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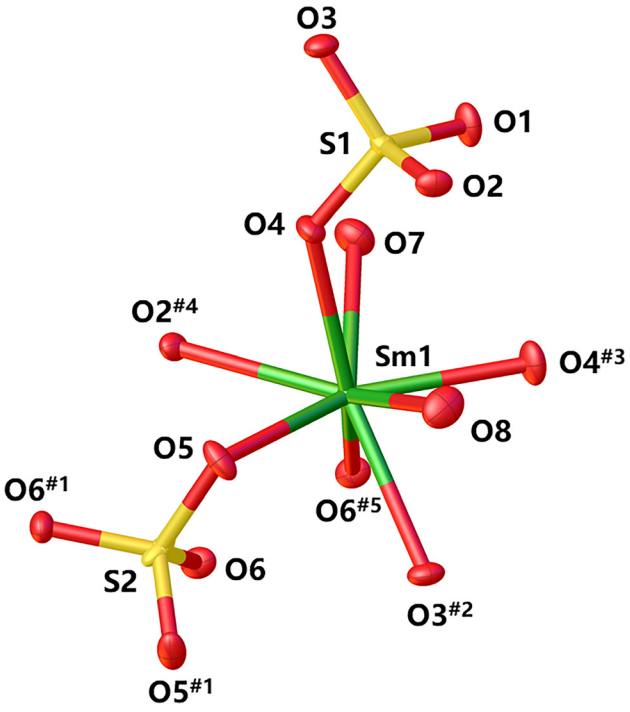
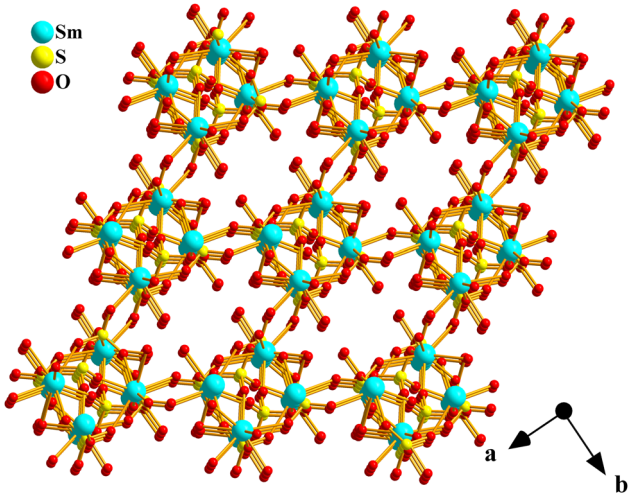
The crystal structure of samarium sulfate pentahydrate, $Sm_2(SO_4)_3(H_2O)_5$





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Abstract

$Sm_2(SO_4)_3(H_2O)_5$, monoclinic, $C2/c$ (no. 15), a = 15.653(3) Å, b = 9.5369(19) Å, c = 10.199(2) Å, β = 120.36(3)°, V = 1313.9(6) Å^3, Z = 1, $R_{gt}(F)$ = 0.0281, $wR_{ref}(F^2)$ = 0.0664, T = 293 K.

CCDC no.: 2303100

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.38 × 0.28 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	9.42 mm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω
θ_{max}, completeness:	27.5°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6284, 1492, 0.080
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1413
$N(param)_{refined}$:	118
Programs:	SHELX [1, 2]

1 Source of materials

All reagents are commercially available A. R. grade reagents and are used without further purification: 0.176 g (0.5 mmol) Sm_2O_3 was added to 2.0 mL of ethylene glycol and 2.0 mL of deionized water solution under stirring, 1.5 mL of 1 M H_2SO_4 was added dropwise to adjust pH 1.5. The resulting mixture was placed in a 24 mL Teflon-lined steel vessel and heated at 170 °C for 4 days. The autoclave is slowly cooled to room temperature, then the product is filtered and dried in the air for day to obtain colorless block crystals.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sm1	0.11951 (2)	0.85634 (2)	0.06717 (2)	0.00974 (14)
S1	0.36140 (8)	0.71166 (11)	0.27428 (12)	0.0105 (2)
S2	0.000000	0.88092 (17)	0.250000	0.0108 (3)
O1	0.2832 (2)	0.8012 (4)	0.2703 (3)	0.0171 (7)
O2	0.3363 (2)	0.5636 (3)	0.2776 (4)	0.0154 (7)
O3	0.4565 (2)	0.7479 (3)	0.4077 (4)	0.0169 (7)
O4	0.3655 (3)	0.7373 (4)	0.1350 (4)	0.0202 (7)
O5	−0.0842 (3)	0.7953 (4)	0.2278 (4)	0.0197 (7)
O6	−0.0273 (2)	0.9686 (4)	0.1158 (3)	0.0164 (7)
O7	0.2460 (3)	1.0092 (4)	0.0459 (4)	0.0250 (8)
H7A	0.294 (3)	0.974 (6)	0.045 (7)	0.037*
H7B	0.234 (4)	1.084 (4)	0.000 (7)	0.037*
O8	0.1254 (3)	0.5981 (4)	0.0973 (4)	0.0259 (8)
H8A	0.169 (4)	0.555 (6)	0.171 (4)	0.039*
H8B	0.105 (4)	0.549 (6)	0.022 (4)	0.039*
O9[aa]	0.0102 (8)	0.4903 (10)	−0.1965 (10)	0.046 (2)
H9	−0.047610	0.451250	−0.257680	0.069*

[a]Occupancy: 0.5

2 Experimental details

The structure was solved by Direct Methods with the SHELXS and further refined with the SHELXL program. The oxygen-bound H atoms were located on a difference Fourier map and refined with distances O–H = 0.85 Å and (U_{iso}(H) = 1.5U_{eq}(O)) (Tables 1 and 2).

3 Comment

Over the years, efforts have been made to synthesize a variety of one-, two- and three-dimensional inorganic materials [3, 4]. Solvothermal techniques have been used to synthesize various inorganic framework compounds containing anionic components, such as phosphates [5], silicates [6], selenites [7] and sulfates [8, 9]. Among these compounds that have been synthesized and characterized, compounds involving sulfates are a very important family [10, 11]. Compared with the relatively fixed coordination it is easy to form stable three-dimensional structures, intricate topological structures and types [12–14]. In addition, lanthanide compounds have become an important class of luminescent materials due to the unique photophysical properties of lanthanide centers, such as long lifetime, large Stokes shift, high quantum yield and linear emission bands generated by 4f–4f transitions [15, 16]. At present, many lanthanide sulfates have been reported in the literatures, such as $Sm_2(SO_4)(H_2O)_8$ [17], $La_2(SO_4)_3(H_2O)_9$ [18], $Eu_2(SO_4)_3(H_2O)_8$ [19], but the three-dimensional inorganic framework for lanthanide sulfates has been relatively limited.

The title compound is isomorphous with $Nd_2(SO_4)_3(H_2O)_5$ [20]. The space group of the title compound is *C*2/*c*. As shown in the Figure (left part), the Sm atom is coordinated by eight oxygen atoms in a dodecahedral configuration, in which six O atoms (O1, O2, O3, O4, O5, O6) are derived from five sulfate radical groups, and two O atoms (O7, O8) are derived from two coordinating water molecules. The long distances of the Sm–O bond lengths are 2.365(3)–2.554(4) Å, and the corresponding bond angles of the formed O–Sm–O bond are 52.4(1)°–150.2(1)°. The two sulfate groups have tetrahedral coordination, and the S–O bond distances are 1.463(3)–112.4(3) Å, corresponding to S–O–S bond angles of 106.0(2)° to 112.4(3)°. The two sulfate groups (S1 and S2 groups) are both in the μ_4 coordination mode. The skeleton structure of the compound is formed by the adjacent SmO_8 and SO_4 polyhedra sharing a single corner or edge. The oxygens of the sulfate groups corresponding to S2 connect four Sm atoms forming one-dimensional chains along [001] direction. The SO_4 groups corresponding to S1 connect three Sm atoms interspersed in the above chains. The adjacent chains are further connected by O1 (for S1 groups) forming the three-dimensional framework (Figure, right part).

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