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# **Sr<sub>3</sub>MgPtO<sub>6</sub>**

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Single crystals of the mixed alkaline earth platinate, tristrontium magnesium platinum hexaoxide, Sr<sub>3</sub>MgPtO<sub>6</sub>, were grown from a KOH flux at 1273 K. The compound adopts the rhombohedral K<sub>4</sub>CdCl<sub>6</sub> structure type, featuring chains of face-shared, distorted MgO<sub>6</sub> trigonal prisms (Mg site symmetry 32) and PtO<sub>6</sub> octahedra (Pt site symmetry  $\bar{3}$ ) surrounded by columns of Sr<sup>2+</sup> ions (Sr site symmetry 2).

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**Key indicators**

Single-crystal X-ray study

T = 293 K

Mean  $\sigma(\text{Mg}-\text{O}) = 0.004 \text{ \AA}$ 

R factor = 0.024

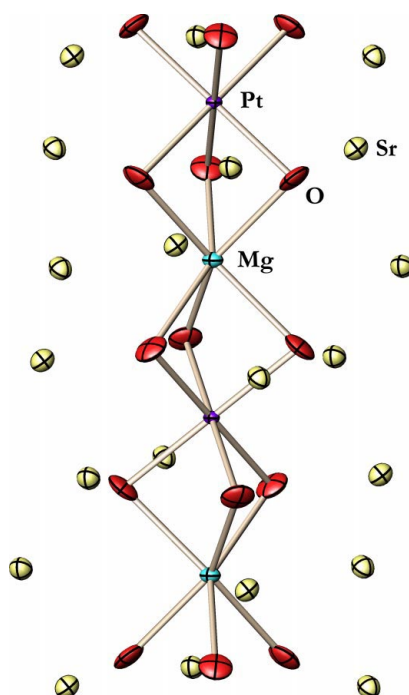
wR factor = 0.049

Data-to-parameter ratio = 24.5

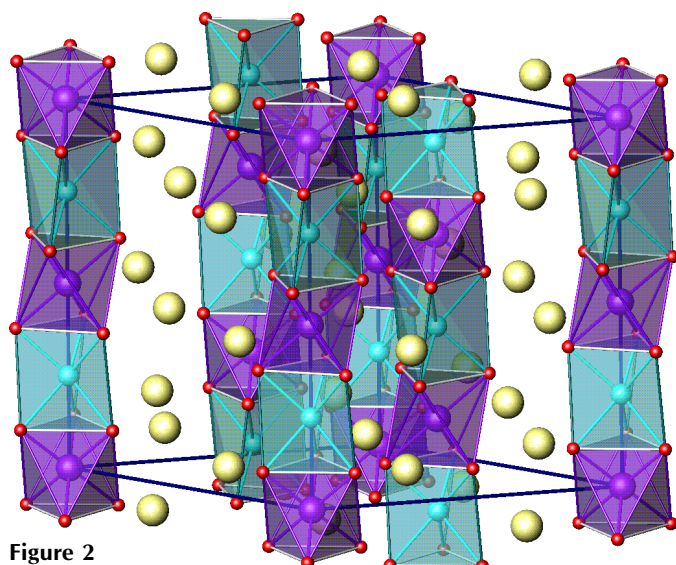
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

**Comment**

The structure of Sr<sub>3</sub>MgPtO<sub>6</sub> was determined in 1997 (Núñez *et al.*, 1997) by powder X-ray diffraction on a polycrystalline sample prepared by conventional sintering techniques, and was shown to adopt the K<sub>4</sub>CdCl<sub>6</sub> structure type (Bergerhoff & Schmitz-Dumont, 1956). This structure type features two crystallographically and chemically distinct K<sup>+</sup> positions and consists of chains along [001] of face-shared, distorted KCl<sub>6</sub> trigonal prisms and CdCl<sub>6</sub> octahedra. The polyhedral chains are surrounded by spiral columns of K<sup>+</sup> ions. To date, a large and compositionally diverse group of oxides adopting this structure type has been reported, typically as polycrystalline materials [reviewed in Stitzer *et al.* (2001)]. High-temperature flux growth from molten KOH has proven to be an effective oxide crystal growth medium. Single crystals of Sr<sub>3</sub>MgPtO<sub>6</sub> were readily grown from molten KOH at 1273 K, using

**Figure 1**

Fragment of the face-shared polyhedral chains in Sr<sub>3</sub>MgPtO<sub>6</sub>. Displacement ellipsoids are drawn at the 90% probability level.



**Figure 2**  
Polyhedral view of the unit cell of  $\text{Sr}_3\text{MgPtO}_6$ , viewed approximately along  $[110]$ .

$(\text{NH}_4)_2\text{PtCl}_6$  as the platinum source.  $\text{Sr}_3\text{MgPtO}_6$  represents an Mg-substituted form of the  $\text{K}_4\text{CdCl}_6$ -type oxide  $\text{Sr}_4\text{PtO}_6$  (Randall & Katz, 1959), with Mg ordered in the trigonal prism site (site-symmetry 32, Wyckoff symbol 6a) and  $\text{Pt}^{4+}$  in a rhombohedrally elongated octahedral site (site symmetry  $\bar{3}$ , Wyckoff symbol 6b). Fig. 1 illustrates the local coordination of these metal centers. The  $\text{Sr}^{2+}$  ion resides in an irregular eight-coordinate site (Wyckoff symbol 18e) of site symmetry 2. Fig. 2 shows a view of the polyhedral chains and  $\text{Sr}^{2+}$  columns. Bond lengths and angles from the present single-crystal determination of  $\text{Sr}_3\text{MgPtO}_6$  are very close to those derived from powder data [ $\text{Mg}-\text{O} = 2.172(16) \text{ \AA}$ ,  $\text{Pt}-\text{O} = 2.011(16) \text{ \AA}$  and  $\text{Sr}-\text{O} = 2.498(17)-2.742(17) \text{ \AA}$ ]. Refinement of the site occupancies for Mg and Pt showed no significant deviation from whole occupancy, indicating a stoichiometric compound, and no Sr/Mg mixing on the trigonal prism site.

## Experimental

The  $(\text{NH}_4)_2\text{PtCl}_6$  precursor was prepared according to a published method (Kaufman, 1967). Subsequently,  $\text{SrCO}_3$  (Alfa, 99.95%),  $\text{MgCO}_3$  (Alfa, 99.8%), and  $(\text{NH}_4)_2\text{PtCl}_6$  (stoichiometric amounts, ca 1 g total reagent mass) and KOH (Fisher, reagent grade; ~10 times by mass the total reagent amount) were loaded into a covered alumina crucible. The mixture was heated at 1273 K for 2 h, cooled to 1023 K at a rate of  $1 \text{ K h}^{-1}$ , at which point the furnace was shut off and allowed to cool to room temperature radiatively. The KOH matrix was dissolved with distilled water, leaving plentiful transparent brown crystals with a rhombohedral habit.

### Crystal data

$\text{Sr}_3\text{MgPtO}_6$	Mo $K\alpha$ radiation
$M_r = 578.26$	Cell parameters from 1132 reflections
Trigonal, $R\bar{3}c$	$\theta = 4.2-36.3^\circ$
$a = 9.6432(4) \text{ \AA}$	$\mu = 50.13 \text{ mm}^{-1}$
$c = 11.1112(6) \text{ \AA}$	$T = 293(2) \text{ K}$
$V = 894.82(7) \text{ \AA}^3$	Rhombohedral, brown
$Z = 6$	$0.11 \times 0.05 \times 0.04 \text{ mm}$
$D_x = 6.439 \text{ Mg m}^{-3}$	

### Data collection

Bruker SMART APEX CCD diffractometer	490 independent reflections
$\omega$ scans	431 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$R_{\text{int}} = 0.037$
$T_{\text{min}} = 0.073$ , $T_{\text{max}} = 0.239$	$\theta_{\text{max}} = 36.3^\circ$
2412 measured reflections	$h = -16 \rightarrow 7$
	$k = -11 \rightarrow 16$
	$l = -18 \rightarrow 8$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0227P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.024$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.049$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 2.32 \text{ e \AA}^{-3}$
490 reflections	$\Delta\rho_{\text{min}} = -3.12 \text{ e \AA}^{-3}$
20 parameters	Extinction correction: <i>SHELXL97</i>
	Extinction coefficient: 0.00118 (8)

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ).

$\text{Sr}-\text{O}$	2.472 (3)	$\text{Mg}-\text{O}$	2.177 (3)
$\text{Sr}-\text{O}^{\text{i}}$	2.637 (3)	$\text{Mg}-\text{Pt}$	2.77780 (15)
$\text{Sr}-\text{O}^{\text{ii}}$	2.663 (3)	$\text{Pt}-\text{O}$	2.031 (3)
$\text{Sr}-\text{O}^{\text{iii}}$	2.731 (3)		

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

Systematic absences in the dataset confirmed a  $c$ -glide operation, indicating the space groups  $R3c$  and  $R\bar{3}c$ . Preliminary powder X-ray diffraction showed the compound to be isostructural with  $\text{K}_4\text{CdCl}_6$  (space group  $R\bar{3}c$ ); therefore, the expected centrosymmetric space group was chosen and confirmed by the structure solution. The largest difference peak and hole were located less than  $0.8 \text{ \AA}$  from the Pt atom.

Data collection: *SMART-NT* (Bruker, 1999); cell refinement: *SAINT-Plus-NT* (Bruker, 1999); data reduction: *SAINT-Plus-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 2001); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

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## References

- Bergerhoff, G. & Schmitz-Dumont, O. (1956). *Z. Anorg. Allg. Chem.* **284**, 10–19.
- Bruker (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1999). *SMART-NT* (Version 5.611), *SAINT-Plus-NT* (Version 6.02a) and *SADABS* (Version 1.0). Bruker AXS Inc., Madison, Wisconsin, USA.
- Dowty, E. (2001). *ATOMS for Windows*. Version 5.1. Shape Software, 521 Hidden Valley Road, Kingsport, TN 37663, USA.
- Kaufman, G. S. (1967). In *Inorganic Syntheses*, Vol. 9, edited by S. Y. Tyree Jr, pp. 182–185. New York: McGraw-Hill.
- Núñez, P., Trail, S. & zur Loye, H.-C. (1997). *J. Solid State Chem.* **130**, 35–41.
- Randall, J. J. Jr & Katz, L. (1959). *Acta Cryst.* **12**, 519–521.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Stitzer, K. E., Darriet, J. & zur Loye, H.-C. (2001). *Curr. Op. Solid State Mater. Sci.* **5**, 535–544.