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β-Sm(OH)$_2$Cl: a new lamellar variety

Cyril Jouve, Jerome Marrot and Didier Riou*

Institut Lavoisier, IREM, UMR CNRS C 8637, Université de Versailles Saint-Quentin-en-Yvelines, 45 Avenue des Etats Unis, 78035 Versailles CEDEX, France
Correspondence e-mail: riou@chimie.uvsq.fr

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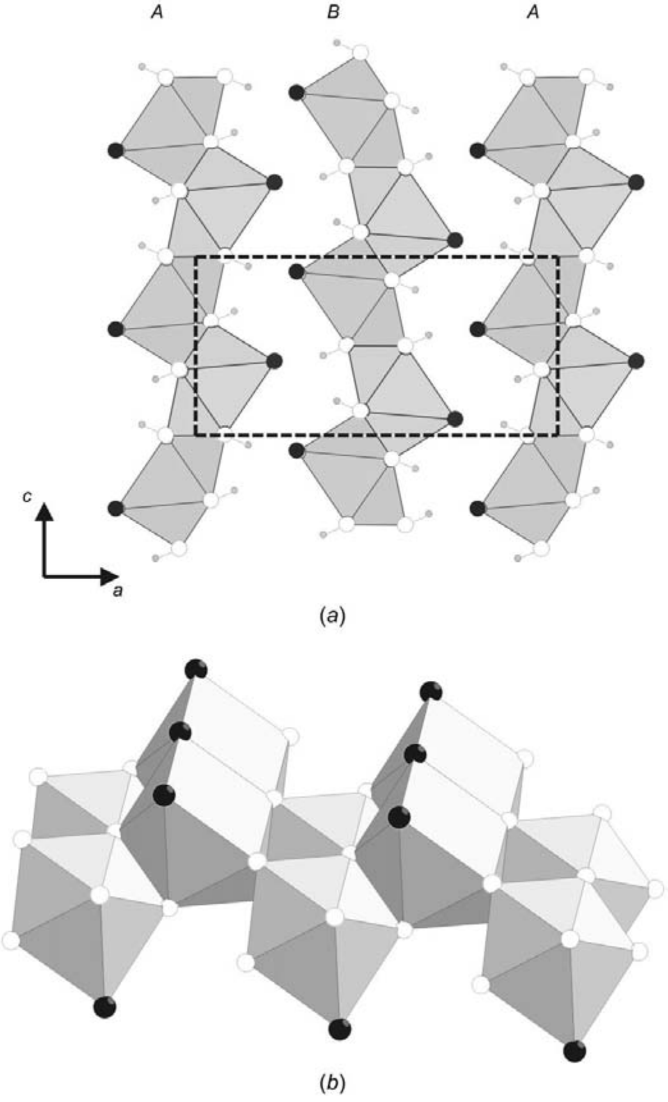
Crystals of dihydroxysamarium chloride were synthesized hydrothermally at 473 K. The orthorhombic structure was determined by single-crystal X-ray diffraction analysis. β-Sm(OH)$_2$Cl exhibits a lamellar structure built up from the stacking of neutral slabs; all the atoms lie on crystallographic mirror planes. The structure is stabilized by strong hydrogen bonds between the OH groups and the Cl ions of adjacent layers.

Comment

Research into the field of compounds with a microporous framework has been dramatically enhanced by the use of a hydrothermal technique (Cheetham *et al.*, 1999). Inside this large family of compounds, the hybrids whose structures are built up from the iono-covalent association of inorganic and organic moieties show great porosity with, for example, metallocarboxylates (Li *et al.*, 1999) or metallophosphonates (Riou *et al.*, 2000).

In our work with metallosulfonates with open structures, we obtained a by-product which corresponds to a new polymorph of dihydroxysamarium chloride, denoted β-Sm(OH)$_2$Cl to distinguish it from the already reported form (Klevtsova & Glinskaya, 1969). β-Sm(OH)$_2$Cl is a bidimensional structure built up from the stacking along [100] of neutral layers (denoted *A* on Fig. 1*a*) in strict alternation with their equivalent (denoted *B*) layer from an *n*-mirror glide. Inside the layers, each Sm atom is eight-coordinated, with six Sm—O distances in the range 2.383 (5)–2.452 (4) Å and two longer Sm—Cl distances of 2.930 (2) Å (Table 1). The edge-connections of these polyhedra form corrugated layers (Fig. 1*b*) whose mean plane is parallel to (100).

The stabilization of the structure is ensured *via* hydrogen bonds between the hydroxy H atoms and the Cl ions from an adjacent layer. It is worth noting that two successive layers along [100] are arranged in such a way that the OH groups from one layer and the Cl ions of the other are face to face (Fig. 1*a*), resulting in short Cl$\cdots$O distances (<3.5 Å), implying strong hydrogen bonds which stabilize the structure (Table 2). In a first examination, the structure of the previously reported Sm(OH)$_2$Cl (Klevtsova & Glinskaya, 1969) looks very different from the title compound, (I), since it exhibits a three-dimensional framework with a larger density (4.991 *versus* 4.892 Mg m^{-3}). Nevertheless, some correlations between the two structure types are observed. The coordination around Sm is similar, with six Sm—O distances in the range 2.36 (2)–2.46 (1) Å and two longer Sm—Cl distances [3.176 (5) and 2.936 (5) Å]. Furthermore, the framework (Fig. 2) is built up from the connection *via* the OH groups of layers almost identical to the *B* layers shown in Fig. 1(*a*). In fact, just the relative location of the Cl ions in the Sm(OH)$_6$Cl$_2$ polyhedra differs. In (I), they are all located on the outer edges of the corrugated planes (Fig. 1*b*) and point alternatively to each side of the layers, whereas they constitute the internal edge-sharing of the similar planes in the other form. This difference prohibits any phase transition between the two forms without a complete rearrangement of the structure.



(*a*)
(*b*)

Figure 1
(*a*) A projection of β-Sm(OH)$_2$Cl along [010] showing the bidimensional feature of the structure (black and white circles are Cl and O atoms, respectively, and small circles are H atoms) and (*b*) a perspective view of one corrugated plane of β-Sm(OH)$_2$Cl.

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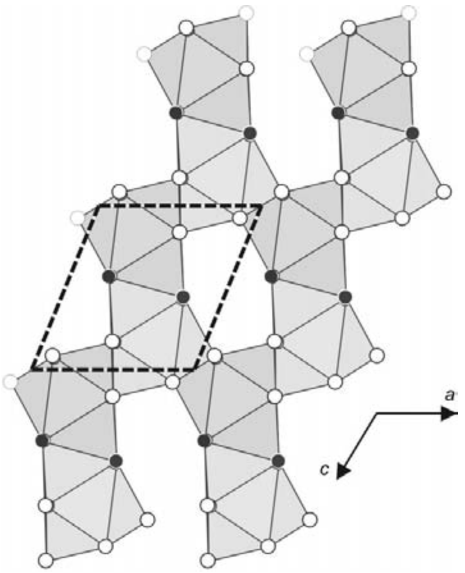

Figure 2
A projection of $Sm(OH)_2Cl$ (Klevtsova & Glinskaya, 1969) along [010] showing the three-dimensional feature of the structure (black and white circles are Cl and OH groups, respectively).

Experimental

The title compound, (I), was prepared from a mixture of samarium trichloride hexahydrate, ethylsulfonic acid and deionized water in the molar ratio 1:2:300. The initial pH was adjusted to 8 by the dropwise addition of a concentrated NaOH solution. The resulting mixture was sealed in a teflon-lined autoclave (Parr) then heated for two days at 473 K under autogenous pressure. After cooling to room temperature, the pH was measured as 4 and the solid was separated from the solution by filtration, washed with water and dried in air. β-$Sm(OH)_2Cl$ was obtained pure in the form of light-yellow rhombic platelets. Attempts to synthesize β-$Sm(OH)_2Cl$ without ethylsulfonic acid failed, leading instead to a mixture of the two varieties of $Sm(OH)_2Cl$. A single crystal suitable for X-ray diffraction analysis was chosen by optical microscopy and glued to a glass fiber.

Crystal data

β-$Sm(OH)_2Cl$
$M_r = 219.5$
Orthorhombic, *Pnma*
$a = 12.6014\ (14)$ Å
$b = 3.7706\ (4)$ Å
$c = 6.2740\ (7)$ Å
$V = 298.11\ (6)$ Å^3
$Z = 4$
$D_x = 4.892$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1259 reflections
$\theta = 3.2$–$29.9°$
$\mu = 20.32$ mm^{-1}
$T = 296\ (2)$ K
Rhombohedral, light yellow
$0.04 \times 0.04 \times 0.04$ mm

Data collection

Bruker CCD area-detector diffractometer
ω scans
Absorption correction: semi-empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.497$, $T_{max} = 0.497$
1935 measured reflections
454 independent reflections
347 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 29.9°$
$h = -15 \rightarrow 17$
$k = -4 \rightarrow 5$
$l = -3 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.060$
$S = 1.12$
454 reflections
30 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.13$ e Å^{-3}
$\Delta\rho_{min} = -1.11$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0034 (8)

Table 1
Selected interatomic distances (Å).

Sm—O1^{i}	2.383 (5)	Sm—O1	2.452 (4)
Sm—O2	2.416 (3)	Sm—O1^{ii}	2.452 (4)
Sm—O2^{ii}	2.416 (3)	Sm—Cl	2.9301 (15)
Sm—O2^{iii}	2.430 (5)	Sm—Cl^{iv}	2.9301 (15)

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $x, 1+y, z$; (iii) $-x, 1-y, 1-z$; (iv) $x, y-1, z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$Cl^{i}	0.90 (2)	2.54 (2)	3.193 (4)	129.7 (18)
O2—H2$\cdots$Cl^{ii}	0.89 (2)	2.58 (3)	3.461 (5)	170 (8)

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

The data set is complete to 98.2% at $\theta = 28.96°$. The OH groups were defined both from electroneutrality and bond-valence calculations (O'Keeffe & Brese, 1992). H atoms were refined with constraints on the O—H distances.

Data collection: *SMART* (Bruker, 1994); cell refinement: *SMART*; data reduction: *SMART*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1996); software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1144). Services for accessing these data are described at the back of the journal.

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