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Pr_3MoO_7

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Mo–O) = 0.004 Å
R factor = 0.029
wR factor = 0.067
Data-to-parameter ratio = 33.1

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

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Tripraseodymium molybdenum heptaoxide, Pr_3MoO_7, is isostructural with La_3MoO_7. Its crystal structure consists of chains of corner-linked MoO_6 octahedra that are parallel with the b axis and separated from each other by seven-coordinate Pr–O polyhedra.

Comment

The Ln_3MO_7 compounds, where M is a pentavalent 4d or 5d transition element such as Nb, Mo, Ru, Ir, Os or Ta, and Ln is a rare earth, present an ordered double-fluorite structure and crystallize in various orthorhombic space groups, such as *Pnma, Cmcm, C*222_1 or $P2_12_12_1$. The main structural feature of the Ln_3MoO_7 compounds is the occurrence of zigzag chains of *trans*-corner-sharing MO_6 octahedra that are separated by seven- or eight-coordinate Ln–O polyhedra. Because of this quasi-one-dimensionality, La_3RuO_7, La_3OsO_7 (Lam *et al.*, 2002), Ln_3OsO_7 (Ln = Pr, Nd, Sm; Plaisier *et al.*, 2002), La_3MoO_7 (Greedan *et al.*, 1997), Ln_3RuO_7 (Ln = Sm, Eu; Harada & Hinatsu, 2001) and Pr_3MO_7 (M = Nb, Ta; Vente *et al.*, 1994) have been extensively studied in respect of their physical properties.

We present here the crystal structure of Pr_3MoO_7. This compound was first synthesized as a powder sample by Prévost-Czeskleba (1987) and found to crystallize in the orthorhombic space group *Cmcm*, as does Nd_3NbO_7 (Rossel, 1979).

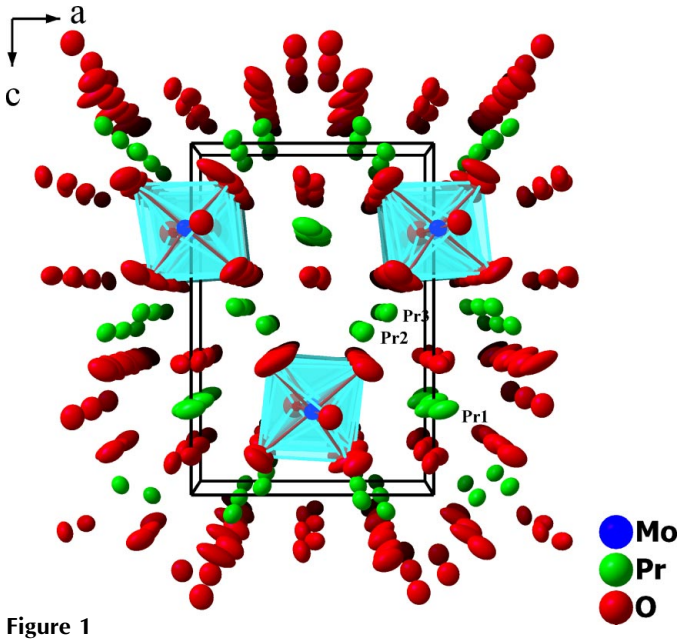

Figure 1
Perspective view of Pr_3MoO_7 along the b axis. Displacement ellipsoids are drawn at the 97% probability level.

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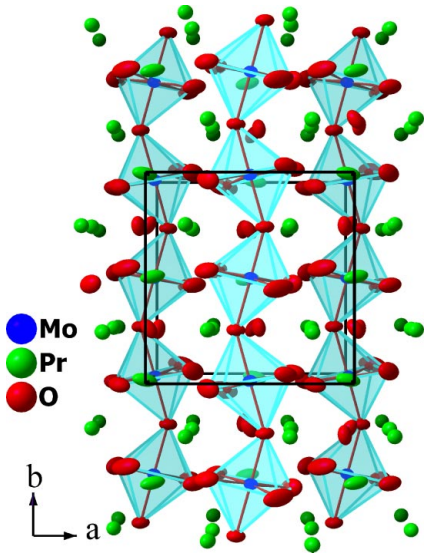

Figure 2
Perspective view of Pr_3MoO_7 along the *c* axis.

Our investigation on a single crystal indicates that Pr_3MoO_7 crystallizes in the space group $P2_12_12_1$ and is isostructural with La_3MoO_7 (Greedan *et al.*, 1997). Perspective views of Pr_3MoO_7 along the *b* and *c* axes are shown in Figs. 1 and 2, respectively. The Mo—O distances within the MoO_6 octahedra range from 1.854 (4) to 2.088 (5) Å [1.861 (3)–2.098 (4) Å in La_3MoO_7], with an average value of 1.974 Å compared to 1.981 Å in La_3MoO_7. The three crystallographically independent Pr^{3+} ions are each surrounded by seven O atoms. The oxygen environment of Pr1 can be viewed as a highly distorted cube,with one apex missing, and those of Pr2 and Pr3 as distorted pentagonal bipyramids. The Pr—O distances are in the ranges 2.382 (4)–2.690 (5), 2.302 (3)–2.658 (3) and 2.280 (4)–2.581 (3) Å for the Pr1, Pr2 and Pr3 sites, respectively. As mentioned in the Experimental, the structure shows a *Pbnm* pseudosymmetry. The non-centrosymmetric nature of the structure probably arises from the Pr1 ion that does not reside at an inversion center, as revealed by the refinement carried out in the space group *Pbnm*.

Experimental

Single crystals of Pr_3MoO_7 were prepared from a stoichiometric amount of Pr_6O_{11}, MoO_3 and Mo. The initial mixture (*ca* 5 g) was cold-pressed and loaded into a molybdenum crucible, which was sealed under a low argon pressure using an arc-welding system. The charge was heated at a rate of 300 K h^{-1} to 1973 K, held at this temperature for 10 min, then cooled at a rate of 100 K h^{-1} to 1373 K and finally furnace-cooled.

Crystal data

Pr_3MoO_7
M_r = 630.67
Orthorhombic, $P2_12_12_1$
a = 7.5087 (1) Å
b = 7.6412 (2) Å
c = 10.8952 (2) Å
V = 625.12 (2) Å^3
Z = 4
D_x = 6.701 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11425 reflections
θ = 1–37.8°
μ = 24.91 mm^{-1}
T = 293 (2) K
Irregular block, black
0.09 × 0.06 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.139, T_{max} = 0.225
16521 measured reflections
3345 independent reflections
3222 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 37.8°
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 12$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.067
S = 1.05
3345 reflections
101 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0091P)^2 + 7.1924P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 4.07 e Å^{-3} (0.55 Å from Pr1)
$\Delta\rho_{min}$ = −4.05 e Å^{-3} (0.55 Å from Pr1)
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0110 (3)
Absolute structure: Flack (1983)
Flack parameter = 0.49 (3)

Table 1
Selected geometric parameters (Å, °).

Mo—O2^{i}	1.854 (4)	Pr2—O7	2.349 (3)
Mo—O6^{ii}	1.920 (5)	Pr2—O4^{ii}	2.392 (4)
Mo—O5^{iii}	1.954 (3)	Pr2—O1^{ix}	2.416 (4)
Mo—O1^{i}	2.008 (5)	Pr2—O6	2.524 (4)
Mo—O5^{iv}	2.018 (3)	Pr2—O2^{x}	2.527 (4)
Mo—O4^{ii}	2.088 (5)	Pr2—O5^{iv}	2.658 (3)
Pr1—O7^{v}	2.382 (4)	Pr3—O3	2.280 (4)
Pr1—O7	2.406 (4)	Pr3—O3^{xi}	2.293 (4)
Pr1—O3^{vi}	2.414 (5)	Pr3—O4	2.414 (4)
Pr1—O3^{iv}	2.420 (5)	Pr3—O1^{x}	2.458 (4)
Pr1—O4^{ii}	2.447 (5)	Pr3—O6^{i}	2.509 (4)
Pr1—O6^{ii}	2.678 (5)	Pr3—O2^{viii}	2.531 (4)
Pr1—O1^{vii}	2.690 (5)	Pr3—O5	2.581 (3)
Pr2—O7^{viii}	2.302 (3)		
Mo^{xii}—O5—Mo^{xiii}	148.3 (2)		

Symmetry codes: (i) $\frac{3}{2}-x, 2-y, z-\frac{1}{2}$; (ii) $\frac{3}{2}-x, 2-y, \frac{1}{2}+z$; (iii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x, y, 1+z$; (v) $2-x, \frac{1}{2}+y, \frac{3}{2}-z$; (vi) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$; (vii) $\frac{5}{2}-x, 2-y, z-\frac{1}{2}$; (viii) $x-\frac{1}{2}, \frac{3}{2}-y, 1-z$; (ix) $x-\frac{1}{2}, \frac{3}{2}-y, 2-z$; (x) $2-x, y-\frac{1}{2}, \frac{3}{2}-z$; (xi) $x-\frac{1}{2}, \frac{3}{2}-y, -z$; (xii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (xiii) $x, y, z-1$.

Systematic absences were consistent only with the non-centrosymmetric space group $P2_12_12_1$. The atomic coordinates of La, Mo and O from La_3MoO_7 (Greedan *et al.*, 1997) were used as starting positions in the first stages of the refinement in the present study. Attempts to refine the structure in the space group *Pbnm*, as suggested by *PLATON* (Spek, 1998), were unsuccessful and led to an *R* factor of about 0.10. Refinement of the Flack (1983) parameter gave a value of 0.49 (3), indicating that the crystal studied is a racemic twin.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *SHELXL*97.

Intensity data were collected on the Nonius KappaCCD X-ray diffactometer system of the 'Centre de diffactométrie de l'Université de Rennes I'.

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