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Dy_3RuO_7 with partial structural disorder

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Key indicators: single-crystal X-ray study; T = 295 K; mean σ(Ru–O) = 0.008 Å; disorder in main residue; R factor = 0.025; wR factor = 0.022; data-to-parameter ratio = 28.8.

The flux-grown single crystals of tridysprosium ruthenium heptaoxide, Dy_3RuO_7, have a noncentrosymmetric orthorhombic structure composed of infinite single chains of corner-linked RuO_6 octahedra embedded in a Dy_3O matrix. Partial disorder of Dy atoms was faintly observed. Two Dy atom sites out of the six crystallographically independent Dy sites split into two positions, which are separated by approximately 0.3–0.5 Å and have different coordination environments. The investigated crystal was an inversion twin.

Related literature

For related literature, see: Bontchev *et al.* (2000); Gemmill *et al.* (2004, 2005); Harada & Hinatsu (2001); Ishizawa *et al.* (2006, 2007).

Experimental

Crystal data

Dy_3RuO_7
M_r = 700.57
Orthorhombic, $P2_1nb$
a = 10.516 (2) Å
b = 14.560 (3) Å
c = 7.333 (2) Å
V = 1122.8 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 42.09 mm^{-1}
T = 295 K
0.06 × 0.04 × 0.03 mm

Data collection

Bruker SMART APEXII diffractometer
Absorption correction: numerical (*APEX2-W2K/NT*; Bruker, 2006)
T_{min} = 0.272, T_{max} = 0.530
158509 measured reflections
17319 independent reflections
3980 reflections with $F > 3\sigma(F)$
R_{int} = 0.098

Refinement

$R[F > 3\sigma(F)]$ = 0.025
$wR(F)$ = 0.022
S = 1.43
3980 reflections
138 parameters
$\Delta\rho_{max}$ = 3.91 e Å^{-3}
$\Delta\rho_{min}$ = −3.33 e Å^{-3}
Absolute structure: Flack (1983), 8131 Friedel pairs
Flack parameter: 0.51 (3)

Data collection: *APEX2-W2K/NT* (Bruker, 2006); cell refinement: *APEX2-W2K/NT*; data reduction: *DIFDAT*, *SORTRF* and *ADDREF* in *Xtal3.7* (Hall *et al.*, 2003); program(s) used to solve structure: *Xtal3.7*; program(s) used to refine structure: *CRYLSQ* in *Xtal3.7*; molecular graphics: *ATOMS* (Dowty, 2005); software used to prepare material for publication: *BONDLA* and *CIFIO* in *Xtal3.7*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2012).

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supplementary materials

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Comment

The room temperature structure of the title compound, (I), has been confirmed as the $P2_1nb$ modification of Ln_3MO_7 family composed of trivalent lanthanide (Ln) and pentavalent transition metal (*M*=Ru, Os) oxides (Figs. 1 and 2). The family is so far known to have two orthorhombic modifications *Cmcm* and $P2_1nb$ modifications related by the phase transition (Bontchev *et al.*, 2000, Harada & Hinatsu, 2003, Gemmill *et al.*, 2004, 2005).

A positional disorder at two Ln atom sites, *i.e.*, Ln5 and Ln6, was first suspected for Gd_3RuO_7 and confirmed in Tb_3RuO_7 (Ishizawa *et al.*, 2006, 2007). A similar but faint disorder has also been detected in Dy_3RuO_7 in the present study using single-crystal diffraction data collected with relatively high resolution level (d>0.42 Å). The split atom sites at Dy5b and Dy6b are populated 3.0 (7) and 2.2 (8) % by the Dy atoms. The coordination numbers of Dy1, 2, 3, 4, 5a and 6a are 7 in the range 2.17–2.62 Å, whereas those of Dy5b and Dy6b are 5 in the range 2.10–2.53 Å. The mean Ru···Ru interatomic distance and Ru—O—Ru angle along the $[RuO_5]^{\infty}$ octahedral chain are 3.667 (4) Å and 141.4 (5)°.

Refinement

The refinement was carried out based on the starting parameters imported from the $P2_1nb$ modification of Tb_3RuO_7 (Ishizawa *et al.*, 2007), employing anisotropic atomic displacement parameters for metal atoms and isotropic ones for O atoms. The non-split atom model indicated Δρ peaks of 10 e $Å^{-3}$ near Dy5 and Dy6 atom sites in a similar manner to Tb_3RuO_7. The peak heights are smaller than 33 e $Å^{-3}$ observed in Tb_3RuO_7 under approximately the same resolution level of d>0.42 Å. The split pairs of Dy5a and Dy5b, and Dy6a and Dy6b were constrained to have the same anisotropic atomic displacement parameters in the further refinements. The Flack parameter of ~0.5 (Flack, 1983) suggested an existence of inversion related twins as is common for the $P2_1nb$ modifications of Ln_3RuO_7 (Ln=Gd, Tb).

Figures

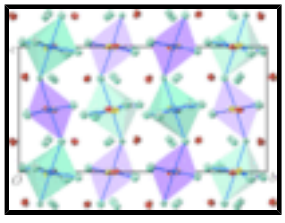

Fig. 1. The structure of Dy_3RuO_7 projected along *a*, containing $Ru1O_6$ (blue) and $Ru2O_6$ (purple) octahedral chains in the Tb (red) and O (light blue) matrix. The less populated Dy5b and Dy6b are drawn in yellow. The ADP ellipsoids are drawn at the 97% probability level.

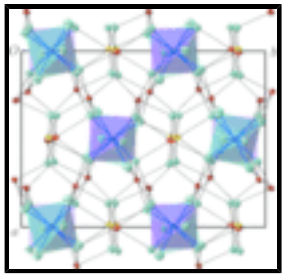

Fig. 2. The structure of Dy_3RuO_7 projected along *c* with the same colour scheme and probability level of the ADP ellipsoid as in Fig. 1. The Dy—O bonds except for Dy5b and Dy6b are given in black thin lines.

tridysprosium ruthenium heptaoxide

Crystal data

$Dy_3Ru_1O_7$	F_{000} = 2384
M_r = 700.57	D_x = 8.289 Mg m^{-3}
Orthorhombic, $P2_1nb$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: p -2bc 21	Cell parameters from 12 reflections
a = 10.516 (2) Å	θ = 23–29º
b = 14.560 (3) Å	μ = 42.09 mm^{-1}
c = 7.333 (2) Å	*T* = 295 K
V = 1122.8 (4) Å^3	Prism, black
Z = 8	0.06 × 0.04 × 0.03 mm

Data collection

Bruker SMART APEX II diffractometer	7765 reflections with $F > 3\sigma(F)$
Detector resolution: 67 pixels mm^{-1}	R_{int} = 0.098
T = 293 K	θ_{max} = 61.7º
ω and φ scans	θ_{min} = 2.8º
Absorption correction: numerical (SMART APEXII XPREP; Bruker, 2006)	*h* = −25→25
T_{min} = 0.272, T_{max} = 0.530	*k* = −35→35
158509 measured reflections	*l* = −16→16
17319 independent reflections	

Refinement

Refinement on *F*	$(\Delta/\sigma)_{max}$ = <0.001
Least-squares matrix: full	$\Delta\rho_{max}$ = 3.91 e Å^{-3}
$R[F^2 > 2\sigma(F^2)]$ = 0.025	$\Delta\rho_{min}$ = −3.33 e Å^{-3}
$wR(F^2)$ = 0.022	Extinction correction: Becker & Coppens (1974). Acta Cryst. A30,148–153
S = 1.43	Extinction coefficient: 196 (5)
3980 reflections	Absolute structure: Flack (1983), 8131 Friedel pairs
138 parameters	Flack parameter: 0.51 (3)

Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(F_o)]$

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Dy1	−0.2206 (3)	0.02169 (10)	−0.23588 (8)	0.0052 (2)	
Dy2	−0.2325 (3)	0.22160 (12)	0.24493 (7)	0.0049 (4)	
Dy3	−0.2815 (3)	0.27324 (10)	−0.25774 (7)	0.0048 (2)	
Dy4	−0.2691 (3)	−0.02693 (12)	0.25048 (5)	0.0047 (4)	
Dy5a	0.0061 (3)	0.38826 (8)	0.00271 (13)	0.00570 (17)	.970 (7)
Dy5b	−0.010 (3)	0.363 (3)	−0.002 (3)	0.00570 (17)	.030 (7)
Dy6a	−0.0095 (3)	0.36200 (8)	−0.49762 (11)	0.00519 (17)	.978 (8)
Dy6b	−0.010 (4)	0.387 (4)	−0.507 (4)	0.00519 (17)	.022 (8)
Ru1	0.00000	0.1261 (2)	−0.0007 (4)	0.00357 (16)	
Ru2	−0.00179 (8)	0.1246 (2)	−0.5036 (3)	0.00306 (17)	
O1	0.0125 (9)	0.0843 (6)	−0.2517 (7)	0.0047 (13)*	
O2	−0.0051 (11)	0.1712 (7)	0.2485 (7)	0.0067 (14)*	
O3	0.1231 (8)	0.2211 (7)	−0.4660 (9)	0.0052 (11)*	
O4	−0.3634 (11)	0.1056 (7)	0.2322 (12)	0.0061 (12)*	
O5	0.1269 (8)	0.0327 (7)	−0.5443 (10)	0.0065 (10)*	
O6	−0.1342 (11)	0.3808 (8)	−0.2338 (12)	0.0070 (14)*	
O7	−0.1339 (8)	0.2192 (7)	−0.4651 (10)	0.0067 (11)*	
O8	−0.1343 (10)	−0.1446 (7)	0.2649 (11)	0.0054 (12)*	
O9	−0.1299 (9)	0.0309 (7)	−0.5342 (10)	0.0072 (12)*	
O10	−0.3736 (11)	0.1313 (8)	−0.2432 (10)	0.0067 (14)*	
O11	−0.1009 (9)	0.0138 (6)	0.0410 (10)	0.0073 (11)*	
O12	0.1044 (8)	0.2378 (6)	−0.0407 (10)	0.0071 (11)*	
O13	0.1604 (8)	0.0652 (5)	0.0454 (10)	0.0094 (10)*	
O14	−0.1516 (9)	0.1925 (6)	−0.0490 (11)	0.0112 (12)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Dy1	0.0045 (3)	0.0057 (3)	0.00540 (14)	0.0006 (2)	0.00020 (19)	0.00136 (17)
Dy2	0.0045 (5)	0.0044 (3)	0.0058 (3)	−0.0009 (3)	−0.00010 (13)	−0.00035 (12)
Dy3	0.0044 (3)	0.0048 (3)	0.00531 (18)	−0.0007 (2)	0.00082 (16)	−0.00004 (13)
Dy4	0.0050 (5)	0.0044 (4)	0.0046 (3)	0.0006 (3)	−0.00046 (9)	−0.00034 (11)
Dy5a	0.00470 (19)	0.0084 (2)	0.00403 (11)	0.00042 (17)	−0.00019 (16)	0.0007 (2)
Dy5b	0.00470 (19)	0.0084 (2)	0.00403 (11)	0.00042 (17)	−0.00019 (16)	0.0007 (2)
Dy6a	0.00399 (19)	0.0073 (2)	0.00427 (11)	0.00025 (17)	−0.00004 (17)	−0.0002 (2)
Dy6b	0.00399 (19)	0.0073 (2)	0.00427 (11)	0.00025 (17)	−0.00004 (17)	−0.0002 (2)
Ru1	0.0029 (2)	0.00453 (16)	0.00325 (12)	−0.0001 (2)	0.0000 (2)	−0.00049 (16)
Ru2	0.0033 (2)	0.00344 (12)	0.00242 (17)	−0.0003 (2)	−0.0003 (2)	−0.00005 (16)

Geometric parameters (Å, °)

Dy1^{i}—O6^{ii}	2.25 (1)	Dy5b—O8^{iii}	2.18 (3)

Dy1^{i}—O13^{iii}	2.263 (8)	Dy5b—O12	2.20 (4)
Dy1^{i}—O10^{i}	2.266 (12)	Dy5b—O10^{i}	2.38 (3)
Dy1^{i}—O9^{i}	2.391 (8)	Dy5b—O4^{viii}	2.53 (3)
Dy1^{i}—O11^{i}	2.392 (8)	Dy5b—O9^{iv}	2.77 (4)
Dy1^{i}—O5^{iv}	2.408 (8)	Dy5b—O5^{iv}	2.88 (4)
Dy1^{i}—O1^{i}	2.617 (10)	Dy5b—O14	2.91 (4)
Dy2^{v}—O4^{v}	2.181 (11)	Dy6a^{ix}—Dy6b^{ix}	.37 (5)
Dy2^{v}—O8^{i}	2.206 (10)	Dy6a^{ix}—O10^{i}	2.301 (9)
Dy2^{v}—O3^{iii}	2.323 (8)	Dy6a^{ix}—O4^{i}	2.329 (10)
Dy2^{v}—O14^{v}	2.356 (8)	Dy6a^{ix}—O6^{ix}	2.353 (10)
Dy2^{v}—O7^{ii}	2.366 (8)	Dy6a^{ix}—O8^{iii}	2.361 (9)
Dy2^{v}—O12^{iii}	2.401 (8)	Dy6a^{ix}—O11^{iii}	2.431 (9)
Dy2^{v}—O2^{v}	2.501 (12)	Dy6a^{ix}—O7^{ix}	2.468 (10)
Dy3^{ii}—O6^{ii}	2.210 (12)	Dy6a^{ix}—O3^{ix}	2.492 (10)
Dy3^{ii}—O10^{ii}	2.285 (12)	Dy6b^{ix}—O11^{iii}	2.10 (5)
Dy3^{ii}—O7^{ii}	2.311 (9)	Dy6b^{ix}—O10^{i}	2.26 (4)
Dy3^{ii}—O3^{iv}	2.365 (7)	Dy6b^{ix}—O8^{iii}	2.34 (4)
Dy3^{ii}—O14^{ii}	2.365 (9)	Dy6b^{ix}—O4^{i}	2.34 (4)
Dy3^{ii}—O12^{iii}	2.403 (8)	Dy6b^{ix}—O6^{ix}	2.39 (4)
Dy3^{ii}—O2^{iii}	2.486 (11)	Dy6b^{ix}—O7^{ix}	2.78 (5)
Dy4^{vi}—O4^{vi}	2.174 (11)	Dy6b^{ix}—O3^{ix}	2.81 (5)
Dy4^{vi}—O8^{vi}	2.226 (11)	Ru1—O14	1.897 (9)
Dy4^{vi}—O9^{vii}	2.312 (9)	Ru1—O13	1.935 (8)
Dy4^{vi}—O13^{i}	2.360 (7)	Ru1—O1	1.943 (6)
Dy4^{vi}—O11^{vi}	2.417 (9)	Ru1—O2	1.943 (7)
Dy4^{vi}—O5^{i}	2.418 (8)	Ru1—O11	1.973 (9)
Dy4^{vi}—O1^{i}	2.444 (10)	Ru1—O12	1.984 (9)
Dy5a—Dy5b	.41 (4)	Ru1—Ru2^{ix}	3.646 (4)
Dy5a—O10^{i}	2.270 (9)	Ru1—Ru2	3.688 (4)
Dy5a—O6	2.279 (10)	Ru2^{ix}—O5^{ix}	1.926 (9)
Dy5a—O8^{iii}	2.305 (9)	Ru2^{ix}—O9^{ix}	1.930 (10)
Dy5a—O4^{viii}	2.414 (10)	Ru2^{ix}—O2	1.940 (7)
Dy5a—O12	2.442 (9)	Ru2^{ix}—O3^{ix}	1.943 (10)
Dy5a—O5^{iv}	2.476 (10)	Ru2^{ix}—O1^{ix}	1.944 (6)
Dy5a—O9^{iv}	2.532 (10)	Ru2^{ix}—O7^{ix}	1.976 (9)
Dy5b—O6	2.16 (3)		
?···?	?		
Ru1^{i}—O1^{i}—Ru2^{i}	143.1 (5)	Ru2^{ii}—O2^{v}—Ru1^{v}	139.7 (6)
?—?—?—?	?		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
?—?···?	?	?	?	?

Fig. 1

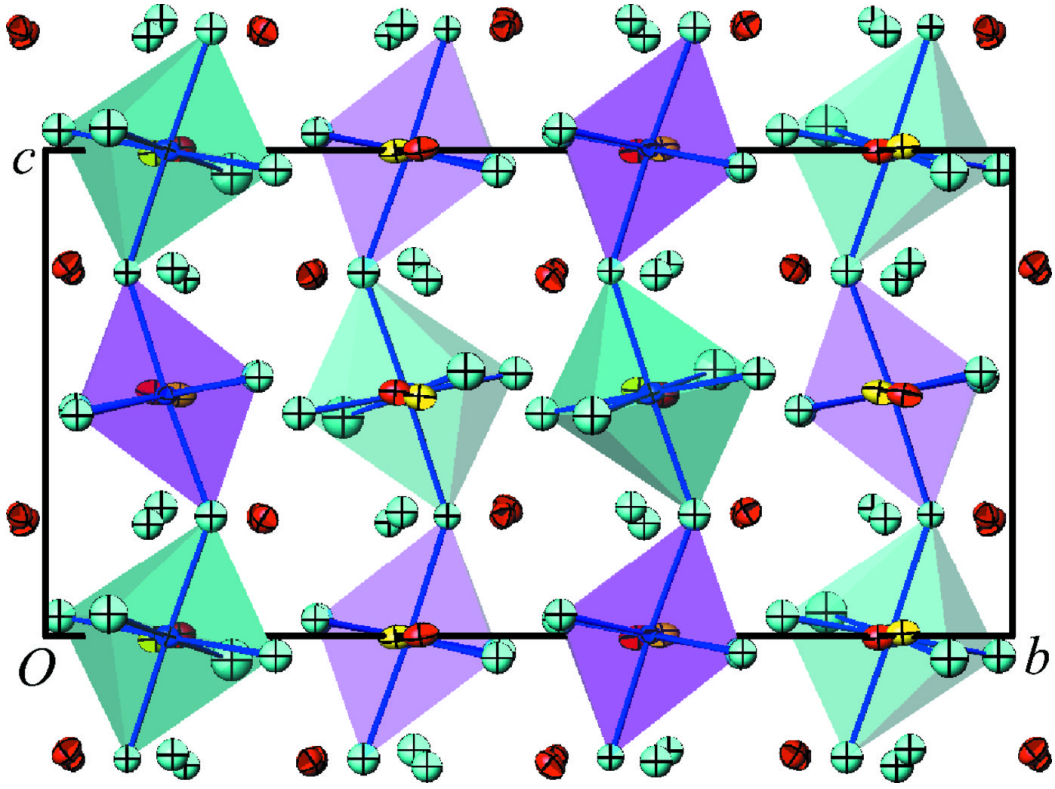

Fig. 2