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$SrMgP_2O_7$

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Mg–O) = 0.003 Å
R factor = 0.036
wR factor = 0.071
Data-to-parameter ratio = 17.8

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

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The structure of strontium magnesium diphosphate, $SrMgP_2O_7$, belongs to the α-$Ca_2P_2O_7$ isotypical pyrophosphates series $SrMP_2O_7$ (M = Cr, Mn, Fe, Co, Ni, Cu, Zn). Mg^{II} atoms occupy square-pyramidal coordination and are isolated in the structure. [MgO_5] units and the [P_2O_7] groups form a three-dimensional framework with channels along [100] and [010], in which are located Sr^{2+} ions.

Comment

Systematic preparative and structural studies of pyrophosphates $M_2P_2O_7$ and $(A,M)_2P_2O_7$ with A and/or M an alkaline earth or divalent 3d-metal ion, have been undertaken during the last two decades. The pseudo-binary system $Sr_2P_2O_7$—$Mg_2P_2O_7$ has been investigated by Calvo, who reported the α-$Ca_2P_2O_7$ (Calvo, 1968*a*) isostructural pyrophosphate $SrMgP_2O_7$, but with insufficient crystal data (Calvo, 1968*b*). We undertook the study of this system to determine the crystal structure of $SrMgP_2O_7$. The structures of the limiting phases

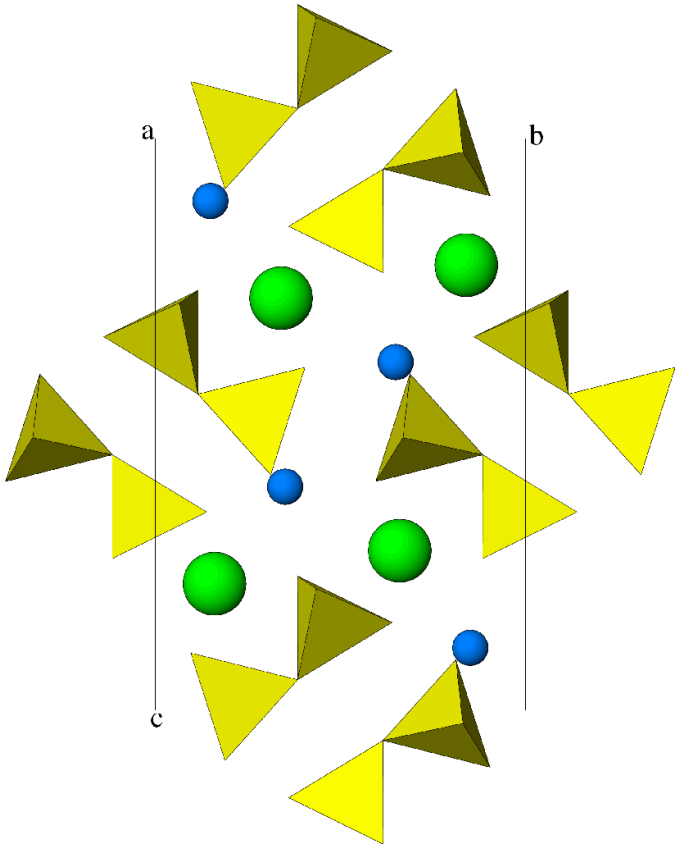

Figure 1
Projection of the crystal structure of $SrMgP_2O_7$ along the a axis. Sr and Mg are shown as green and blue balls respectively. PO_4 tetrahedra are yellow.

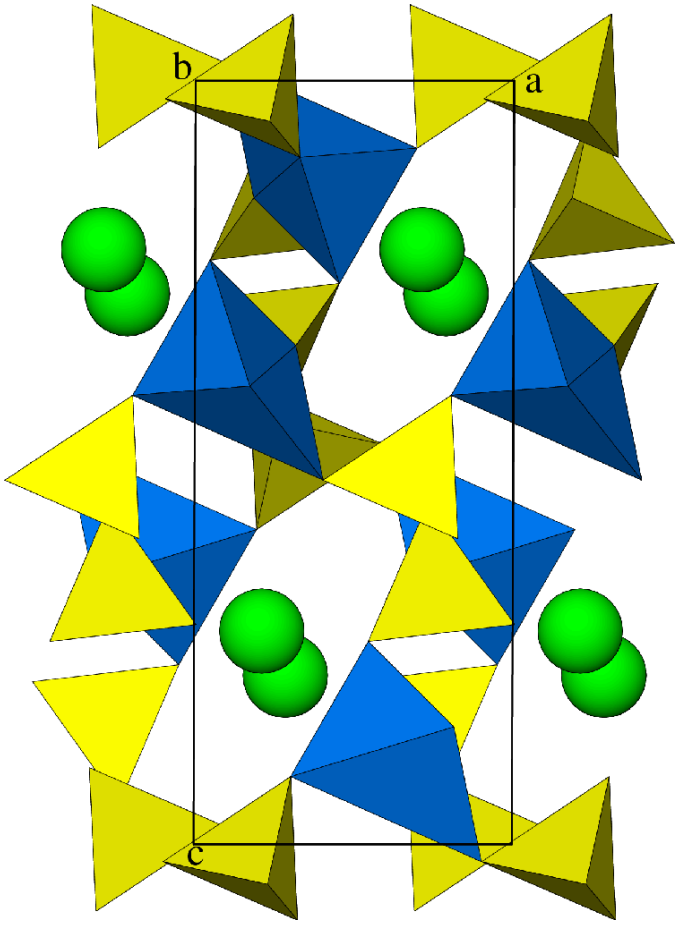

Figure 2
Projection of the crystal structure along the *b* axis with Mg polyhedra (blue). Sr atoms are shown as green balls and PO_4 as yellow tetrahedra.

are known: $Sr_2P_2O_7$ (Hagmann, 1968), $Mg_2P_2O_7$ [α- (Calvo, 1967) and β- (Calvo, 1965)]. Mixed pyrophosphates $SrMP_2O_7$ form a series of compounds, all isostructural to α-$Ca_2P_2O_7$. The main feature of these pyrophosphates, compared to the structure of α-$Ca_2P_2O_7$, is that *A* and *M* occupy respectively the Ca(1) and Ca(2) positions of the Ca in the structure. The result of such 'substitution' is a decrease of coordination from 8 to 5 for *M*, which leads to isolated [MO_5] groups instead of [Ca_2O_x] dimers. Known isotypic pyrophosphates $SrMP_2O_7$ [*M* = Cr (Maaß & Glaum, 2000), Mn (Maaß *et al.*, 1999), Fe (LeMeins & Courbion, 1999), Co (Riou & Raveau, 1991), Ni (El–Bali *et al.*, 2001), Cu (Moqine *et al.*, 1993), Zn (Murashova *et al.*, 1991)] all show square-pyramidal [MO_5] units. To complete this investigation, we report in this paper the synthesis of $SrMgP_2O_7$ and its crystal structure refinement from X-ray single-crystal data.

The structure of the title pyrophosphate can be described as an association of [MgO_5] and [P_2O_7] groups. Corner-sharing of these two building units results in a three-dimensional framework with tunnels running along [001] and [010], in which Sr^{+2} ions are located. Fig. 1 depicts an *ATOMS* projection (Dowty, 1999), on to [100], of the unit cell of $SrMgP_2O_7$. This structure belongs to the previously mentioned

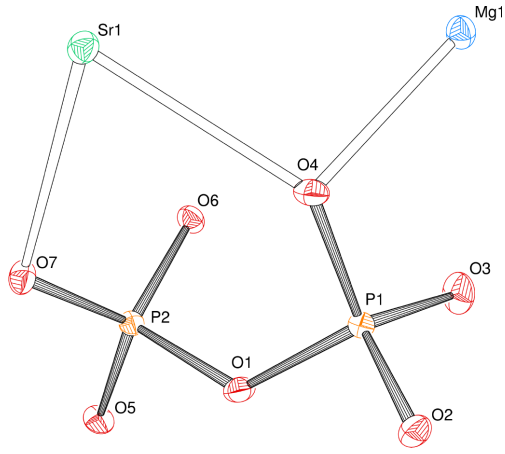

Figure 3
Atomic displacement ellipsoids at 50% probability.

set of pyrophosphates $SrMP_2O_7$; it is thus isostructural to α-$Ca_2P_2O_7$. Mg behaves like the other *M* atoms in this series, showing a square-pyramidal coordination. Each Mg^{2+} is coordinated by five O atoms from five of the $[P_2O_7]^{4-}$ counter-ions. The five short Mg—O contacts range from 1.986 to 2.116 Å, with an average d_{Mg-O} of 2.046 Å. This latter value can be compared to the similar distance, 2.044 Å, found in the homologous coordination in α-$Mg_2P_2O_7$. The MgO_5 polyhedra are isolated in the structure, where the two nearest Mg^{2+} ions are at 3.768 Å from each other. Fig. 2 is a projection, on to [010], showing the distribution of Mg in the structure of $SrMgP_2O_7$. The asymmetric unit contains two crystallographically independent phosphorus centers tetrahedrally coordinated; the two PO_4 share O1 to form the P_2O_7 group (Fig. 3). Average bridging and terminal P—O distances are 1.601 and 1.516 Å. A bridging angle φ(P,O,P) = 129.8 (2)° is observed for P_2O_7. All these values are quite usual in the series $SrMP_2O_7$ [d(P—O) and angle φ(P,O,P): $SrCrP_2O_7$ (1.600 Å, 128.1°), $SrNiP_2O_7$ (1.597 Å, 128.3°)]. The strontium ion is surrounded by eight O atoms. Mean distance Sr—O in the SrO_8 polyhedra is 2.641 Å, which is close to 2.622 Å found in $SrNiP_2O_7$.

Experimental

A powder has been prepared, according to the chemical formula $SrMgP_2O_7$, starting from equimolar mixtures of the starting materials $SrCO_3$, $MgCO_3$ and $(NH_4)_2HPO_4$. The mixture was heated progressively in air at increasing temperatures up to 1173 K. An X-ray diagram of the resulting powder shows it is isotypic to the homologous $SrMP_2O_7$ pyrophosphates. However, direct melting does not seem to provide crystals of sufficient quality for X-ray data collection. Thus, we have tried the following method: *ca* 200 mg powder of $SrMgP_2O_7$ was mixed with 100 mg iodine and 10 mg red phosphorus and the mixture sealed in an evacuated silica ampoule (l ~ 10 cm and d ~ 1.6 cm). The ampoule was transferred to a tubular furnace where it was heated for 5 d at 1073 K. The reaction products were washed with dilute NaOH and water and dried at 373 K. This resulted in small prismatic crystals from which we have selected the one used for the present crystal structure determination.

Crystal data

$SrMgP_2O_7$	D_x = 3.394 Mg m^{-3}
M_r = 285.87	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1188 reflections
a = 5.3046 (8) Å	θ = 5.9–57.1°
b = 8.3053 (13) Å	μ = 10.30 mm^{-1}
c = 12.700 (2) Å	T = 293 (2) K
β = 90.502 (3)°	Prism, colorless
V = 559.48 (15) Å^3	0.06 × 0.05 × 0.04 mm
Z = 4	

Data collection

Bruker CCD area-detector diffractometer	1798 independent reflections
ω scans	1317 reflections with $I > 2\alpha(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.069
T_{min} = 0.604, T_{max} = 0.662	θ_{max} = 31.7°
7156 measured reflections	$h = -7 \rightarrow 7$
	$k = -12 \rightarrow 12$
	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.071	$\Delta\rho_{max}$ = 0.82 e Å^{-3}
S = 0.95	$\Delta\rho_{min}$ = −0.77 e Å^{-3}
1798 reflections	Extinction correction: *SHELXL*97
101 parameters	Extinction coefficient: 0.0029 (6)

Table 1
Selected geometric parameters (Å, °).

Sr1—O3^{i}	2.525 (3)	Mg1—O7^{vi}	2.062 (3)
Sr1—O3^{ii}	2.537 (3)	Mg1—O4	2.116 (3)
Sr1—O4	2.565 (3)	P1—O2	1.501 (3)
Sr1—O6^{ii}	2.613 (3)	P1—O3	1.512 (3)
Sr1—O2^{iii}	2.665 (3)	P1—O4	1.519 (3)
Sr1—O7	2.714 (3)	P1—O1	1.596 (3)
Sr1—O4^{iii}	2.743 (3)	P2—O5	1.516 (3)
Sr1—O5^{iv}	2.766 (3)	P2—O7	1.520 (3)
Mg1—O2^{i}	1.986 (3)	P2—O6	1.527 (3)
Mg1—O5^{iv}	2.027 (3)	P2—O1	1.605 (3)
Mg1—O6^{v}	2.038 (3)		
O2—P1—O1—P2	156.4 (2)	O3—P1—P2—O6	−37.33 (16)
O5—P2—O1—P1	156.3 (2)	O4—P1—P2—O7	−37.85 (15)
O2—P1—P2—O5	−58.6 (2)		

Symmetry codes: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $x-1, y, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (vi) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 1999) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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