

Rietveld refinement of $\text{Sr}_5(\text{AsO}_4)_3\text{Cl}$ from high-resolution synchrotron dataAnthony M. T. Bell,^{a*} C. Michael B. Henderson,^b Richard F. Wendlandt^c and Wendy J. Harrison^c^aSynchrotron Radiation Source, STFC Daresbury Laboratory, Daresbury, Warrington, Cheshire WA4 4AD, England, ^bSchool of Earth, Atmospheric and Environmental Sciences, University of Manchester, Manchester, M13 9PL, England, and^cDepartment of Geology and Geological Engineering, Colorado School of Mines, Golden, CO 80401, USA

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Key indicators: powder synchrotron study; $T = 298\text{ K}$; mean $\sigma(\text{As}-\text{O}) = 0.020\text{ \AA}$; R factor = 0.052; wR factor = 0.066; data-to-parameter ratio = 14.1.

The apatite-type compound, pentastrontrium tris[arsenate(V)] chloride, $\text{Sr}_5(\text{AsO}_4)_3\text{Cl}$, has been synthesized by ion exchange at high temperature from a synthetic sample of mimetite [$\text{Pb}_5(\text{AsO}_4)_3\text{Cl}$] with SrCO_3 as a by-product. The results of the Rietveld refinement, based on high resolution synchrotron X-ray powder diffraction data, show that the title compound crystallizes in the same structure as other halogenoapatites with general formula $A_5(\text{YO}_4)_3X$ (A = divalent cation, Y = pentavalent cation, and X = F, Cl or Br) in the space group $P6_3/m$. The structure consists of isolated tetrahedral AsO_4^{3-} anions (the As atom and two O atoms have m symmetry), separated by two crystallographically independent Sr^{2+} cations that are located on mirror planes and threefold rotation axes, respectively. One Sr atom is coordinated by nine O atoms and the other by six. The chloride anions (site symmetry $\bar{3}$) are at the $2a$ sites and are located in the channels of the structure.

Related literature

For crystal chemistry of apatites, see: Mercier *et al.* (2005); White & ZhiLi (2003); Wu *et al.* (2003). For powder diffraction data on Sr As-apatite, see: Kreidler & Hummel (1970). Atomic coordinates as starting parameters for the Rietveld (Rietveld, 1969) refinement of the present phases were taken from Bell *et al.* (2008); Dai *et al.* (1991); de Villiers *et al.* (1971). For related Sr–Cl-apatites, see: Đorđević *et al.* (2008); Sudarsanan & Young, (1974, 1980); Beck *et al.* (2006); Noetzold *et al.* (1995); Noetzold & Wulff (1996, 1997, 1998); Swafford & Holt (2002); Wardojo & Hwu (1996). For synthetic work, see: Baker (1966); Essington (1988); Harrison *et al.* (2002).

Experimental

Crystal data

$\text{Sr}_5(\text{AsO}_4)_3\text{Cl}$
 $M_r = 890.31$
 Hexagonal, $P6_3/m$
 $a = 10.1969$ (1) \AA
 $c = 7.28108$ (9) \AA
 $V = 655.63$ (2) \AA^3
 $Z = 2$
 Synchrotron radiation

$\lambda = 0.998043\text{ \AA}$
 $T = 298\text{ K}$
 Specimen shape: cylinder
 $40 \times 0.7 \times 0.7\text{ mm}$
 Specimen prepared at 100 kPa
 Specimen prepared at 1258 K
 Particle morphology: powder, white

Data collection

In-house design diffractometer
 Specimen mounting: capillary
 Specimen mounted in transmission mode

Scan method: step
 Absorption correction: fixed at 0
 $2\theta_{\min} = 2$, $2\theta_{\max} = 60^\circ$
 Increment in $2\theta = 0.01^\circ$

Refinement

$R_p = 0.052$
 $R_{wp} = 0.066$
 $R_{exp} = 0.047$
 $R_B = 0.090$
 $S = 2.00$
 Excluded region(s): $2-6^\circ 2\theta$

Profile function: Pseudo Voigt
 225 reflections
 16 parameters
 Preferred orientation correction: none

Table 1

Selected geometric parameters (\AA , $^\circ$).

Sr1–O1	2.49 (2)	Sr2–O1 ^v	3.02 (2)
Sr1–O2 ⁱ	2.59 (2)	Sr2–Cl1 ^{iv}	3.156 (3)
Sr1–O3 ⁱ	3.01 (1)	As1–O3	1.57 (1)
Sr2–O2 ⁱⁱ	2.53 (2)	As1–O1	1.72 (2)
Sr2–O3 ⁱⁱⁱ	2.44 (1)	As1–O2	1.70 (2)
Sr2–O3 ^{iv}	2.94 (1)		
O3–As1–O3 ^{vi}	121 (1)	O3–As1–O2	106.3 (6)
O3–As1–O1	105.8 (7)	O1–As1–O2	112 (1)

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

Data collection: local software; cell refinement: *CELREF* (Laugier & Bochu, 2003) and *GSAS* (Larson & Von Dreele (2004)); data reduction: local software; method used to solve structure: coordinates taken from a related compound; program(s) used to refine structure: *GSAS* and *EXPGUI* (Toby, 2001); molecular graphics: *VESTA* (Momma & Izumi, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2096).

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