

Zhi-Wei Xu,^a Jun-Ying Yang,^a
Yun-Long Fu^a and Seik Weng
Ng^{b*}^aSchool of Chemistry and Materials Science,
Shanxi Normal University, Linfen 041004,
People's Republic of China, and ^bDepartment of
Chemistry, University of Malaya, 50603 Kuala
Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
 $T = 291$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.040
 wR factor = 0.081
Data-to-parameter ratio = 16.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Poly[tris(ethylene-1,2-diammonium) bis[aqua-
(μ -sulfato- $\kappa^3\text{O},\text{O}':\text{O}''$)(μ -sulfato- $\kappa^4\text{O},\text{O}':\text{O}''',\text{O}''''$)-
(sulfato- κO)terbate(III)] tetrahydrate]

The Tb atom in the title compound, $\{(\text{C}_2\text{H}_{10}\text{N}_2)_3[\text{Tb}(\text{SO}_4)_3(\text{H}_2\text{O})_2] \cdot 4\text{H}_2\text{O}\}_n$, exists in a nine-coordinate geometry; the anion is linked through sulfate bridges into a three-dimensional network. The counter-ions and water molecules occupy the spaces within the framework and they consolidate the network structure through extensive hydrogen bonds.

Received 13 July 2006

Accepted 14 July 2006

Comment

The Ho atom in the dimethylammonium rare-earth sulfate double-salt $[(\text{CH}_3)_2\text{NH}_2][\text{Ho}(\text{SO}_4)_2(\text{H}_2\text{O})_2]$ exists in a bicapped trigonal prism in which four water molecules and two chelating sulfato groups comprise the coordination polyhedron. The study also reported the space group of the Y, Tb, Dy and Er analogues (Arhar *et al.*, 1984); presumably, the Tb^{III} compound has the same structure.

In the ethylenediammonium sulfatoterbate(III) (I), the $[\text{Tb}(\text{SO}_4)_3(\text{H}_2\text{O})]^{3-}$ anions are linked through sulfato bridges into a three-dimensional network (Fig. 1). The counter-ions and water molecules occupy the spaces within the framework and they consolidate the structure through extensive hydrogen bonds (Table 2). The metal is nine-coordinate (Fig. 2).

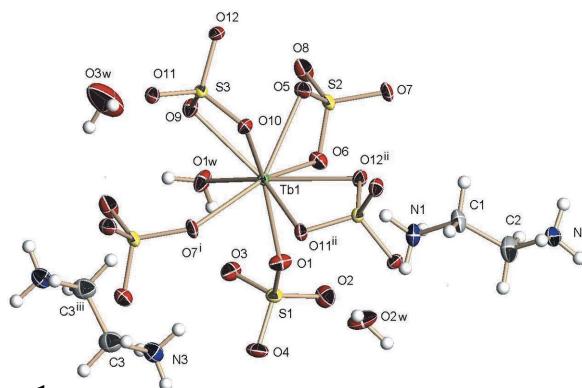
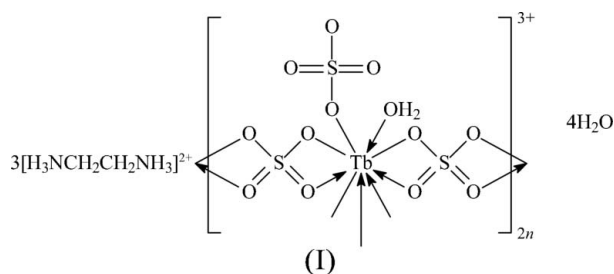


Figure 1
Part of the polymeric structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. [Symmetry codes: (i) $x + 1, y, z$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x, -y + 1, -z + 1$.]

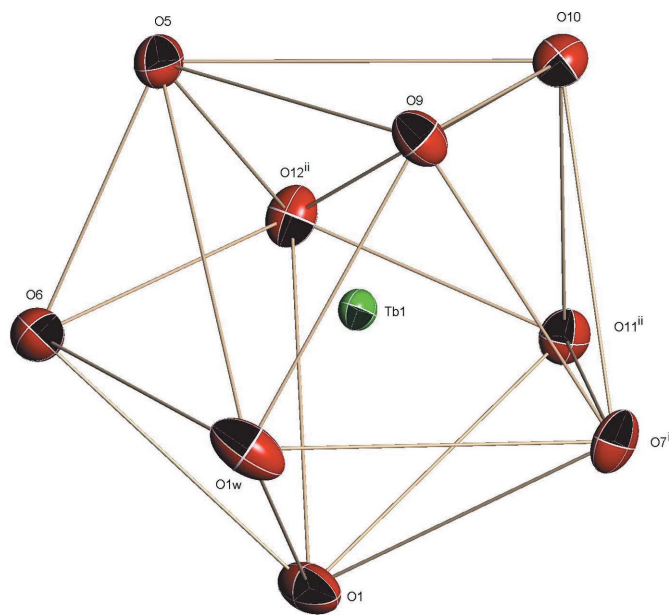


Figure 2
ORTEP (Johnson, 1976) plot illustrating the nine-coordinate geometry of Tb. Symmetry codes are as in Fig. 1 and Table 1.

Experimental

Terbium oxide, Tb₄O₇ (0.02 g, 0.03 mmol), was dissolved in an ethanol/water mixture (5 ml/7 ml), and to the solution were added concentrated sulfuric acid (0.12 ml) and ethylenediamine (0.06 ml, slight molar excess). The mixture was transferred into a 15 ml Teflon-lined stainless steel Parr bomb. The bomb was heated at 383 K for 2 d. After cooling the bomb to room temperature, colourless rod-like crystals were harvested by filtration in about 50% yield.

Crystal data

(C₂H₁₀N₂)₃[Tb(SO₄)₃(H₂O)]₂·4H₂O
M_r = 1188.66
 Monoclinic, *P*2₁/*c*
a = 6.5024 (4) Å
b = 26.392 (2) Å
c = 9.9070 (7) Å
 β = 103.733 (1)°
V = 1651.5 (2) Å³

Z = 2
D_x = 2.390 Mg m⁻³
 Mo Kα radiation
 μ = 4.75 mm⁻¹
T = 291 (2) K
 Rod, colourless
 0.14 × 0.06 × 0.03 mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.556, *T_{max}* = 0.871

13711 measured reflections
 3763 independent reflections
 3244 reflections with *I* > 2σ(*I*)
R_{int} = 0.050
θ_{max} = 27.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.040
wR(*F*²) = 0.081
S = 1.11
 3763 reflections
 229 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0304*P*)² + 2.0826*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 1.19 e Å⁻³
 Δρ_{min} = -1.53 e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Tb1—O1	2.307 (4)	Tb1—O10	2.489 (4)
Tb1—O5	2.465 (4)	Tb1—O11 ⁱⁱ	2.521 (4)
Tb1—O6	2.506 (4)	Tb1—O12 ⁱⁱ	2.479 (4)
Tb1—O7 ⁱ	2.368 (4)	Tb1—O1w	2.385 (4)
Tb1—O9	2.530 (4)		
O1—Tb1—O5	129.7 (1)	O6—Tb1—O11 ⁱⁱ	121.8 (1)
O1—Tb1—O6	74.2 (1)	O6—Tb1—O12 ⁱⁱ	72.7 (1)
O1—Tb1—O7 ⁱ	80.7 (1)	O6—Tb1—O1w	73.3 (1)
O1—Tb1—O9	141.7 (1)	O7 ⁱ —Tb1—O9	78.0 (1)
O1—Tb1—O10	147.2 (1)	O7 ⁱ —Tb1—O10	77.9 (1)
O1—Tb1—O11 ⁱⁱ	77.4 (1)	O7 ⁱ —Tb1—O11 ⁱⁱ	71.6 (1)
O1—Tb1—O12 ⁱⁱ	86.7 (1)	O7 ⁱ —Tb1—O12 ⁱⁱ	127.4 (1)
O1—Tb1—O1w	77.2 (1)	O7 ⁱ —Tb1—O1w	80.5 (1)
O5—Tb1—O6	56.4 (1)	O9—Tb1—O10	55.8 (1)
O5—Tb1—O7 ⁱ	148.2 (1)	O9—Tb1—O11 ⁱⁱ	124.1 (1)
O5—Tb1—O9	71.4 (1)	O9—Tb1—O12 ⁱⁱ	131.4 (1)
O5—Tb1—O10	77.8 (1)	O9—Tb1—O1w	68.1 (1)
O5—Tb1—O11 ⁱⁱ	119.2 (1)	O10—Tb1—O11 ⁱⁱ	72.4 (1)
O5—Tb1—O12 ⁱⁱ	71.0 (1)	O10—Tb1—O12 ⁱⁱ	86.8 (1)
O5—Tb1—O1w	95.9 (1)	O10—Tb1—O1w	122.7 (1)
O6—Tb1—O7 ⁱ	147.0 (1)	O11 ⁱⁱ —Tb1—O12 ⁱⁱ	55.8 (1)
O6—Tb1—O9	109.3 (1)	O11 ⁱⁱ —Tb1—O1w	144.8 (1)
O6—Tb1—O10	133.5 (1)	O12 ⁱⁱ —Tb1—O1w	145.2 (1)

Symmetry codes: (i) *x* + 1, *y*, *z*; (ii) *x*, -*y* + ½, *z* + ½.

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1w—H1w1...O3	0.85	1.79	2.623 (6)	165
O1w—H1w2...O8 ⁱ	0.85	2.36	2.942 (6)	127
O2w—H2w1...O2	0.85	1.93	2.750 (6)	161
O2w—H2w2...O4 ^{vii}	0.85	1.94	2.785 (6)	176
O3w—H3w1...O2w ^{vii}	0.85	2.04	2.852 (8)	159
O3w—H3w2...O9 ^{iv}	0.85	2.24	2.888 (7)	133
N1—H1N1...O4 ^v	0.85	1.97	2.811 (6)	173
N1—H1N2...O6	0.85	2.17	2.984 (6)	159
N1—H1N3...O3w	0.85	2.10	2.891 (8)	156
N2—H2N1...O10 ^{vi}	0.85	2.11	2.958 (6)	177
N2—H2N2...O5 ^{iv}	0.85	2.28	3.009 (6)	145
N2—H2N3...O12 ^{iv}	0.85	2.17	2.959 (7)	155
N3—H3N1...O6 ⁱⁱⁱ	0.85	2.50	3.308 (6)	159
N3—H3N2...O2w	0.85	1.97	2.758 (7)	155
N3—H3N3...O3 ^v	0.85	1.99	2.835 (6)	171

Symmetry codes: (i) *x* + 1, *y*, *z*; (iii) -*x*, -*y* + 1, -*z* + 1; (iv) *x*, *y*, *z* + 1; (v) *x* - 1, *y*, *z*; (vi) *x* - 1, -*y* + ½, *z* + ½; (vii) -*x* + 1, -*y* + 1, -*z* + 2.

H atoms were placed at calculated positions (C—H = 0.97 Å, N—H = 0.85 Å and O—H = 0.85 Å) and were included in the refinement in the riding model approximation, with *U*_{iso}(H) = 1.2*U*_{eq}(C,O) and 1.5*U*_{eq}(N); the amino groups were rotated to fit the electron density. The largest peak was about 1 Å from Tb1 and the deepest hole was about 1 Å from O9.

Data collection: SMART (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP (Johnson, 1976); software used to prepare material for publication: SHELXL97.

We thank the Natural Scientific Foundation Committee of Shanxi Province (No. 20041031) and the University of Malaya for generously supporting this study.

References

- Arhar, A., Golic, L., Jordanovska, V. & Siftar, J. (1984). *Vestn. Slov. Kem. Drus. (Bull. Solv. Chem. Soc.)*, **28**, 311–320.
- Bruker (2003). *SAINT* (Version 6.36A) and *SMART* (Version 6.36A). Bruker AXS Inc., Madison, Wisconsin, USA.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.