Crystal structure of hexaqua-(μ₃-methylenedisulphonato)-bis(μ₂-methylenedisulphonato)disamarium(III), Sm₂(H₂O)₆(CH₂O₆S₂)₃

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Received April 29, 2009, accepted and available on-line May 6, 2009; CCDC no. 1267/2643

Abstract

C₃H₁₄O₆S₆Sm₂, triclinic, P1̅ (no. 2), a = 9.0017(3) Å, b = 9.2768(4) Å, c = 15.8674(6) Å, α = 74.864(1)°, β = 78.263(1)°, γ = 61.138(1)°, V = 1115.5 Å³, Z = 2, R_{w}^{2}(F) = 0.026, wR_{ref}^{2}(F²) = 0.067, T = 173 K.

Source of material

A mixture of Sm₂O₃ (0.05 mmol), methylenedisulfonic acid (0.16 mmol) and H₂O (10 mL) was sealed in a 25 mL Teflon-lined stainless steel autoclave and heated at 140 °C for 2 days. After the mixture was cooled to room temperature, it was filtrated and the filtrate was slowly evaporated, yielding light yellow crystals of the title compound (83 % based on Sm).

Discussion

Due to the weak coordination strength of monosulfonate ions, most metal complexes of these ligands obtained from aqueous solution are water-coordinated metal sulfonate salts. The coordination chemistry of the sulfonate ion has been less well investigated in comparison with other organic acidato anions such as carbonates and phosphonates [1]. However, by employing disulfonates, which can provide multiple potentially chelating coordination sites, stable networks sustained by sulfonate-metal interactions can be obtained with various dimensionalities [2,3]. The asymmetric unit of the title crystal structure consists of two samarium ions, six coordinating water molecules and three CH₂(SO₃)₂⁻ ligands. Both Sm⁺⁺⁺ are nine-coordinated. Each Sm⁺⁺⁺ is surrounded by six sulfonate O atoms (O₆) and the remaining coordination positions are occupied by coordinating water molecules (O₆w). The bond lengths of Sm—O₆ and Sm—O₆w fall in the range of 2.386(3) - 2.706(4) Å and 2.370(4) - 2.492(3) Å. A CH₂(SO₃)₂⁻ ligand chelates two Sm ions simultaneously by using four of its sulfonate oxygens (O₇, O₈, O₁₀ and O₁₁) into a “paddle-wheel” dimer with an inversion center lying in the middle, and the Sm⋯Sm distance is 5.348(4) Å. Sm₂ ions are chelated and bridged through O₁₃—S₅—O₁₄ and O₁₆—S₆—O₁₈ connectivities into a zigzag chain. The rest CH₂(SO₃)₂⁻ ligand joins the dimer and the chain through two O—S—O bridges (O₁—S₁—O₂ and O₄—S₂—O₅), thus forming a two-dimensional sheet. The three-dimensional structure is stabilized by intricate interlayer hydrogen bonding.

Table 1. Data collection and handling.

| Crystal: | light yellow block, size 0.20 x 0.20 x 0.25 mm |
| Wavelength: | Mo Kα radiation (0.71073 Å) |
| μ: | 58.84 cm⁻¹ |
| Diffractometer, scan mode: | Bruker SMART CCD, φ/ω |
| 2θ_max: | 51° |
| hkl_restricted, N(hkl)unique: | 13394, 4085 |
| Criterion for Iobs, N(hkl): | I_obs > 2σ(I_obs), 3557 |
| N(param): | 316 |
| Programs: | SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6] |

Table 2. Atomic coordinates and displacement parameters (in Å²).

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* Correspondence author (e-mail: overloadzz@hotmail.com)
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References