

O51	0.5582 (6)	0.4765 (5)	0.4534 (3)	0.031 (2)
O52	0.4575 (5)	0.4567 (5)	0.3887 (3)	0.024 (2)
O53	0.0392 (5)	0.4479 (5)	0.4183 (3)	0.021 (2)
O54	-0.0722 (5)	0.4029 (5)	0.3621 (3)	0.020 (2)
O61	0.2015 (5)	0.4232 (5)	0.4644 (3)	0.023 (2)
O62	0.2101 (5)	0.3365 (5)	0.3989 (3)	0.019 (2)
O63	0.0802 (5)	0.3143 (5)	0.4243 (3)	0.017 (2)
O64	0.1628 (5)	0.4276 (5)	0.3729 (3)	0.017 (2)
O1W	0.5291 (10)	0.4235 (10)	1/4	0.050 (4)
O2W	0	0	0.1568 (11)	0.082 (8)
O3W	0.1497 (7)	0.0837 (8)	0.9203 (7)	0.087 (5)
O4WA	0.0825 (14)	0.1478 (14)	0.8048 (8)	0.046 (6)
O4WB	0.1141 (17)	0.1205 (18)	0.8006 (10)	0.066 (7)
O5W	0.5807 (7)	0.1833 (7)	0.9102 (5)	0.051 (3)
O6W	-0.2386 (16)	0.2775 (16)	0.3971 (10)	0.116 (12)
O7W	2/3	1/3	0.981 (2)	0.37 (5)
O8W	0.047 (3)	-0.026 (3)	0.9702 (19)	0.025 (19)

Table 2. Selected geometric parameters (Å)

Tb1—O52	2.502 (11)	S10—O12	1.480 (14)
Tb1—O62	2.507 (10)	S10—O13 ⁱⁱⁱ	1.507 (8)
Tb1—O4	2.525 (9)	S20—O21 ^{iv}	1.462 (14)
Tb1—O3	2.532 (8)	S20—O22 ^{iv}	1.465 (13)
Tb1—O6	2.544 (10)	S20—O23 ^{iv}	1.486 (9)
Tb1—O5	2.553 (9)	S30—O31 ⁱ	1.24 (2)
Tb1—O7	2.555 (10)	S30—O33 ^v	1.475 (9)
Tb1—O2	2.562 (9)	S30—O32 ⁱ	1.58 (2)
Tb1—O42 ⁱ	2.565 (10)	S40—O42 ^{vi}	1.457 (9)
Tb2—O1 ⁱⁱ	2.547 (10)	S40—O41 ^{vi}	1.486 (9)
Tb2—O21	2.598 (14)	S40—O43 ^{vi}	1.513 (10)
Ce3—O33	2.291 (9)	S40—O44 ^{vi}	1.524 (8)
Ce3—O13	2.319 (8)	S50—O52 ^{vii}	1.467 (10)
Ce3—O23	2.320 (9)	S50—O51 ^{vii}	1.487 (9)
Ce3—O63	2.407 (8)	S50—O54 ^{viii}	1.512 (9)
Ce3—O53	2.424 (9)	S50—O53 ^{viii}	1.515 (10)
Ce3—O43	2.446 (8)	S60—O62 ^{ix}	1.470 (9)
Ce3—O54	2.465 (10)	S60—O61 ^{ix}	1.491 (9)
Ce3—O44	2.482 (10)	S60—O63 ^{ix}	1.505 (9)
Ce3—O64	2.513 (10)	S60—O64 ^{ix}	1.505 (8)
S10—O11	1.431 (12)		

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

An area-detector system was used for data collection. Cell dimensions were refined from 250 reflections selected from two regions 90° apart and 5° wide at $\kappa = 0^\circ$. Intensity standards were not measured by the area detector. Possible variations were checked by comparing intensities of common or symmetry-related reflections as they occurred during data collection. In this case no variation was noted.

Data collection: *FAST-MADNESS* (Enraf-Nonius, 1990). Cell refinement: *FAST-MADNESS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: LI1136). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monoclinic Mg₂B₂O₅

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Abstract

The title monoclinic magnesium pyroborate was synthesized by solid-state reaction and its crystal structure determined by X-ray diffraction techniques. The structure is built from tetrameric chains of *trans*-edge-shared Mg octahedra (Mg₄O₁₈), held together by B atoms which form B₂O₅ (double-triangle) groups. The monoclinic and triclinic forms of Mg₂B₂O₅ are heteromorphic.

Comment

The MgO–B₂O₃ system has been studied extensively (Toropov & Kononov, 1940; Davis & Knight, 1945; Block, Burley, Perloff & Mason, 1959; Nefedov, 1961; Kuzel, 1964; Mutluer & Timucin, 1975). Takeuchi (1952) has described the crystal structure of the monoclinic mineral suanite from the Suan mine, North Korea. Mrose & Fleischer (1963) studied and compared X-ray diffraction data of the monoclinic and triclinic forms of Mg₂B₂O₅. A recent paper by us (Guo, Cheng, Chen, Huang & Zhang, 1995) describes the crystal structure of triclinic Mg₂B₂O₅. In this work, we report the synthesis and crystal structure of the monoclinic form of Mg₂B₂O₅.

The monoclinic and triclinic forms of Mg₂B₂O₅ are heteromorphic. Fig. 1 shows the structure of monoclinic Mg₂B₂O₅. The structure of the monoclinic form of Mg₂B₂O₅ is based on Mg₄O₁₈ tetramers, composed of four Mg octahedra linked by three shared edges, and connected by sharing four O···O edges to form

chains extending along [010]. The B atoms hold the chains together and form B₂O₅ (double-triangle) groups. The Mg···Mg distances in the Mg₄O₁₈ tetramers are 3.148 (3) and 3.152 (3) Å. The O atoms of each B₂O₅ group are not quite coplanar; the dihedral angle between the two slanting trigonal-planar BO₃ groups is 14.90°.

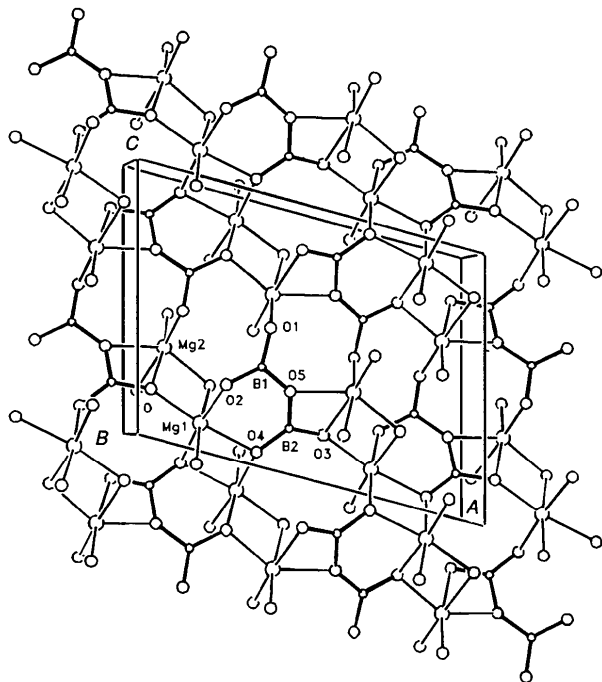


Fig. 1. The structure of Mg₂B₂O₅ viewed along [010]. The large circles represent Mg atoms, medium circles O atoms, and small circles B atoms.

Experimental

Crystals of Mg₂B₂O₅ with the monoclinic structure were grown from a mixture containing H₃BO₃ and Mg₂(OH)₂CO₃. The mixture was preheated in a platinum crucible in air at 773 K for 24 h, and at 1223 K for 24 h, and then kept at 1473 K for 48 h. The temperature was quickly quenched to 1093 K and slowly cooled from 1093 to 1053 K at a rate of 0.56 K h⁻¹, and then air-quenched. Colourless crystals of the monoclinic form of Mg₂B₂O₅ were obtained. Crystals of triclinic Mg₂B₂O₅ are obtained when the temperature is slowly cooled from 1273 to 1223 K, at a rate of 0.26 K h⁻¹.

Crystal data

Mg₂B₂O₅
M_r = 150.24
 Monoclinic
*P*2₁/*c*
a = 9.197 (4) Å
b = 3.1228 (9) Å
c = 12.303 (4) Å
 β = 104.26 (3)°
V = 342.5 (4) Å³
Z = 4
D_x = 2.91 Mg m⁻³

Mo K α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 10–20°
 μ = 0.57 mm⁻¹
T = 293 K
 Rectangular
 0.30 × 0.10 × 0.10 mm
 Colourless

Data collection

Rigaku AFC-5R diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.47, *T_{max}* = 1.00
 2517 measured reflections
 2297 independent reflections
 642 observed reflections
 $[I > 4\sigma(I)]$

R_{int} = 0.044
 θ_{\max} = 40.0°
h = 0 → 16
k = 0 → 5
l = -22 → 21
 3 standard reflections monitored every 200 reflections
 intensity decay: 0.4%

Refinement

Refinement on *F*
R = 0.049
 wR = 0.053
S = 1.29
 642 reflections
 82 parameters
 $w = 1/\sigma^2(F)$
 $(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.65 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.65 \text{ e \AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Mg1	0.1023 (2)	0.2133 (8)	0.1858 (1)	0.0056 (4)
Mg2	0.6440 (2)	0.2961 (8)	0.8947 (1)	0.0065 (5)
O1	0.4957 (4)	-0.802 (1)	0.1370 (3)	0.0071 (8)
O2	0.2582 (4)	-0.278 (1)	0.2381 (3)	0.0058 (8)
O3	0.1804 (4)	0.220 (1)	0.0463 (3)	0.0071 (8)
O4	-0.0535 (4)	-0.291 (1)	0.1522 (3)	0.0065 (8)
O5	0.3005 (4)	-0.426 (1)	-0.0693 (3)	0.0069 (8)
B1	0.3573 (5)	-0.228 (2)	0.3411 (4)	0.005 (1)
B2	0.1683 (6)	-0.654 (2)	-0.0606 (4)	0.005 (1)

Table 2. Selected geometric parameters (Å, °)

Mg1—Mg1 ⁱⁱ	3.152 (3)	Mg2—O2 ^{vi}	2.050 (4)
Mg1—Mg2 ⁱⁱⁱ	3.148 (3)	Mg2—O3 ^{vi}	2.270 (4)
Mg1—O2	2.090 (4)	Mg2—O3 ⁱⁱ	2.200 (4)
Mg1—O2 ⁱⁱⁱ	2.131 (4)	Mg2—O5 ^{vi}	2.122 (4)
Mg1—O3	2.017 (4)	B1—O1 ^{viii}	1.332 (6)
Mg1—O4	2.100 (5)	B1—O2	1.375 (5)
Mg1—O4 ⁱⁱⁱ	2.082 (5)	B1—O5 ^{viii}	1.416 (7)
Mg1—O4 ^{iv}	2.147 (4)	B2—O3 ^{ix}	1.352 (6)
Mg2—O1 ^v	1.983 (5)	B2—O4 ^x	1.352 (5)
Mg2—O1 ^{vi}	2.013 (5)	B2—O5	1.435 (7)
O2—Mg1—O2 ⁱⁱⁱ	95.4 (2)	O1 ^v —Mg2—O3 ⁱⁱ	170.5 (2)
O2—Mg1—O3	84.7 (2)	O1 ^v —Mg2—O5 ^{vi}	109.3 (2)
O2—Mg1—O4	83.9 (2)	O1 ^{vi} —Mg2—O2 ⁱⁱ	104.6 (2)
O2—Mg1—O4 ⁱⁱⁱ	173.7 (2)	O1 ^{vi} —Mg2—O3 ⁱⁱ	171.4 (2)
O2—Mg1—O4 ^{iv}	89.7 (2)	O1 ^{vi} —Mg2—O3 ⁱⁱⁱ	84.6 (2)
O2 ⁱⁱⁱ —Mg1—O3	84.0 (2)	O1 ^{vi} —Mg2—O5 ^{vi}	91.6 (2)
O2 ⁱⁱⁱ —Mg1—O4	173.8 (2)	O2 ^{vi} —Mg2—O3 ⁱⁱ	79.5 (2)
O2 ⁱⁱⁱ —Mg1—O4 ⁱⁱⁱ	83.4 (2)	O2 ^{vi} —Mg2—O3 ⁱⁱⁱ	81.5 (2)
O2 ⁱⁱⁱ —Mg1—O4 ^{iv}	90.2 (2)	O2 ^{vi} —Mg2—O5 ^{vi}	140.2 (2)
O3—Mg1—O4	102.1 (2)	O3 ^{vi} —Mg2—O3 ⁱⁱ	88.6 (1)
O3—Mg1—O4 ⁱⁱⁱ	101.3 (2)	O3 ^{vi} —Mg2—O5 ^{vi}	80.7 (1)
O3—Mg1—O4 ^{iv}	171.5 (1)	O3 ^{vi} —Mg2—O5 ^{vi}	63.9 (1)
O4—Mg1—O4 ⁱⁱⁱ	96.6 (2)	O1 ^{viii} —B1—O2	125.1 (5)
O4—Mg1—O4 ^{iv}	83.7 (2)	O1 ^{viii} —B1—O5 ^{viii}	116.9 (4)
O4 ⁱⁱⁱ —Mg1—O4 ^{iv}	84.1 (2)	O2—B1—O5 ^{viii}	118.0 (4)
O1 ^v —Mg2—O1 ^{vi}	102.8 (2)	O3 ^{ix} —B2—O4 ^x	129.4 (5)
O1 ^v —Mg2—O2 ^{vi}	102.2 (2)	O3 ^{ix} —B2—O5	110.4 (4)
O1 ^v —Mg2—O3 ^{vi}	83.5 (2)	O4 ^x —B2—O5	120.2 (5)

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

The unit cell is similar to that of the monoclinic mineral suanite. Hence, at the beginning of the refinement, the Mg, O and B atomic positions from the previously studied structure [which was determined using rotation and Weissenberg photography by trial-and-error and Fourier series methods (Takeuchi, 1952)] were used, but the refinement failed. The structure was then solved by direct methods (Gilmore, 1983). The positions of the Mg atoms were located by direct methods and those of other atoms from successive Fourier syntheses. The structure was refined anisotropically using full-matrix least-squares techniques.

Data collection: *CONTROL* (Molecular Structure Corporation, 1988). Cell refinement: *CONTROL*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). 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: DU1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tripraseodymium Chloride Bis-(orthosilicate), $\text{Pr}_3(\text{SiO}_4)_2\text{Cl}$

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Abstract

$\text{Pr}_3(\text{SiO}_4)_2\text{Cl}$ is isostructural with $\text{La}_3(\text{SiO}_4)_2\text{Cl}$. The $\text{Pr}(1)\text{O}_7\text{Cl}$ polyhedron is irregular. The $\text{Pr}(2)\text{O}_8\text{Cl}_2$ polyhedron is a distorted square antiprism with the Cl atom capping distorted square faces. The site of $\text{Pr}(2)$ can also be described as at the centre of a $\text{Pr}(2)(\text{SiO}_4)_4\text{Cl}_2$ octahedron. The structure comprises layers of $\text{Pr}(2)(\text{SiO}_4)_4\text{Cl}_2$ octahedra separated in the *a* direction by $\text{Pr}(1)$ cations occupying the $\text{Pr}(1)\text{O}_7\text{Cl}$ polyhedral sites. The Cl atom is four-coordinate forming a distorted square.

Comment

Few lanthanide chlorosilicates have been prepared. The structure of the rare earth chlorosilicate orthorhombic $\text{Yb}_3(\text{SiO}_4)_2\text{Cl}$ was the first to be determined (Ayasse & Eick, 1973). Yamada, Kano & Tanabe (1978) reported the isostructural phases $\text{Y}_3(\text{SiO}_4)_2\text{Cl}$ and $\text{Gd}_3(\text{SiO}_4)_2\text{Cl}$, and their luminescent properties when doped with Ce^{3+} . Gravereau, Es-Sakhi & Fouassier (1988) reported the crystal structure of monoclinic $\text{La}_3(\text{SiO}_4)_2\text{Cl}$, and an X-ray structural study on a single crystal of orthorhombic $\text{La}_3(\text{SiO}_4)\text{Cl}_5$ (Gravereau, Es-Sakhi & Fouassier, 1989). The crystal structure of monoclinic $\text{La}_3(\text{Si}_2\text{O}_7)\text{Cl}_3$ was recently determined (Chen, Guo, Huang & Zhang, 1995). The present paper reports the crystal structure of $\text{Pr}_3(\text{SiO}_4)_2\text{Cl}$.

$\text{Pr}(1)$ and $\text{Pr}(2)$ have O_7Cl and O_8Cl_2 environments, respectively. The $\text{Pr}(1)\text{O}_7\text{Cl}$ polyhedron is irregular (Fig. 1). The $\text{Pr}(2)\text{O}_8\text{Cl}_2$ polyhedron is a distorted