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Two-dimensional dysprosium(III) triiodate(V) dihydrate, $Dy(IO_3)_3(H_2O)\cdot H_2O$

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Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Dy–O) = 0.008 Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 16.0.

During our research into novel nonlinear optical materials using 1,10-phenanthroline as an appending ligand on lanthanide iodates, crystals of an infinite layered Dy^{III} iodate compound, $Dy(IO_3)_3(H_2O)\cdot H_2O$, were obtained under hydrothermal conditions. The Dy^{III} cation has a dicapped trigonal prismatic coordination environment consisting of one water O atom and seven other O atoms from seven iodate anions. These iodate anions bridge the Dy^{III} cations into a two-dimensional structure. Through O—H$\cdots$O hydrogen bonds, all of these layers stack along [111], giving a supramolecular channel, with the solvent water molecules filling the voids.

Related literature

For related materials with non-linear optical propertie, see: Rosenzweig & Morosin (1966); Liminga *et al.* (1977); Ok & Halasyamani (2005). The method of preparation was based on HIO_3, which is different to the previous method of obtaining periodates (Douglas *et al.*, 2004; Assefa *et al.*, 2006). For noncentrosymmetric inorganic–organic framework structures synthesized from organic ligands, see: Sun *et al.* (2009). For related structrues, see: Sun *et al.* (2009); Assefa *et al.* (2006); Douglas *et al.* (2004); Ok & Halasyamani (2005); Chen *et al.* (2005).

Experimental

Crystal data

$Dy(IO_3)_3H_2O\cdot H_2O$
M_r = 723.23
Triclinic, $P\overline{1}$
a = 7.15990 (10) Å
b = 7.4292 (1) Å
c = 10.64430 (10) Å
α = 95.161 (12)°
β = 104.858 (7)°
γ = 110.081 (8)°
V = 504.00 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 16.65 mm^{-1}
T = 293 K
0.16 × 0.12 × 0.06 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.136, T_{max} = 0.435 (expected range = 0.115–0.368)
3819 measured reflections
2260 independent reflections
2067 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.06
2260 reflections
141 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 2.79 e Å^{-3}
$\Delta\rho_{min}$ = −3.20 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O10—H10$A\cdots$O3^{i}	0.80	2.29	2.873 (10)	131
O10—H10$B\cdots$O9^{i}	0.80	2.33	2.753 (11)	114
O11—H11$A\cdots$O8^{ii}	0.80	2.22	2.954 (11)	153
O11—H11$B\cdots$O7^{iii}	0.80	2.26	2.946 (11)	145

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-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) and *PLATON* (Spek, 2009; van der Sluis & Spek, 1990); 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: BR2111).

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supplementary materials

Acta Cryst. (2009). E**65**, i61-i62 [**doi:10.1107/S1600536809027068**]

Two-dimensional dysprosium(III) triiodate(V) dihydrate, $Dy(IO_3)_3(H_2O)\cdot H_2O$

W. Chai, **L. Song**, **H. Shi**, **L. Qin** and **K. Shu**

Comment

In the 1970s, metal iodates have been extensively studied by Bell Laboratories not only for their nonlinear optical (NLO) properties but also for ferroelectric, piezoelectric and pyroelectric properties (Rosenzweig & Morosin, 1966; Liminga *et al.*, 1977). In attempts to prepare noncentrosymmetric structures of lanthanide iodates, about six anhydrous structure types have been reported, in addition to numerous hydrated structures ranging from hemihydrates to pentahydrates. (Assefa *et al.*, 2006). After comparing these structure types, herein, we find that the hydrated structures favor of adopting centrosymmetric structures. Then organic ligands are come into our view because they could form noncentrosymmetric inorganic–organic framework structures with metal ion. (Sun *et al.*, 2009). Here, we firstly report a infinite layered Dy^{III} iodate dihydrate synthesized from the hydrothermal reaction of Dy_2O_3, HIO_3 and 1,10-phenanthroline.

In the title compound, the Dy^{III} cation has dicapped trigonal prismatic coordination sphere. The coordination enciroments of the rare earth Dy^{III} cation consist of eight O atoms derived from seven iodate anions and one water molecule (see Fig. 1). And these seven iodates are classed two types, one is three 3-connected iodates (of I2) through three O atoms, and the other is four iodates 2-connected (of I1 or I3) through two O atoms. Then these iodate anions bridge Dy atoms into two dimensional structure. And between the adjacent layers, there are two types of hydrogen bonds, one is O10—H10A···O3 bond, the other is O10—H10B···O9 bond. Then through these hydrogen bonds, all of these layers stacking along [111] axis to give out of a supramolecular channel. And the solvent water molecules fill in the channels, and stick on the channel with two hydrogen bonds of O11—H11A···O8 and O11—H11B···O7. (see Fig. 2) The hydrogen bonding data of lengths and angles are in the range of ordinary examples and have been examined by the *PLATON* program (Spek, 2009; van der Sluis & Spek, 1990).

Experimental

All chemicals were obtained from commercial sources and were used as received. The title compound was handily synthesized by a hydrothermal reaction from iodic acid. To a 25 ml stainless steal Teflon-lined reaction vessel, Dy_2O_3 (0.2 mmol, 75 mg), HIO_3 (0.8 mmol, 141 mg), 1,10-phenanthroline (0.4 mmol, 80 mg) and 13 ml H_2O were added and stirred thoroughly for 1 h, then heated at 393 K for 2 d. After cooling down to room temperature, some colorless crystalline product (I) was obtained.

Refinement

The structure was solved using direct methods and refined by full-matrix least-squares techniques. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. All H atoms were added at calculated positions and refined using a riding model.(Sheldrick, 2008). The maximum (2.79) and minumum (-3.20) in the difference electron density were found at 0.0198 0.3244 0.7024 [1.01 Å from DY1] and 0.2071 0.4512 0.7963 [0.60 Å from DY1], respectively.

The O6 has ADP max/min ratio 6.70. This result may be due to the packing of supramolecule.

Figures

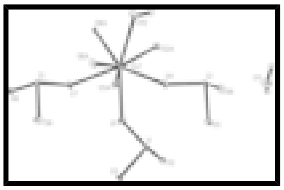

Fig. 1. Structure and labeling of the title compound, with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radii.

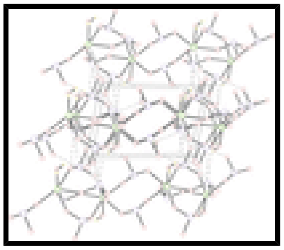

Fig. 2. The packing diagram viewed along the a-direction, Dy: green diagonal; I: purple inner dot; O: red; and H: small blue circles. And hydrogen bonds are denoted as dash lines.

dysprosium(III) triiodate(V) dihydrate

Crystal data

$Dy(IO_3)_3H_2O\cdot H_2O$

$M_r = 723.23$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

$a = 7.15990$ (10) Å

$b = 7.4292$ (1) Å

$c = 10.64430$ (10) Å

$\alpha = 95.161$ (12)°

$\beta = 104.858$ (7)°

$\gamma = 110.081$ (8)°

$V = 504.00$ (5) Å^3

$Z = 2$

$F_{000} = 634$

$D_x = 4.766$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å

Cell parameters from 1561 reflections

$\theta = 2.0$–27.5°

$\mu = 16.65$ mm^{-1}

$T = 293$ K

Block, colourless

0.16 × 0.12 × 0.06 mm

Data collection

Rigaku R-AXIS RAPID diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 14.6306 pixels mm^{-1}

$T = 293$ K

CCD profile fitting scans

Absorption correction: multi-scan (ABSCOR; Higashi, 1995)

$T_{min} = 0.136$, $T_{max} = 0.435$

3819 measured reflections

2260 independent reflections

2067 reflections with $I > 2\sigma(I)$

$R_{int} = 0.027$

$\theta_{max} = 27.5$°

$\theta_{min} = 3.2$°

$h = -9 \rightarrow 9$

$k = -7 \rightarrow 9$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.109$	$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 5.3292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2260 reflections	$\Delta\rho_{max} = 2.79$ e Å^{-3}
141 parameters	$\Delta\rho_{min} = -3.20$ e Å^{-3}
2 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0126 (8)

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}
Dy1	0.11491 (7)	−0.58338 (7)	−0.21096 (4)	0.01105 (18)
I1	0.30890 (8)	−0.14149 (8)	0.07303 (5)	0.00659 (18)
I2	0.28110 (8)	−0.63445 (8)	0.16834 (5)	0.00652 (18)
I3	0.27848 (9)	−0.26870 (9)	−0.45631 (6)	0.00862 (19)
O1	0.1496 (11)	−0.2711 (12)	−0.0929 (7)	0.0178 (15)
O2	0.0966 (11)	−0.1450 (11)	0.1365 (7)	0.0144 (14)
O3	0.3778 (11)	0.1033 (10)	0.0380 (7)	0.0134 (14)
O4	0.2940 (11)	−0.5350 (11)	0.0209 (7)	0.0155 (15)
O5	0.2291 (10)	−0.4405 (11)	0.2502 (7)	0.0131 (14)
O6	0.5556 (10)	−0.5510 (11)	0.2555 (7)	0.0121 (14)
O7	0.0908 (11)	−0.3710 (11)	−0.3699 (7)	0.0130 (14)
O8	0.1065 (11)	−0.1986 (11)	−0.5813 (7)	0.0125 (14)
O9	0.4335 (11)	−0.0352 (12)	−0.3515 (8)	0.0187 (16)
O10	0.2235 (12)	−0.8611 (12)	−0.2319 (8)	0.0185 (16)
H10A	0.198 (15)	−0.928 (9)	−0.179 (7)	0.028*

H10B	0.346 (4)	−0.8277 (13)	−0.221 (10)	0.028*
O11	0.2419 (13)	−0.7837 (13)	0.3943 (9)	0.0257 (18)
H11A	0.225 (3)	−0.897 (15)	0.3811 (19)	0.039*
H11B	0.144 (13)	−0.7725 (18)	0.412 (2)	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Dy1	0.0094 (3)	0.0136 (3)	0.0131 (3)	0.00658 (19)	0.00455 (18)	0.00444 (19)
I1	0.0048 (3)	0.0061 (3)	0.0105 (3)	0.0031 (2)	0.0028 (2)	0.0041 (2)
I2	0.0040 (3)	0.0067 (3)	0.0111 (3)	0.0040 (2)	0.0027 (2)	0.0039 (2)
I3	0.0076 (3)	0.0109 (3)	0.0101 (3)	0.0063 (2)	0.0032 (2)	0.0026 (2)
O1	0.014 (3)	0.023 (4)	0.012 (3)	0.010 (3)	−0.004 (3)	−0.002 (3)
O2	0.011 (3)	0.014 (4)	0.022 (4)	0.006 (3)	0.009 (3)	0.007 (3)
O3	0.020 (3)	0.006 (3)	0.020 (4)	0.007 (3)	0.011 (3)	0.006 (3)
O4	0.019 (4)	0.013 (4)	0.014 (3)	0.006 (3)	0.005 (3)	0.006 (3)
O5	0.006 (3)	0.014 (4)	0.021 (4)	0.005 (3)	0.007 (3)	0.002 (3)
O6	0.001 (3)	0.016 (4)	0.018 (3)	0.002 (3)	0.003 (3)	0.007 (3)
O7	0.014 (3)	0.020 (4)	0.013 (3)	0.012 (3)	0.007 (3)	0.013 (3)
O8	0.014 (3)	0.013 (4)	0.013 (3)	0.005 (3)	0.007 (3)	0.006 (3)
O9	0.011 (3)	0.018 (4)	0.022 (4)	0.005 (3)	0.000 (3)	−0.002 (3)
O10	0.022 (4)	0.023 (4)	0.025 (4)	0.019 (3)	0.013 (3)	0.011 (3)
O11	0.022 (4)	0.022 (4)	0.034 (5)	0.007 (4)	0.012 (4)	0.005 (4)

Geometric parameters (Å, °)

Dy1—O4	2.401 (7)	I2—O6	1.798 (6)
Dy1—O2^{i}	2.408 (7)	I2—O4	1.804 (7)
Dy1—O8^{ii}	2.412 (7)	I2—O5	1.812 (7)
Dy1—O6^{iii}	2.415 (6)	I3—O9	1.783 (8)
Dy1—O7	2.429 (6)	I3—O8	1.812 (7)
Dy1—O1	2.438 (8)	I3—O7	1.813 (7)
Dy1—O10	2.453 (7)	O2—Dy1^{i}	2.408 (7)
Dy1—O5^{i}	2.461 (6)	O5—Dy1^{i}	2.461 (6)
I1—O1	1.804 (7)	O6—Dy1^{iii}	2.415 (6)
I1—O2	1.809 (7)	O8—Dy1^{ii}	2.412 (7)
I1—O3	1.814 (7)		
O4—Dy1—O2^{i}	75.1 (2)	O7—Dy1—O10	126.1 (2)
O4—Dy1—O8^{ii}	149.7 (2)	O1—Dy1—O10	151.9 (2)
O2^{i}—Dy1—O8^{ii}	78.5 (2)	O4—Dy1—O5^{i}	112.2 (2)
O4—Dy1—O6^{iii}	90.6 (2)	O2^{i}—Dy1—O5^{i}	73.6 (2)
O2^{i}—Dy1—O6^{iii}	142.8 (2)	O8^{ii}—Dy1—O5^{i}	73.6 (2)
O8^{ii}—Dy1—O6^{iii}	101.7 (2)	O6^{iii}—Dy1—O5^{i}	142.8 (2)
O4—Dy1—O7	135.1 (3)	O7—Dy1—O5^{i}	72.9 (2)
O2^{i}—Dy1—O7	141.9 (2)	O1—Dy1—O5^{i}	69.4 (2)

O8^{ii}—Dy1—O7	75.1 (2)	O10—Dy1—O5^{i}	132.9 (3)
O6^{iii}—Dy1—O7	70.3 (2)	O1—I1—O2	96.8 (3)
O4—Dy1—O1	69.2 (2)	O1—I1—O3	97.2 (3)
O2^{i}—Dy1—O1	111.8 (3)	O2—I1—O3	97.7 (3)
O8^{ii}—Dy1—O1	136.0 (2)	O6—I2—O4	99.6 (3)
O6^{iii}—Dy1—O1	93.9 (3)	O6—I2—O5	97.8 (3)
O7—Dy1—O1	72.0 (2)	O4—I2—O5	95.5 (3)
O4—Dy1—O10	84.5 (3)	O9—I3—O8	99.4 (3)
O2^{i}—Dy1—O10	68.8 (3)	O9—I3—O7	101.4 (3)
O8^{ii}—Dy1—O10	72.1 (2)	O8—I3—O7	96.1 (3)
O6^{iii}—Dy1—O10	75.9 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O10—H10A···O3^{iv}	0.80	2.29	2.873 (10)	131
O10—H10B···O9^{iv}	0.80	2.33	2.753 (11)	114
O11—H11A···O8^{v}	0.80	2.22	2.954 (11)	153
O11—H11B···O7^{i}	0.80	2.26	2.946 (11)	145

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

Fig. 1

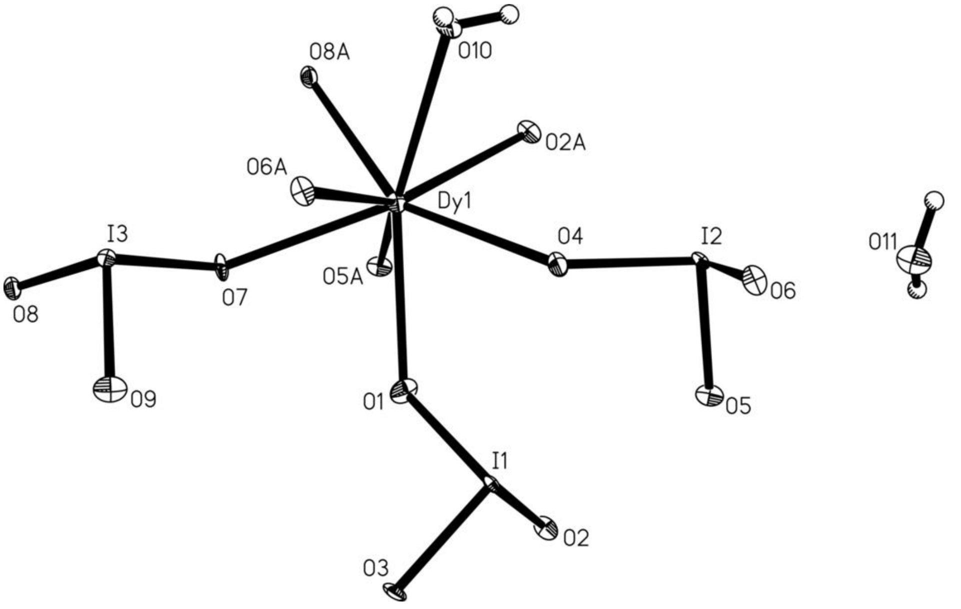

Fig. 2