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Tripraseodymium pentairon(III) dodecaoxide, Pr₃Fe₅O₁₂: a synchrotron radiation study

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Key indicators: single-crystal synchrotron study; $T = 298$ K; mean $\sigma(\text{Pr}-\text{Fe}) = 0.000$ Å; R factor = 0.019; wR factor = 0.021; data-to-parameter ratio = 550.1.

The title compound, pentairon tripraseodymium dodecaoxide (PrIG), has an iron garnet structure. There are two Fe site symmetries. One of the Fe atoms is coordinated by six O atoms, forming a slightly distorted octahedron, and has $\bar{3}$ site symmetry. The other Fe atom is coordinated by four O atoms, forming a slightly distorted tetrahedron, and has $\bar{4}$ site symmetry. FeO₆ octahedra and FeO₄ tetrahedra are linked together by corners. The Pr atom is coordinated by eight O atoms, forming a distorted dodecahedron, and has 222 site symmetry. The O atoms occupy the general positions.

Related literature

The title compound is isotypic with the $Ia\bar{3}d$ form of Y₃Fe₅O₁₂ (YIG). For related structures, see: Bonnet *et al.* (1975). For details of the crystal growth from low-temperature liquid-phase epitaxy, see: Fratello *et al.* (1986). For the extinction correction, see: Becker & Coppens (1975). X-ray intensities were measured avoiding multiple diffraction, see: Takenaka *et al.* (2008).

Experimental

Crystal data

Pr₃Fe₅O₁₂
 $M_r = 893.98$
Cubic, $Ia\bar{3}d$
 $a = 12.6302$ (3) Å
 $V = 2014.79$ (8) Å³
 $Z = 8$

Synchrotron radiation
 $\lambda = 0.67171$ Å
 $\mu = 17.41$ mm⁻¹
 $T = 298$ K
0.035 mm (radius)

Data collection

Rigaku AFC four-circle diffractometer
Absorption correction: for a sphere [transmission coefficients for spheres tabulated in *International Tables C* (1992), Table 6.3.3.3, were interpolated with Lagrange's method (four-point interpolation; Yamauchi *et al.*, 1965)]
 $T_{\min} = 0.413$, $T_{\max} = 0.441$
9351 measured reflections
1728 independent reflections
1728 reflections with $F > 3\sigma(F)$
 $R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.021$
 $S = 1.06$
9351 reflections
17 parameters
 $\Delta\rho_{\max} = 2.52$ e Å⁻³
 $\Delta\rho_{\min} = -3.16$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pr1—O1	2.42410 (10)	Fe1—O1	2.03220 (10)
Pr1—O1 ⁱ	2.54010 (10)	Fe2—O1 ⁱⁱ	1.87450 (10)
O1—Fe1—O1 ⁱ	85.87 (1)	O1 ⁱⁱ —Fe2—O1 ^{iv}	100.02 (1)
O1 ⁱⁱ —Fe2—O1 ⁱⁱⁱ	114.39 (1)		

Symmetry codes: (i) z, x, y ; (ii) $x + \frac{1}{2}, y, -z + \frac{1}{2}$; (iii) $-x + \frac{1}{4}, z - \frac{1}{4}, y + \frac{1}{4}$; (iv) $x + \frac{1}{2}, -y, z$.

Data collection: *AFC-5*, specially designed for PF-BL14A (Rigaku, 1984) and *IUANGLE* (Tanaka *et al.*, 1994); cell refinement: *RSLC-3 UNICS* system (Sakurai & Kobayashi, 1979); data reduction: *RDEDIT* (Tanaka, 2008); program(s) used to solve structure: *QNTAO* (Tanaka *et al.*, 2008); program(s) used to refine structure: *QNTAO*; molecular graphics: *ATOMS for Windows* (Dowty, 2000); software used to prepare material for publication: *RDEDIT*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2121).

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supplementary materials

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Tripraseodymium pentairon(III) dodecaoxide, $\text{Pr}_3\text{Fe}_5\text{O}_{12}$: a synchrotron radiation study

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Comment

The title compound, $\text{Pr}_3\text{Fe}_5\text{O}_{12}$ (PrIG), was difficult to be grown. It was grown by the low-temperature-liquid-phase epitaxy for the first time by Fratello *et al.* (1986). Though the crystal structure was assumed as iron-garnet-type structure by lattice constant and extinction rule, the complete structure was not determined. In this paper, we determine the O atom position and the complete structure by the full matrix least-squares program QNTAO. Since the R-factor is small and the residual density has no significant peaks where no atoms exists, the structure was finally determined to be iron-garnet structure. It is isotopic with the $\text{Ia}\bar{3}\text{d}$ form of $\text{Y}_3\text{Fe}_5\text{O}_{12}$ (YIG). (Bonnet *et al.*, 1975). The Pr atom is coordinated by eight oxygen atoms. It forms a distorted dodecahedron. There are two Fe site symmetries. One of the Fe atom is coordinated by six oxygen atoms. It forms a slightly distorted octahedron. The other Fe atom is coordinated by four oxygen atoms. It forms a slightly distorted tetrahedron. FeO_6 octahedron and FeO_4 tetrahedron are linked together by corners. The structure of PrIG is drawn in Fig.1. And displacement ellipsoids of PrO_8 is drawn in Fig.2.

Experimental

Single crystals of praseodymium iron garnet were prepared by low temperature liquid phase epitaxy on $\text{Sm}_3(\text{ScGa})_5\text{O}_{12}$ seeds with lattice parameters near the projected values for PrIG.

Refinement

X-ray intensities were measured avoiding multiple diffraction. (Takenaka *et al.*, 2008).

Figures

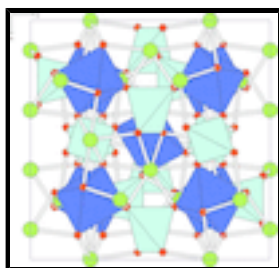


Fig. 1. The structure of $\text{Pr}_3\text{Fe}_5\text{O}_{12}$. Small red and large green spheres represent O and Pr atoms, respectively. Purple octahedron and blue tetrahedron represent FeO_6 and FeO_4 units, respectively.

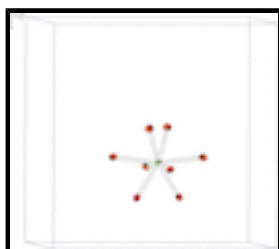


Fig. 2. View of PrO_8 with displacement ellipsoids at the 90% probability level. Green and red ellipsoids represent Pr and O atoms, in Fig.1.

Pentairon tripraseodymium dodecaoxide

Crystal data

Pr ₃ Fe ₅ O ₁₂	$D_x = 5.894 \text{ Mg m}^{-3}$
$M_r = 893.98$	Synchrotron radiation, $\lambda = 0.67171 \text{ \AA}$
Cubic, $Ia\bar{3}d$	Cell parameters from 9 reflections
Hall symbol: -I 4bd 2c 3	$\theta = 17.5\text{--}52.3^\circ$
$a = 12.6302 (3) \text{ \AA}$	$\mu = 17.41 \text{ mm}^{-1}$
$V = 2014.79 (8) \text{ \AA}^3$	$T = 298 \text{ K}$
$Z = 8$	Sphere, black
$F(000) = 3224$	0.04 mm (radius)

Data collection

Rigaku AFC four-circle diffractometer	1728 independent reflections
Si 111	1728 reflections with $F > 3\sigma(F)$
Detector resolution: $1.25 \times 1.25^\circ \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.016$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 68.3^\circ$, $\theta_{\text{min}} = 3.7^\circ$
Absorption correction: for a sphere [Transmission coefficients for spheres tabulated in International Tables C (1992), Table 6.3.3.3, were interpolated with Lagrange's method (four-point interpolation (Yamauchi <i>et al.</i> , 1965))]	$h = -9 \rightarrow 34$
$T_{\text{min}} = 0.413$, $T_{\text{max}} = 0.441$	$k = -9 \rightarrow 32$
9351 measured reflections	$l = -9 \rightarrow 34$

Refinement

Refinement on F	Primary atom site location: isomorphous structure methods
Least-squares matrix: full	Weighting scheme based on measured s.u.'s
$R[F^2 > 2\sigma(F^2)] = 0.019$	$(\Delta/\sigma)_{\text{max}} = 0.003$
$wR(F^2) = 0.021$	$\Delta\rho_{\text{max}} = 2.52 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -3.16 \text{ e \AA}^{-3}$
9351 reflections	Extinction correction: B-C type 1 Gaussian isotropic (Becker & Coppens, 1975)
17 parameters	Extinction coefficient: 0.255 (5)

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pr1	0.125000	0.000000	0.250000	0.00531 (1)
Fe1	0.000000	0.000000	0.000000	0.00512 (1)
Fe2	0.375000	0.000000	0.250000	0.00533 (1)
O1	-0.029622 (2)	0.052553 (2)	0.149166 (2)	0.00711 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pr1	0.00406 (2)	0.00594 (2)	0.00594 (2)	0	0	0.00111 (1)
Fe1	0.00512 (2)	0.00512 (2)	0.00512 (2)	-0.00023 (1)	-0.00023 (1)	-0.00023 (1)
Fe2	0.00411 (3)	0.00594 (2)	0.00594 (2)	0	0	0
O1	0.00718 (8)	0.00829 (8)	0.00587 (7)	-0.00004 (6)	0.00080 (6)	0.00038 (6)

Geometric parameters (\AA , $^\circ$)

Pr1—O1	2.42410 (10)	Fe1—O1 ⁱ	2.03220 (10)
Pr1—O1 ⁱ	2.54010 (10)	Fe1—O1 ^{viii}	2.03220 (10)
Pr1—O1 ⁱⁱ	2.42410 (10)	Fe1—O1 ^{ix}	2.03220 (10)
Pr1—O1 ⁱⁱⁱ	2.54010 (10)	Fe1—O1 ^x	2.03220 (10)
Pr1—O1 ^{iv}	2.42410 (10)	Fe1—O1 ^{xi}	2.03220 (10)
Pr1—O1 ^v	2.54010 (10)	Fe2—O1 ^{xii}	1.87450 (10)
Pr1—O1 ^{vi}	2.42410 (10)	Fe2—O1 ^{iv}	1.87450 (10)
Pr1—O1 ^{vii}	2.54010 (10)	Fe2—O1 ^{xiii}	1.87450 (10)
Fe1—O1	2.03220 (10)	Fe2—O1 ^{vi}	1.87450 (10)
O1—Pr1—O1 ⁱ	67.75 (1)	O1—Fe1—O1 ^{viii}	85.87 (1)
O1—Pr1—O1 ⁱⁱ	72.66 (1)	O1—Fe1—O1 ^{ix}	180.00
O1—Pr1—O1 ⁱⁱⁱ	124.91 (1)	O1—Fe1—O1 ^x	94.13 (1)
O1—Pr1—O1 ^{iv}	111.18 (1)	O1—Fe1—O1 ^{xi}	94.13 (1)
O1—Pr1—O1 ^v	73.25 (1)	O1 ^{xii} —Fe2—O1 ^{vi}	114.39 (1)
O1—Pr1—O1 ^{vi}	159.51 (1)	O1 ^{xii} —Fe2—O1 ^{iv}	114.39 (1)
O1—Pr1—O1 ^{vii}	95.43 (1)	O1 ^{xii} —Fe2—O1 ^{xiii}	100.02 (1)
O1—Fe1—O1 ⁱ	85.87 (1)		

Symmetry codes: (i) z, x, y ; (ii) $x, -y, -z+1/2$; (iii) $z, -x, -y+1/2$; (iv) $-x+1/4, -z+1/4, -y+1/4$; (v) $-z+1/4, -y+1/4, -x+1/4$; (vi) $-x+1/4, z-1/4, y+1/4$; (vii) $-z+1/4, y-1/4, x+1/4$; (viii) y, z, x ; (ix) $-x, -y, -z$; (x) $-z, -x, -y$; (xi) $-y, -z, -x$; (xii) $x+1/2, y, -z+1/2$; (xiii) $x+1/2, -y, z$.

Fig. 1

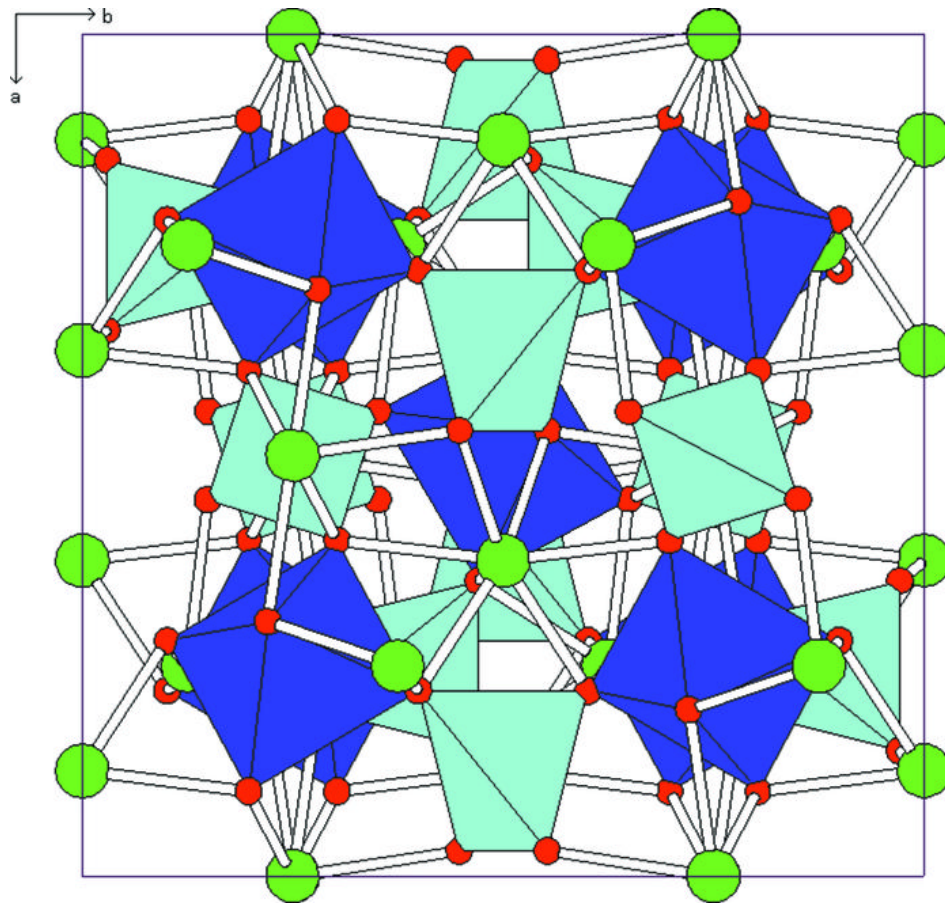


Fig. 2

