

Samarium(III) carbonate hydroxide

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{O}-\text{C}) = 0.007$ Å
 R factor = 0.047
 wR factor = 0.122
Data-to-parameter ratio = 10.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

$\text{Sm}(\text{CO}_3)(\text{OH})$ was obtained hydrothermally from an aqueous solution of samarium oxide, formic acid, triethylenetetramine and dimethylformamide. The structure features deca-coordinated samarium (site symmetry m) with carbonate and hydroxide acting as bridging ligands.

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Comment

Over recent decades, the synthesis of three-dimensional frameworks with new topological structures has received great attention due to their functional applications in catalysis, adsorption, ion-exchange and radioactive waste remediation. As the building elements of open-frameworks, both silicon and germanium have been chosen to synthesize new open frameworks (Li *et al.*, 1998; Lin *et al.*, 2003; Plévert *et al.*, 2001; Xu Fan, Chino *et al.*, 2004; Xu, Fan, Elangovan *et al.*, 2004). In the last few years, an important advance in porous materials has been achieved by the study of transition metal carbonates. Diamond-type $\text{Na}_2\text{Zn}_3(\text{CO}_3)_4 \cdot 3\text{H}_2\text{O}$ was reported by Gier *et al.* (1996), while face-centred (FC) cubic $[\text{Zn}_6(\text{CO}_3)_{12}(\text{CH}_6\text{N}_3)_8\text{Na}_3[\text{N}(\text{CH}_3)_4] \cdot 2\text{H}_2\text{O}]$ was synthesized by Abrahams

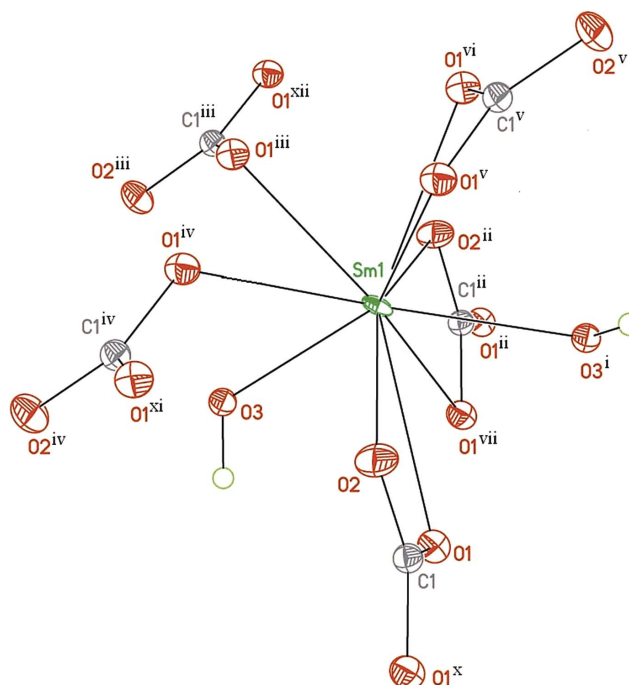


Figure 1

The Sm coordination in the title compound. Displacement ellipsoids are drawn at the 70% probability level. Only one position is shown for the disordered H atoms. [Symmetry codes: (i) $x - \frac{1}{2}, y, -z + \frac{3}{2}$; (ii) $x, y + 1, z$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x + \frac{1}{2}, y, -z + \frac{3}{2}$; (v) $-x + \frac{1}{2}, -y, z - \frac{1}{2}$; (vi) $-x + \frac{1}{2}, y + \frac{1}{2}, z - \frac{1}{2}$; (vii) $x, -y + \frac{1}{2}, z$; (x) $x, -y - \frac{1}{2}, z$; (xi) $x + \frac{1}{2}, -y - \frac{1}{2}, -z + \frac{3}{2}$; (xii) $x + \frac{1}{2}, y + 1, -z + \frac{3}{2}$.]

et al. (2004). In this work, we have designed and synthesized the title compound, which features a three-dimensional framework.

The building unit of title compound is constructed from SmO_{10} polyhedra and carbonates (Fig. 1). The asymmetric unit contains one Sm^{3+} , one CO_3^{2-} group and one hydroxide group, all of which lie on positions with site symmetry m (Wyckoff position 4c). The coordination about Sm is achieved by five bridging carbonate ions, three of them acting as bidentate ligands with respect to the central Sm, and two acting as monodentate ligands. The coordination is completed by two hydroxide ions, which act as bridging ligands between two Sm^{3+} .

The Sm atom has typical geometrical parameters, with Sm—O distances of 2.315 (3)–2.751 (4) Å (Table 1). The bond distances and angles are in agreement with those found in reported rare-earth compounds (Zhang *et al.*, 2004; Yuan *et al.*, 2004). The geometry of the carbonate ions is unexceptional. Fig. 2 shows the three-dimensional arrangement in the unit cell, displaying the way the Sm ions are connected by bridging ligands.

Experimental

Yellow block-shaped crystals were synthesized hydrothermally from a mixture of Sm_2O_3 , HCO_2H , H_2O , triethylenetetramine and dimethylformamide. In a typical synthesis, Sm_2O_3 (0.15 g) was dissolved in a mixture of dimethylformamide (1.13 g) and water (10.0 g) followed by the addition of HCO_2H (0.55 g) and triethylenetetramine (0.23 g) with constant stirring. Finally, the mixture was kept in a 25 ml Teflon-lined steel autoclave at 453 K for 10 d. The autoclave was slowly cooled to room temperature; the product was then filtered off, washed with distilled water and dried at room temperature. Yellow block-shaped crystals of the title compound were obtained.

Crystal data

$\text{Sm}(\text{CO}_3)(\text{OH})$	$Z = 4$
$M_r = 227.37$	$D_x = 5.052 \text{ Mg m}^{-3}$
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
$a = 7.161$ (6) Å	$\mu = 19.46 \text{ mm}^{-1}$
$b = 4.929$ (4) Å	$T = 293$ (2) K
$c = 8.470$ (7) Å	Block, yellow
$V = 299.0$ (4) Å ³	$0.06 \times 0.05 \times 0.05 \text{ mm}$

Data collection

Bruker APEX-2 CCD diffractometer	1471 measured reflections
ω scans	402 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	374 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.388$, $T_{\max} = 0.443$ (expected range = 0.331–0.378)	$R_{\text{int}} = 0.060$
	$\theta_{\text{max}} = 28.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0822P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.047$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta\sigma)_{\text{max}} = 0.023$
$S = 1.17$	$\Delta\rho_{\text{max}} = 3.90 \text{ e \AA}^{-3}$
402 reflections	$\Delta\rho_{\text{min}} = -3.98 \text{ e \AA}^{-3}$
39 parameters	Extinction correction: SHELXL97
All H-atom parameters refined	Extinction coefficient: 0.0121 (10)

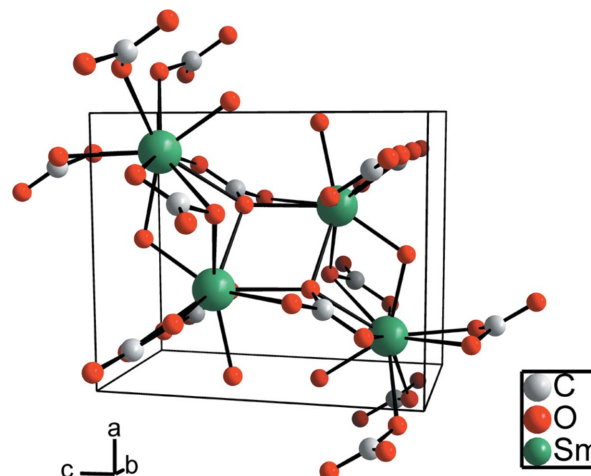


Figure 2
The packing of $\text{Sm}(\text{CO}_3)(\text{OH})$.

Table 1

Selected bond lengths (Å).

Sm1—O3	2.315 (3)	Sm1—O1 ⁱⁱ	2.555 (3)
Sm1—O3 ⁱ	2.340 (4)	Sm1—O1 ⁱⁱⁱ	2.593 (4)
Sm1—O2	2.520 (2)	Sm1—O1	2.751 (4)

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

The highest peak in the difference map is 0.85 (2) Å from Sm1, while the deepest hole is 0.79 (2) Å from Sm1. The H atom was located in a difference Fourier map. Since its site symmetry (1) was different from that of the carrier atom O3 (m), it was refined using a split model with an occupancy of 0.5 for both positions. The O—H distance is 0.880 (3) Å.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997b); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997b); molecular graphics: SHELXTL (Sheldrick, 1997a); software used to prepare material for publication: SHELXTL.

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