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Dysprosium Tantalum Oxide, $DyTa_7O_{19}$

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Abstract

Crystals of $DyTa_7O_{19}$ were obtained by a chemical transport reaction using NH_4Cl as the transport agent. The structure was refined in space group $P\bar{6}c2$ and is built up from double layers of Ta2 polyhedra, which may be described as edge-shared pentagonal bipyramids, alternating with layers of $[Ta_3Dy_3O_{30}]$ rings.

Comment

Rare earth niobates and tantalates have been studied extensively because of their potentially interesting physical properties. Members of the systems $LnTa_7O_{19}$ (Ln = rare earth) have been reported (Bodiot, 1968; Gatehouse, 1979; Putilin, Krylov, Men'shenina & Evdokimov, 1985; Langenbach-Kuttert, Strurm & Gruehn, 1986; Pushcharovskü, Yamnova, Leonyuk & Bogdanova, 1987; Schaffrath & Gruehn, 1990). Johnson & Gatehouse (1980) redetermined the space group of $CeTa_7O_{19}$ by convergent-beam diffraction to be $P\bar{6}c2$ and not $P6_3/mcm$ as previously reported from X-ray diffraction. We report here the determination of the single-crystal structure of $DyTa_7O_{19}$, which was finally refined in space group $P\bar{6}c2$.

The polyhedron of Ta2 can be described as a distorted pentagonal bipyramid with a long Ta—O distance of 2.45 (1) Å. Fig. 1 shows a layer of edge-shared pentagonal Ta_2O_7 bipyramids along [001]. The layers of pentagonal bipyramids are corner-shared with another layer through the O1 atoms in the c direction to form double layers (see Fig. 3). The average Ta—O distance in the double layers is 2.06 Å.

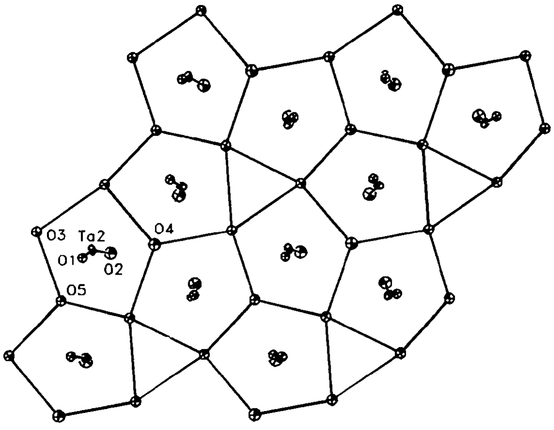

Fig. 1. A layer of Ta2 pentagonal bipyramids projected along [001] (bonds Ta2—O3, Ta2—O4 and Ta2—O5 omitted).

The Ta1 atoms are octahedrally coordinated by six O2 atoms. The Dy polyhedron is a distorted bicapped trigonal antiprism. The $Ta1O_6$ octahedra are edge-shared with DyO_8 bicapped trigonal antiprisms through O2 atoms to form six-membered $[Ta_3Dy_3O_{30}]$ rings. These six-membered rings intersect each other in the ab plane to form layers of $[Ta_3Dy_3O_{30}]$ (Fig. 2).

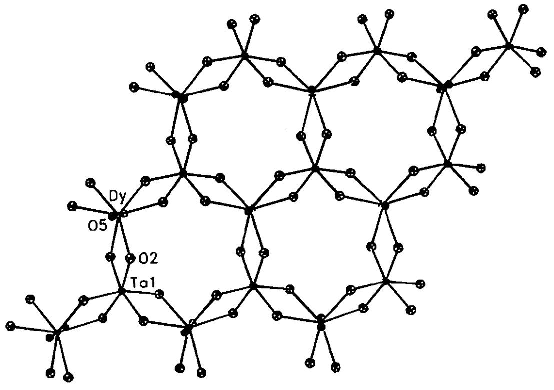

Fig. 2. A layer of six-membered $[Ta_3Dy_3O_{19}]$ rings projected along [001].

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Fig. 3 shows the crystal structure of $DyTa_7O_{19}$ along [100]. The structure is built up from the double layers of Ta2 octahedra alternating with the layers of $[Ta_3Dy_3O_{30}]$ through sharing the O2 and O5 atoms in the c direction.

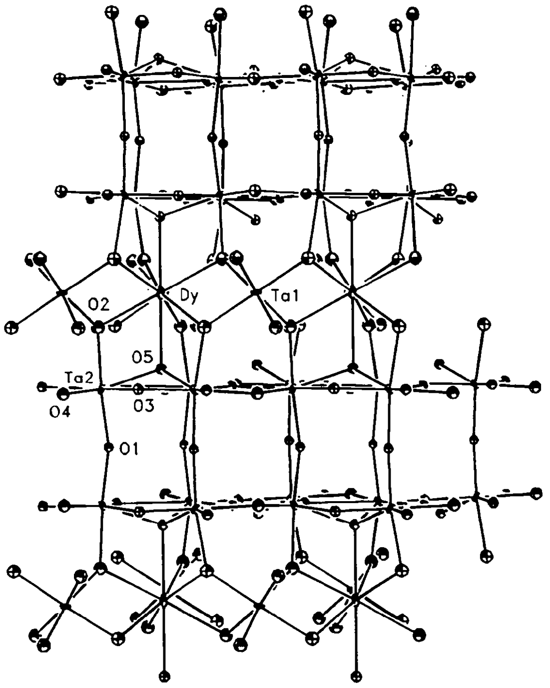

Fig. 3. Crystal structure of $DyTa_7O_{19}$ projected along [100].

Experimental

The appropriate quantities of powdered DyOCl, Ta_2O_5 and Ta (2:1:1 mol ratio) were mixed, pressed into a pellet and heated in a sealed and evacuated quartz tube at 1223 K for 9 d. The product mixture and *ca* 30 mg NH_4Cl were sealed in an evacuated silica tube. The tube was progressively heated to 1223 K for 7 d, then cooled to 1073 K at a rate of 6.25 K h^{-1} and finally cooled to room temperature. The yellow crystals, which are stable in air, were chosen from the product mixture. Electron-probe micro-analysis of a single crystal for X-ray diffraction analysis showed that it contained the elements Dy and Ta, and no Cl.

Crystal data

$DyTa_7O_{19}$	Mo $K\alpha$ radiation
$M_r = 1733.12$	$\lambda = 0.71069$ Å
Hexagonal	Cell parameters from 25 reflections
$P\bar{6}c2$	$\theta = 10$–25°
$a = 6.199$ (3) Å	$\mu = 62.78$ mm^{-1}
$c = 19.859$ (6) Å	$T = 293$ K
$V = 660.9$ (8) Å^3	Prismatic
$Z = 2$	0.30 × 0.10 × 0.10 mm
$D_x = 8.71$ Mg m^{-3}	Yellow

Data collection

Rigaku AFC-5R diffractometer	$R_{\rm int} = 0.139$
$\omega/2\theta$ scans	$\theta_{\rm max} = 40.0°$
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)	$h = 0 \rightarrow 9$
$T_{\rm min} = 0.113$, $T_{\rm max} = 0.995$	$k = 0 \rightarrow 5$
933 measured reflections	$l = 0 \rightarrow 34$
923 independent reflections	3 standard reflections monitored every 150 reflections
463 observed reflections [$I > 3.0\sigma(I)$]	intensity decay: 0.2%

Refinement

Refinement on F	$(\Delta/\sigma)_{\rm max} = 0.0323$
$R = 0.0471$	$\Delta\rho_{\rm max} = 4.47$ e Å^{-3}
$wR = 0.0548$	$\Delta\rho_{\rm min} = -5.90$ e Å^{-3}
$S = 1.23$	Extinction correction: none
463 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
29 parameters	
$w = 1/[\sigma^2(F) + (0.020F)^2 + 1.0] - F$	

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

$U_{\rm iso}$ for O atoms, $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for Ta and Dy atoms.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Ta1	2/3	1/3	0	0.0073 (3)
Ta2	0.3586 (2)	0.3631 (2)	0.15558 (4)	0.0079 (1)
Dy	1/3	2/3	0	0.0097 (5)
O1	0.375 (4)	0.408 (4)	1/4	0.007 (4)
O2	0.423 (4)	0.385 (4)	0.054 (1)	0.016 (4)
O3	0.018 (4)	0.253 (3)	0.1530 (8)	0.010 (3)
O4	2/3	1/3	0.161 (2)	0.014 (7)
O5	1/3	2/3	0.123 (2)	0.010 (6)

Table 2. *Selected geometric parameters (Å, °)*

Ta1—O2	2.01 (2)	Ta2—O4	2.011 (3)
Ta1—O2$^{\rm i}$	2.01 (2)	Ta2—O5	2.07 (1)
Ta1—O2$^{\rm ii}$	2.01 (2)	Dy—O2	2.34 (3)
Ta1—O2$^{\rm iii}$	2.01 (3)	Dy—O2$^{\rm vi}$	2.34 (2)
Ta1—O2$^{\rm iv}$	2.01 (2)	Dy—O2$^{\rm viii}$	2.34 (2)
Ta1—O2$^{\rm v}$	2.01 (2)	Dy—O2$^{\rm iii}$	2.34 (3)
Ta2—O1	1.891 (3)	Dy—O2$^{\rm ix}$	2.34 (2)
Ta2—O2	2.04 (2)	Dy—O2$^{\rm x}$	2.34 (2)
Ta2—O3	1.87 (2)	Dy—O5	2.45 (4)
Ta2—O3$^{\rm vi}$	2.45 (1)	Dy—O5$^{\rm iii}$	2.45 (4)
Ta2—O3$^{\rm vii}$	2.09 (2)		
O2—Ta1—O2$^{\rm i}$	94 (1)	O3$^{\rm vi}$—Ta2—O5	63.3 (6)
O2—Ta1—O2$^{\rm ii}$	93.7 (9)	O3$^{\rm vii}$—Ta2—O4	74.2 (7)
O2—Ta1—O2$^{\rm iii}$	75.8 (9)	O3$^{\rm vii}$—Ta2—O5	150.5 (9)
O2—Ta1—O2$^{\rm iv}$	164.0 (8)	O4—Ta2—O5	127.5 (4)
O2—Ta1—O2$^{\rm v}$	98.9 (9)	O2—Dy—O2$^{\rm vi}$	100.4 (8)
O2$^{\rm i}$—Ta1—O2$^{\rm ii}$	93.7 (8)	O2—Dy—O2$^{\rm viii}$	100.4 (8)
O2$^{\rm i}$—Ta1—O2$^{\rm iii}$	164.0 (8)	O2—Dy—O2$^{\rm iii}$	63.5 (8)
O2$^{\rm i}$—Ta1—O2$^{\rm iv}$	99 (1)	O2—Dy—O2$^{\rm ix}$	99.8 (8)
O2$^{\rm i}$—Ta1—O2$^{\rm v}$	75.8 (9)	O2—Dy—O2$^{\rm x}$	156.2 (7)
O2$^{\rm ii}$—Ta1—O2$^{\rm iii}$	98.9 (9)	O2—Dy—O5	62.5 (5)
O2$^{\rm ii}$—Ta1—O2$^{\rm iv}$	75.8 (8)	O2—Dy—O5$^{\rm iii}$	117.5 (5)
O2$^{\rm ii}$—Ta1—O2$^{\rm v}$	164 (1)	O2$^{\rm vi}$—Dy—O2$^{\rm viii}$	100.4 (7)
O2$^{\rm iii}$—Ta1—O2$^{\rm iv}$	93.7 (9)	O2$^{\rm vi}$—Dy—O2$^{\rm iii}$	99.8 (7)
O2$^{\rm iii}$—Ta1—O2$^{\rm v}$	93.7 (9)	O2$^{\rm vi}$—Dy—O2$^{\rm ix}$	156 (1)

O2$^{\rm iv}$—Ta1—O2$^{\rm v}$	93.7 (8)	O2$^{\rm vi}$—Dy—O2$^{\rm x}$	63.5 (7)
O1—Ta2—O2	166.5 (8)	O2$^{\rm vi}$—Dy—O5	62.5 (5)
O1—Ta2—O3	93 (1)	O2$^{\rm vi}$—Dy—O5$^{\rm iii}$	117.5 (5)
O1—Ta2—O3$^{\rm vi}$	85.3 (7)	O2$^{\rm viii}$—Dy—O2$^{\rm iii}$	156.2 (7)
O1—Ta2—O3$^{\rm vii}$	98.8 (9)	O2$^{\rm viii}$—Dy—O2$^{\rm ix}$	63.5 (7)
O1—Ta2—O4	89 (1)	O2$^{\rm viii}$—Dy—O2$^{\rm x}$	99.8 (8)
O1—Ta2—O5	101 (1)	O2$^{\rm viii}$—Dy—O5	62.5 (5)
O2—Ta2—O3	98.4 (8)	O2$^{\rm viii}$—Dy—O5$^{\rm iii}$	117.5 (5)
O2—Ta2—O3$^{\rm vi}$	81.4 (6)	O2$^{\rm iii}$—Dy—O2$^{\rm ix}$	100.4 (8)
O2—Ta2—O3$^{\rm vii}$	90.1 (8)	O2$^{\rm iii}$—Dy—O2$^{\rm x}$	100.4 (8)
O2—Ta2—O4	84 (1)	O2$^{\rm iii}$—Dy—O5	117.5 (5)
O2—Ta2—O5	75 (1)	O2$^{\rm iii}$—Dy—O5$^{\rm iii}$	62.5 (5)
O3—Ta2—O3$^{\rm vi}$	136.7 (9)	O2$^{\rm ix}$—Dy—O2$^{\rm x}$	100.4 (7)
O3—Ta2—O3$^{\rm vii}$	82.8 (9)	O2$^{\rm ix}$—Dy—O5	117.5 (5)
O3—Ta2—O4	156.9 (6)	O2$^{\rm ix}$—Dy—O5$^{\rm iii}$	62.5 (5)
O3—Ta2—O5	74.9 (6)	O2$^{\rm x}$—Dy—O5	117.5 (5)
O3$^{\rm vi}$—Ta2—O3$^{\rm vii}$	140.2 (9)	O2$^{\rm x}$—Dy—O5$^{\rm iii}$	62.5 (5)
O3$^{\rm vi}$—Ta2—O4	66.4 (6)	O5—Dy—O5$^{\rm iii}$	180

Symmetry codes: (i) $1-y, x-y, z$; (ii) $1-x+y, 1-x, z$; (iii) $1-y, 1-x, -z$; (iv) $1-x+y, y, -z$; (v) $x, x-y, -z$; (vi) $1-y, 1+x-y, z$; (vii) $-x+y, -x, z$; (viii) $-x+y, 1-x, z$; (ix) $-x+y, y, -z$; (x) $x, 1+x-y, -z$.

The structure was solved in space group $P\bar{6}c2$ by direct methods using *MITHRIL* (Gilmore, 1983). The coordinates of the remaining atoms were found in succeeding difference Fourier syntheses. The structure was refined by full-matrix least-squares methods using *MolEN* (Fair, 1990). Attempts were made to refine the structure in space group $P6_3/mcm$ which converged at $R = 0.049$ and $wR = 0.056$. However, taking aspects of structural chemistry and the references into account, the space group $P\bar{6}c2$ was preferably selected. (The results of the refinement in space group $P6_3/mcm$ are available from the authors).

Data collection: *CONTROL* (Molecular Structure Corporation, 1988). Cell refinement: *CONTROL*. Data reduction: Lorentz–polarization using *MolEN* (Fair, 1990). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *GCIF* (local program).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: OH1087). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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