

Qing-Hua Wang

Department of Chemistry & Environmental Science, Zhangzhou Normal University, Zhangzhou, Fujian 363000, People's Republic of China

Correspondence e-mail: wqh_1974@yahoo.com.cn

Key indicators

Single-crystal X-ray study
 T = 292 K
 Mean $\sigma(C-C)$ = 0.026 Å
 R factor = 0.058
 wR factor = 0.158
 Data-to-parameter ratio = 23.6

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

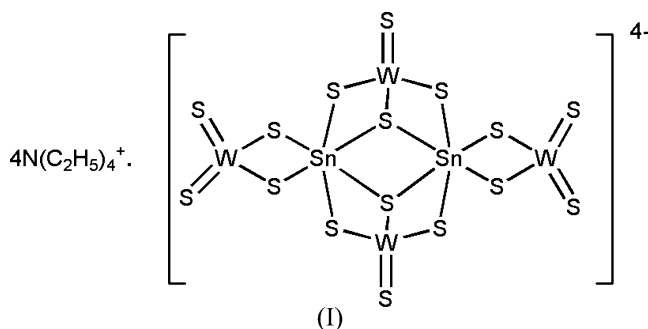
Tetrakis(tetraethylammonium) tetrakis(tetra-thio tungstenio)distannate(II)

In the title mixed-metal complex, $(Et_4N)_4[Sn_2(WS_4)_4]$, the Sn atoms are sixfold coordinated by S atoms of the tetrathio tungstate anions, forming two SnS_6 octahedra sharing a common edge. The anion is centrosymmetric.

Received 21 June 2006
 Accepted 6 July 2006.

Comment

The synthetic and structural chemistry of heterometallic Mo(W)/M/S clusters has attracted considerable attention for their uses as models for the active sites in a variety of metalloenzymes and their potential application as functional materials in several fields (Huang *et al.*, 1996; Riaz *et al.*, 1991). Investigating the structural characteristics of Mo(W)/M/S clusters may help us to understand the structure of the active center of nitrogenase. Here we report the structure of a tin-tungsten sulfido mixed-metal complex, (I).



The structure of (I) consists of discrete cations and centrosymmetric anions (Fig. 1). The structure of the anion is similar to that found in $[(C_6H_5)_4P]_4[Sn_2(WS_4)_4]$ (Müller *et al.*, 1976), which contains two highly distorted SnS_6 octahedra sharing a common edge. These octahedra are bonded to the four non-equivalent WS_4 tetrahedra *via* common edges. There are two modes of coordination of the ligands: two of the WS_4^{2-} ions are coordinated as bidentate ligands to Sn and possess two free S atoms, and the other WS_4^{2-} ions coordinate as tridentate ligands with one free S atom and one triply bonded sulfur atom. The $W \cdots Sn$ and $Sn \cdots Sn$ distances are 3.469 (6)–3.679 (6) and 4.489 (7) Å, respectively, indicating that there are no significant metal–metal interactions. The four-membered WS_2Sn and SnS_2Sn rings are planar. The distance from Sn to the doubly bonded S atoms are shorter than those to the triply bonded S atoms.

Experimental

0.5 mmol $SnCl_2$, 1 mmol $(NH_4)_2WS_4$ and 1 mmol Et_4NCl were dissolved in 10 ml of DMF, the mixture was then stirred for 10 min

and filtered. The filtrate was layered with methanol. Orange crystals of (I) were obtained after one day (yield 0.39 g, 77%).

Crystal data

(C₈H₂₀N)₄[Sn₂(WS₄)₄]
M_r = 2006.74
 Monoclinic, *P*2₁/*c*
a = 12.507 (2) Å
b = 13.188 (3) Å
c = 19.937 (6) Å
 β = 106.19 (5)°
V = 3157.7 (14) Å³

Z = 2
D_x = 2.110 Mg m⁻³
 Mo *K*α radiation
 μ = 8.59 mm⁻¹
T = 292 (2) K
 Prism, red
 0.20 × 0.10 × 0.10 mm

Data collection

Enraf Nonius CAD4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
T_{min} = 0.368, *T_{max}* = 0.432
 6470 measured reflections
 6188 independent reflections

5260 reflections with *I* > 2σ(*I*)
R_{int} = 0.084
 θ_{\max} = 26.0°
 3 standard reflections
 frequency: 60 min
 intensity decay: 0.5%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.058
wR (*F*²) = 0.158
S = 1.03
 6188 reflections
 262 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1158P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 2.69 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -3.29 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (Å).

Sn—S6	2.635 (3)	W1—S1	2.199 (3)
Sn—S7	2.790 (3)	W1—S3	2.206 (2)
Sn—S1	2.844 (3)	W2—S8	2.146 (3)
Sn—S4 ⁱ	2.893 (4)	W2—S5	2.156 (3)
W1—S2	2.144 (3)	W2—S7	2.222 (3)
W1—S4	2.190 (3)	W2—S6	2.236 (2)

Symmetry code: (i) $-x, -y + 1, -z + 1$.

H atoms were positioned geometrically and refined in riding mode [*C*—*H* = 0.96 Å, *U_{iso}*(*H*) = 1.5*U_{eq}*(*C*) for methyl H atoms and *C*—*H* = 0.97 Å, *U_{iso}*(*H*) = 1.2*U_{eq}*(*C*) for others]. The highest residual electron density peak is 0.90 Å from W1 and the deepest hole is 0.89 Å from W2.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*;

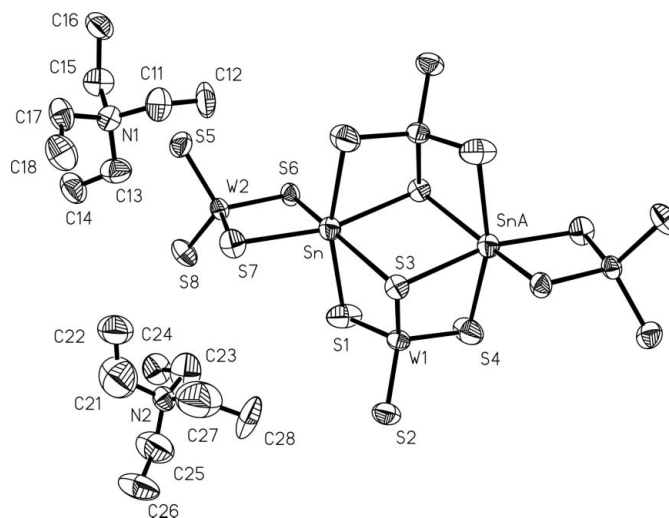


Figure 1

View of the title complex, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are omitted for clarity. [Symmetry code for SnA and unlabelled atoms: $-x, 1 - y, 1 - z$.]

program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ASP* (Chen, 2004); software used to prepare material for publication: *SHELXL97*.

Financial support was provided by the Education Bureau of Fujian Province, China (JA04246).

References

- Chen, J. T. (2004). *ASP*. Version 4.3. Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, China.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Huang, Q., Wu, X. T., Wang, Q. M., Sheng, T. L. & Lu, J. X. (1996). *Angew. Chem. Int. Ed. Engl.* **35**, 868–870.
- Müller, A., Paulat, I., Krebs, B. & Dornfeld, H. (1976). *Angew. Chem. Int. Ed. Engl.* **15**, 633.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Riaz, U., Curnow, O. & Curtis, M. D. (1991). *J. Am. Chem. Soc.* **113**, 1416–1423.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.