lanthanide succinates of the same formula. The Dy^{III} atom is nine-coordinated in a tricapped trigonal–prismatic environment by eight O atoms, derived from six carboxylate groups and a water molecule. One of the independent succinate anions is located about a crystallographic inversion center and the uncoordinated water molecule lies on a twofold axis. The crystal structure comprises edge-shared DyO_9 polyhedra linked by succinate bridges, forming a three-dimensional network architecture. Intra- and intermolecular O—H···O hydrogen bonds are present in the crystal structure.

Related literature

For related compounds, see: Perles *et al.* (2004); Serpaggi & Ferey (1999); He *et al.* (2007); Seguatni *et al.* (2004); Zhou *et al.* (2005); Cui *et al.* (2005); Yu *et al.* (2006); Li (2007).



Experimental

Crystal data

$[Dy_2(C_4H_4O_4)_3(H_2O)_2]\cdot H_2O$ M_r = 727.26
Monoclinic, $C2/c$
a = 19.981 (4) Å
b = 7.7616 (16) Å
c = 13.868 (3) Å
β = 121.49 (3)°
V = 1834.0 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.17 mm^{-1}
T = 293 K
0.45 × 0.20 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.154, T_{max} = 0.319
8691 measured reflections
2093 independent reflections
2016 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.038
S = 1.09
2093 reflections
133 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.73 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O3^{i}	0.85	2.07	2.878 (3)	158
O7—H7B···O2^{ii}	0.85	1.86	2.710 (3)	174
O8—H8W···O7	0.85	2.17	2.949 (4)	153

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{5}{2}, -z$; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2100).

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supporting information

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Poly[[diaqua-μ_6-succinato-di-μ_5-succinato-didysprosium(III)] monohydrate]

Wei Xu, Hai-Sheng Chang and Xia-Xia Guo

S1. Comment

There is considerable interest in the study of coordination frameworks with suitable rigid multidentate ligands. Especially those having long chain dicarboxylates present interesting behavior owing to their conformational flexibility and coordination diversity. Lanthanide ions exhibit high affinity for oxygen and diverse coordination modes. In an attempt to further understand the formation of lanthanide–organic framework materials, we present here the hydrothermal synthesis and crystal structure of a new *Ln*–succinate complex, (I).

The title compound, (I), is isostructural with the known *Ln*–succinate complexes where *Ln* = Y and La (Perles *et al.*, 2004), Pr (Serpaggi & Ferey, 1999), Nd (He *et al.*, 2007), Sm (Seguatni *et al.*, 2004), Gd (Zhou *et al.*, 2005), Tb (Cui *et al.*, 2005), Ho (Yu *et al.*, 2006), and Er (Li, 2007) analogs, but it represents the first reported succinate coordination polymer of dysprosium (III).

The asymmetric unit in (I) comprises a Dy atom, one and a half succinate anions, a coordinated water molecule and half an uncoordinated water molecule (Fig. 1). The complete second succinate dianion, containing O5 and O6, is generated from the half-ion by inversion and the uncoordinated water molecule O atom is located on a twofold axis. The Dy^{III} ion is nine-coordinated within a tricapped trigonal-prismatic geometry defined by eight O atoms, derived from six carboxylate anions, and a water molecule. The crystal structure comprises edge–sharing $DyO_8(OH_2)$ polyhedra forming chains along the *b*–axis direction by sharing one edge with each neigboring polyhedron. The Dy···Dy distance within chains is 4.046 (1) Å. These chains are in turn linked *via* succinate bridges, forming a three-dimensional framework (Fig. 2.). Intra- and intermolecular O–H···O hydrogen bonds are present in the crystal structure (Table 1).

S2. Experimental

A mixture of $Dy(NO_3)_3.7H_2O$ (0.1316 g, 0.30 mmol), succinic acid (0.0354 g, 0.30 mmol), 1,10-phenanthroline (0.0595 g, 0.30 mmol), water (10 ml) was adjusted to a pH = 5.25 by NaOH solution. The mixture was sealed in a Teflon-lined stainless steel reactor and heated at 443 K for 3 d. After the reaction system had cooled slowly to room temperature, a small quantity of colourless crystals was isolated.

S3. Refinement

H atoms bonded to C atoms were placed in their geometrically calculated positions and refined using the riding model, with C–H distances 0.97 Å and U_{iso}(H) = 1.2 U_{eq}(C). H atoms attached to O atoms were found in a difference Fourier map and then refined using the riding model, with O–H distances fixed as initially found and with O–H distances 0.85 Å and U_{iso}(H) values set at 1.2 U_{eq}(O).

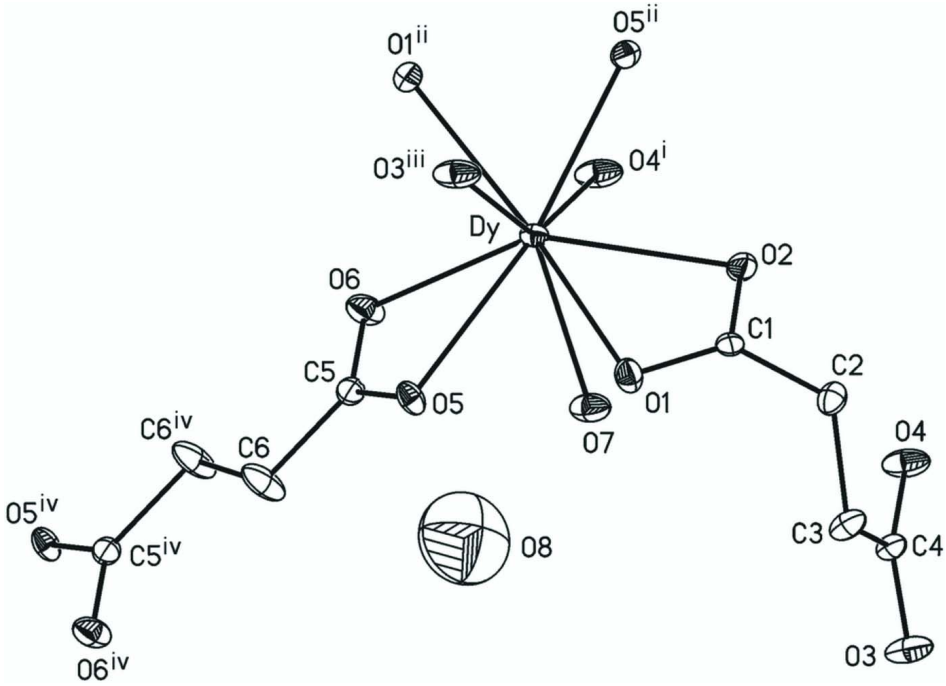

Figure 1

The coordination environment of the Dy^{III} ion in (I), showing the atom labeling and dispacement ellipsoids drawn at the 45% probability level. H atoms have been removed for clarity. [Symmetry codes: (i) -*x* + 2, -*y* + 1, -*z* + 2; (ii) -*x*, -*y*, -*z* + 1; (iii) -*x* + 1/2, *y* - 1/2, -*z* + 1/2; (iv) -*x* + 1, -*y* + 2, -*z* + 1.]

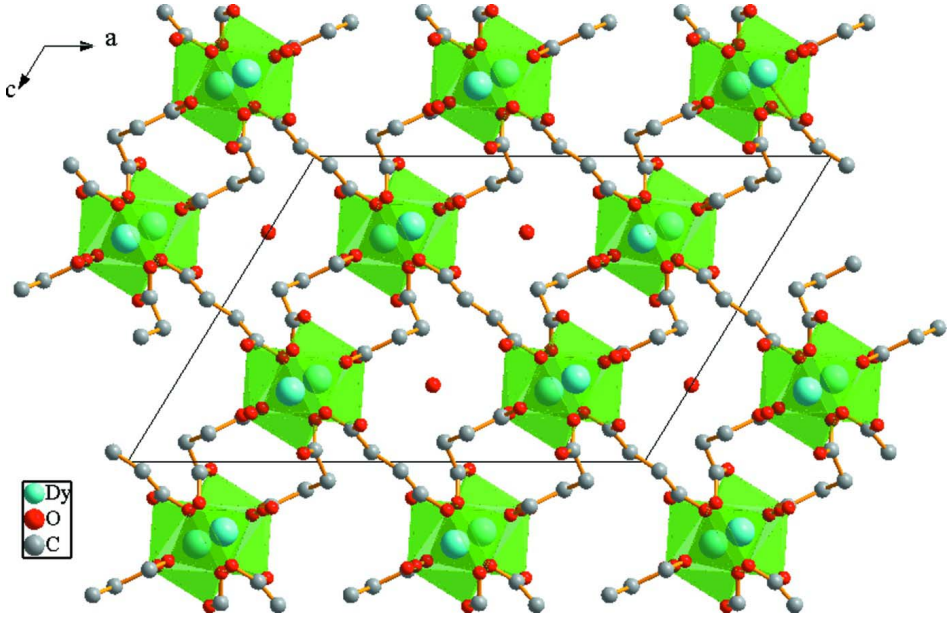

Figure 2

View of the packing in (I), drawing the tricapped trigonal-prismatic geometry of Dy^{III} in green. H atoms have been removed for clarity.

Poly[[diaqua-μ_6-succinato-di-μ_5-succinato- didysprosium(III)] monohydrate]

Crystal data

$[Dy_2(C_4H_4O_4)_3(H_2O)_2]\cdot H_2O$
$M_r = 727.26$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 19.981$ (4) Å
$b = 7.7616$ (16) Å
$c = 13.868$ (3) Å
$\beta = 121.49$ (3)°
$V = 1834.0$ (9) Å^3
$Z = 4$
$F(000) = 1368$
$D_x = 2.634$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8350 reflections
$\theta = 3.0$–$27.5°$
$\mu = 8.17$ mm^{-1}
$T = 293$ K
Prism, colorless
0.45 × 0.20 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.154$, $T_{max} = 0.319$
8691 measured reflections
2093 independent reflections
2016 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -25\rightarrow25$
$k = -10\rightarrow9$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.016$
$wR(F^2) = 0.038$
$S = 1.09$
2093 reflections
133 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0112P)^2 + 12.5672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.78$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00050 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Dy	0.268767 (7)	0.716662 (17)	0.229964 (11)	0.01104 (6)
O1	0.18695 (12)	0.9822 (3)	0.13564 (17)	0.0161 (4)
O2	0.16984 (14)	0.7651 (3)	0.02657 (18)	0.0179 (4)

O3	0.19605 (15)	1.2559 (3)	−0.1745 (2)	0.0225 (5)
O4	0.18010 (14)	0.9767 (3)	−0.1583 (2)	0.0214 (5)
C1	0.15245 (16)	0.9157 (4)	0.0371 (2)	0.0118 (5)
C2	0.09015 (17)	1.0166 (4)	−0.0632 (2)	0.0152 (6)
H2A	0.0506	1.0544	−0.0473	0.018*
H2B	0.0648	0.9413	−0.1286	0.018*
C3	0.12259 (19)	1.1741 (4)	−0.0920 (3)	0.0167 (6)
H3A	0.0791	1.2478	−0.1429	0.020*
H3B	0.1556	1.2388	−0.0231	0.020*
C4	0.16978 (17)	1.1315 (4)	−0.1461 (2)	0.0131 (5)
O5	0.32431 (11)	0.9822 (3)	0.34063 (17)	0.0142 (4)
O6	0.40702 (13)	0.7716 (3)	0.3808 (2)	0.0195 (5)
C5	0.39496 (16)	0.9239 (4)	0.3932 (2)	0.0131 (5)
C6	0.46055 (17)	1.0439 (4)	0.4690 (3)	0.0240 (7)
H6A	0.4498	1.0936	0.5237	0.029*
H6B	0.4624	1.1373	0.4239	0.029*
O7	0.33328 (14)	0.8880 (3)	0.15135 (19)	0.0200 (5)
H7A	0.3304	0.9973	0.1475	0.024*
H7B	0.3319	0.8471	0.0935	0.024*
O8	0.5000	0.9782 (13)	0.2500	0.123 (3)
H8W	0.4592	0.9198	0.2324	0.147*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Dy	0.01269 (8)	0.00799 (8)	0.01316 (8)	−0.00046 (5)	0.00725 (6)	−0.00019 (5)
O1	0.0165 (10)	0.0156 (10)	0.0126 (9)	0.0015 (8)	0.0050 (8)	−0.0025 (8)
O2	0.0279 (12)	0.0099 (10)	0.0135 (10)	0.0003 (9)	0.0091 (9)	0.0001 (8)
O3	0.0366 (14)	0.0109 (10)	0.0342 (13)	0.0015 (9)	0.0285 (12)	0.0030 (9)
O4	0.0332 (13)	0.0104 (10)	0.0346 (13)	0.0020 (9)	0.0274 (11)	0.0002 (9)
C1	0.0120 (13)	0.0136 (14)	0.0121 (12)	−0.0052 (11)	0.0079 (11)	−0.0001 (11)
C2	0.0122 (13)	0.0200 (15)	0.0132 (13)	0.0005 (11)	0.0064 (11)	0.0030 (12)
C3	0.0235 (15)	0.0137 (14)	0.0195 (14)	0.0058 (12)	0.0157 (13)	0.0048 (12)
C4	0.0159 (13)	0.0105 (13)	0.0144 (13)	0.0015 (11)	0.0088 (11)	0.0026 (11)
O5	0.0083 (9)	0.0153 (10)	0.0146 (9)	0.0004 (8)	0.0030 (8)	−0.0041 (8)
O6	0.0127 (10)	0.0118 (10)	0.0261 (12)	0.0008 (8)	0.0046 (9)	−0.0003 (9)
C5	0.0103 (13)	0.0148 (14)	0.0130 (12)	−0.0017 (11)	0.0054 (11)	0.0012 (11)
C6	0.0108 (14)	0.0159 (15)	0.0325 (18)	−0.0014 (12)	0.0024 (13)	−0.0079 (14)
O7	0.0320 (13)	0.0134 (11)	0.0247 (11)	−0.0033 (9)	0.0219 (10)	−0.0024 (9)
O8	0.090 (6)	0.136 (8)	0.139 (7)	0.000	0.058 (6)	0.000

Geometric parameters (Å, º)

Dy—O4^{i}	2.312 (2)	C2—C3	1.531 (4)
Dy—O5^{ii}	2.414 (2)	C2—H2A	0.9700
Dy—O1^{ii}	2.417 (2)	C2—H2B	0.9700
Dy—O3^{iii}	2.434 (2)	C3—C4	1.517 (4)
Dy—O5	2.461 (2)	C3—H3A	0.9700

Dy—O7	2.467 (2)	C3—H3B	0.9700
Dy—O6	2.480 (2)	O5—C5	1.286 (3)
Dy—O2	2.486 (2)	O5—Dy^{iv}	2.414 (2)
Dy—O1	2.529 (2)	O6—C5	1.236 (4)
O1—C1	1.275 (3)	C5—C6	1.500 (4)
O1—Dy^{iv}	2.417 (2)	C6—C6^{vi}	1.508 (6)
O2—C1	1.249 (4)	C6—H6A	0.9700
O3—C4	1.257 (4)	C6—H6B	0.9700
O3—Dy^{v}	2.434 (2)	O7—H7A	0.8500
O4—C4	1.246 (4)	O7—H7B	0.8501
O4—Dy^{i}	2.312 (2)	O8—H8W	0.8503
C1—C2	1.513 (4)		

O4^{i}—Dy—O5^{ii}	75.88 (8)	O6—Dy—C1	133.45 (8)
O4^{i}—Dy—O1^{ii}	77.11 (8)	O2—Dy—C1	25.28 (8)
O5^{ii}—Dy—O1^{ii}	68.86 (8)	O1—Dy—C1	25.96 (7)
O4^{i}—Dy—O3^{iii}	144.52 (8)	C5—Dy—C1	112.44 (9)
O5^{ii}—Dy—O3^{iii}	74.58 (8)	C1—O1—Dy^{iv}	155.0 (2)
O1^{ii}—Dy—O3^{iii}	74.22 (8)	C1—O1—Dy	93.77 (18)
O4^{i}—Dy—O5	130.94 (8)	Dy^{iv}—O1—Dy	109.71 (8)
O5^{ii}—Dy—O5	152.29 (2)	C1—O2—Dy	96.55 (17)
O1^{ii}—Dy—O5	106.58 (7)	C4—O3—Dy^{v}	134.7 (2)
O3^{iii}—Dy—O5	77.89 (7)	C4—O4—Dy^{i}	145.7 (2)
O4^{i}—Dy—O7	73.12 (8)	O2—C1—O1	118.4 (3)
O5^{ii}—Dy—O7	134.18 (7)	O2—C1—C2	121.5 (3)
O1^{ii}—Dy—O7	132.93 (8)	O1—C1—C2	120.0 (3)
O3^{iii}—Dy—O7	142.35 (7)	O2—C1—Dy	58.17 (15)
O5—Dy—O7	69.79 (7)	O1—C1—Dy	60.27 (15)
O4^{i}—Dy—O6	85.79 (8)	C2—C1—Dy	178.43 (19)
O5^{ii}—Dy—O6	138.96 (7)	C1—C2—C3	113.3 (2)
O1^{ii}—Dy—O6	71.41 (8)	C1—C2—H2A	108.9
O3^{iii}—Dy—O6	104.14 (9)	C3—C2—H2A	108.9
O5—Dy—O6	52.36 (7)	C1—C2—H2B	108.9
O7—Dy—O6	70.81 (8)	C3—C2—H2B	108.9
O4^{i}—Dy—O2	83.01 (8)	H2A—C2—H2B	107.7
O5^{ii}—Dy—O2	70.51 (7)	C4—C3—C2	114.3 (3)
O1^{ii}—Dy—O2	137.94 (7)	C4—C3—H3A	108.7
O3^{iii}—Dy—O2	104.93 (9)	C2—C3—H3A	108.7
O5—Dy—O2	114.41 (7)	C4—C3—H3B	108.7
O7—Dy—O2	72.92 (8)	C2—C3—H3B	108.7
O6—Dy—O2	143.72 (8)	H3A—C3—H3B	107.6
O4^{i}—Dy—O1	128.17 (8)	O4—C4—O3	124.9 (3)
O5^{ii}—Dy—O1	104.58 (7)	O4—C4—C3	117.9 (3)
O1^{ii}—Dy—O1	152.80 (2)	O3—C4—C3	117.2 (3)
O3^{iii}—Dy—O1	78.58 (8)	C5—O5—Dy^{iv}	151.66 (19)
O5—Dy—O1	66.36 (7)	C5—O5—Dy	93.61 (17)
O7—Dy—O1	71.21 (7)	Dy^{iv}—O5—Dy	112.19 (8)
O6—Dy—O1	115.49 (7)	C5—O6—Dy	93.98 (17)

O2—Dy—O1	51.24 (7)	O6—C5—O5	119.6 (3)
O4^{i}—Dy—C5	107.57 (9)	O6—C5—C6	121.9 (3)
O5^{ii}—Dy—C5	157.37 (7)	O5—C5—C6	118.5 (3)
O1^{ii}—Dy—C5	89.81 (8)	O6—C5—Dy	60.34 (15)
O3^{iii}—Dy—C5	92.88 (8)	O5—C5—Dy	59.60 (15)
O5—Dy—C5	26.79 (8)	C6—C5—Dy	174.0 (2)
O7—Dy—C5	66.22 (8)	C5—C6—C6^{vi}	112.9 (3)
O6—Dy—C5	25.67 (8)	C5—C6—H6A	109.0
O2—Dy—C5	131.74 (8)	C6^{vi}—C6—H6A	109.0
O1—Dy—C5	90.89 (8)	C5—C6—H6B	109.0
O4^{i}—Dy—C1	105.65 (8)	C6^{vi}—C6—H6B	109.0
O5^{ii}—Dy—C1	87.20 (8)	H6A—C6—H6B	107.8
O1^{ii}—Dy—C1	154.72 (7)	Dy—O7—H7A	122.1
O3^{iii}—Dy—C1	92.12 (8)	Dy—O7—H7B	116.7
O5—Dy—C1	90.70 (8)	H7A—O7—H7B	110.3
O7—Dy—C1	70.00 (8)		

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*+2, *z*+1/2; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) *x*, −*y*+2, *z*−1/2; (vi) −*x*+1, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7*A*···O3^{vii}	0.85	2.07	2.878 (3)	158
O7—H7*B*···O2^{i}	0.85	1.86	2.710 (3)	174
O8—H8*W*···O7	0.85	2.17	2.949 (4)	153

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*; (vii) −*x*+1/2, −*y*+5/2, −*z*.