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Rietveld refinement of $Ba_5(AsO_4)_3Cl$ from high-resolution synchrotron data

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Key indicators: powder synchrotron study; T = 298 K; mean σ(As–O) = 0.040 Å; R factor = 0.059; wR factor = 0.082; data-to-parameter ratio = 22.1.

The apatite-type compound $Ba_5(AsO_4)_3Cl$, pentabarium tris(arsenate(V)) chloride, has been synthesized by ion exchange at high temperature from a synthetic sample of mimetite ($Pb_5(AsO_4)_3Cl$) with $BaCO_3$ 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 $A_5(YO_4)_3X$ (A = divalent cation, Y = pentavalent cation, X = Cl, Br) in space group $P6_3/m$. The structure consists of isolated tetrahedral AsO_4^{3-} anions (m symmetry), separated by two crystallographically independent Ba^{2+} cations that are located on mirror planes and threefold rotation axes, respectively. The Cl^- anions are at the 2b sites ($\bar{3}$ symmetry) 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 Ba-containing As-apatites, see: Kreidler & Hummel (1970); Dunn & Rouse (1978). Atomic coordinates as starting parameters for the Rietveld (Rietveld, 1969) refinement of the present phases were taken from Chengjun *et al.* (2005); Dai *et al.* (1991); de Villiers *et al.* (1971). For related Ba—Cl-apatites, see: Đordevic *et al.* (2008); Hata *et al.* (1979); Reinen *et al.*(1986); Roh & Hong (2005); Schiff-Francois *et al.* (1979). For synthetic work, see: Baker (1966); Essington (1988); Harrison *et al.* (2002).

Experimental

Crystal data

$As_3Ba_5ClO_{12}$
M_r = 1138.85
Hexagonal, $P6_3/m$
a = 10.5570 (1) Å
c = 7.73912 (8) Å
V = 746.98 (1) Å^3
Z = 2
Synchrotron radiation
λ = 0.998043 Å
μ = 56.07 (1) mm^{-1}
T = 298 K
Specimen shape: cylinder
40 × 0.7 × 0.7 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: none
$2\theta_{min}$ = 2, $2\theta_{max}$ = 70°
Increment in 2θ = 0.01°

Refinement

R_p = 0.059
R_{wp} = 0.082
R_{exp} = 0.067
R_B = 0.090
S = 1.23
Excluded region(s): 2-6 degrees 2θ.
Profile function: Fundamental Parameters
464 Bragg reflections
21 parameters
Preferred orientation correction: none

Table 1
Selected geometric parameters (Å, °).

Ba1—O1	2.67 (5)	Ba2—O1^{v}	3.14 (4)
Ba1—O2^{i}	2.81 (4)	Ba2—Cl1^{iv}	3.281 (5)
Ba1—O3^{i}	3.12 (3)	As1—O3	1.64 (2)
Ba2—O2^{ii}	2.59 (4)	As1—O1	1.70 (8)
Ba2—O3^{iii}	2.62 (4)	As1—O2	1.70 (4)
Ba2—O3^{iv}	3.05 (4)		
O3—As1—O3^{vi}	118 (2)	O3—As1—O2	108 (2)
O3—As1—O1	108 (1)	O1—As1—O2	106 (2)

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); data reduction: local software; method used to solve structure: coordinates taken from a related compound; program(s) used to refine structure: *TOPAS* (Coelho, 2000); molecular graphics: *Balls and Sticks* (Kang & Ozawa, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2188).

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