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Disorder in Bis(tetraethylammonium) Bis(μ -sulfido)bis[(1,2,3,4-tetrathiabutane-1,4-diyl-*S,S*)thiotungstate(V)]

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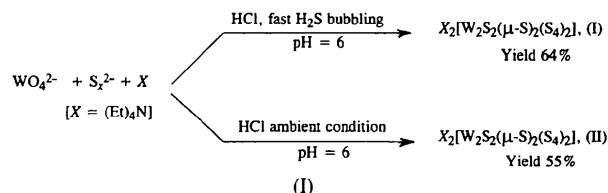
Abstract

Disorder in the terminal η^2 -S₄ ligands of the [W₂S₂(μ -S)₂(η^2 -S₄)₂]²⁻ anions of the title dinuclear tungsten-sulfur complex, di- μ -sulfido-1:2 κ^4 S-disulfido-1 κ S,2 κ S-bis(tetrasulfanediy1)-1 κ^2 S¹,S⁴;2 κ^2 S¹,S⁴-ditungsten(V), (Et₄N)₂[W₂S₂(μ -S)₂(η^2 -S₄)₂], results in two anionic orientations (*A* and *B*). In both orientations, the metal center is bonded to five S atoms, with a distorted square-pyramidal coordination polyhedron. The dihedral angle between orientations *A* and *B*, defined as that between the basal planes through two bridging and two η^2 -S atoms of the anion, is 9.5(3)°. The five-membered chelate rings of the anions in orientations *A* and *B* display envelope geometry, with atoms S9 and S3 at the flaps.

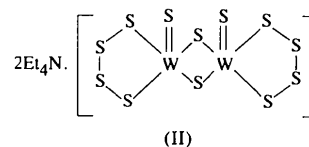
Comment

Polysulfide ions (S_x²⁻, *x* = 2–9) have attracted attention because of their reducing as well as polydentate ligand behavior towards metal ions (Muller, Jaegermann & Enemark, 1982; Draganjac & Rauchfuss, 1985; Shibahara, 1993). Reaction of metal complexes with S_x²⁻ ions is a convenient method for directly introducing the ligand, leading to a variety of mono- or polymetal-polysulfur complexes (Muller & Diemann, 1987; Bhattacharya *et al.*, 1991; Chakraborty, Bhatta-

charya, Pierpont & Bhattacharya, 1992; Yamasaki & Shibahara, 1993). As part of our systematic study of tungstate-polysulfide systems, the title compound, (Et₄N)₂[W₂S₂(μ -S)₂(η^2 -S₄)₂], was obtained in two forms [(I) and (II)] by acidification (6 *M* HCl) of an alkaline solution containing S_x²⁻, WO₄²⁻ and Et₄N⁺ following the reaction scheme below.



The synthesis, electrochemistry, reaction mechanism and crystal structure of (I) have been described previously (Chakraborty *et al.*, 1996). We report here the synthesis and crystal structure of (Et₄N)₂[W₂S₂(μ -S)₂(η^2 -S₄)₂], (II). The structure of (II) consists of dinuclear units of disordered complex [W₂S₁₂]²⁻ anions and disordered tetraethylammonium (Et₄N)⁺ cations. The anions are located at crystallographic twofold sites with half of the dimer comprising the asymmetric unit. The *syn*-[W₂S₂(μ -S)₂]²⁺ core in the anion of (II), unlike that of (I), is coordinated by two bidentate η^2 -S₄ ligands disordered over two well defined unequally occupied orientations, *A* and *B*, with occupancy factors of 0.61 (1) and 0.39 (1), respectively. Orientation *A* comprises atoms S7–S10 and orientation *B*, atoms S3–S6.



A comparative study of the skeletal geometry of the anion with several related tungsten-sulfur complexes is reported in Table 3. The single crystallographically independent W atom in [W₂S₁₂]²⁻ is bonded to five S atoms in a distorted square-pyramidal arrangement. The distortion of the coordination sphere is reflected in the wide range of angles formed by the S atoms at the metal center; S_{apical}—W—S_{basal} 101.3(3)–119.7(3)°, *cis*-S_{basal}—W—S_{basal} 66.4(4)–100.7(1)° and *trans*-S_{basal}—W—S_{basal} 131.7(3)–152.2(2)°. The apical site occupied by the S1 atom is 2.746(5) (for orientation *A*) and 2.925(5) Å (for *B*) away from the basal planes through S2, S2ⁱ, S7, S10 and S2, S2ⁱ, S3, S6, respectively, with the metal W1 atom deviating by 0.642(3) (for orientation *A*) and 0.835(3) Å (for *B*) in the same direction as the S1 atom. The dihedral angle between the basal planes is 9.5(3)°. The bridging S atoms (S_b) are symmetrically bonded to W atoms and the W—S_b [2.308(8) and 2.332(9) Å] and W=S [2.106(4) Å] bond lengths are comparable to the corresponding distances in similar systems (Table 3).

The five-membered WS_4 chelate rings in orientations *A* and *B* display envelope geometry, with the S9 and S3 atoms forming the flaps of the envelope and deviating by 1.19 (1) and 1.32 (2) Å, respectively, from the corresponding least-squares planes through the remaining endocyclic atoms. The dihedral angle between the planar parts of the chelate rings in the two orientations is 32.5 (3)°. The discrepancy between the two $W-S_x$ (η^2-S) distances [W1—S7 2.366 (8) and W1—S10 2.531 (7), and W1—S3 2.426 (10) and W1—S6 2.326 (8) Å] in the two orientations can be attributed to the unresolved disorder of the η^2-S atoms. The $W \cdots W$ separation of 2.816 (1) Å is indicative of a metal–metal single bond in the anion. The $(Et_4N)^+$ cations lie on symmetry sites $\bar{4}$ and 2 and are well behaved, while the cations occupying 222 sites are statistically disordered about the ethyl C-atom positions, with occupancy factors of 0.52 (3) and 0.48 (3). Contrary to the previous reports on $(Et_4N)_2[W_2S_2(\mu-S)_2(\eta^2-S_4)_2]$, with unresolved anionic disorder, a short S—S bond length of 1.67 (2) Å (Cohen & Stiefel, 1985) and unusually high temperature factors for the S_4 -ligand atoms (Chakraborty *et al.*, 1996), the present structure analysis does not show any anomalies in either bond distances or displacement parameters.

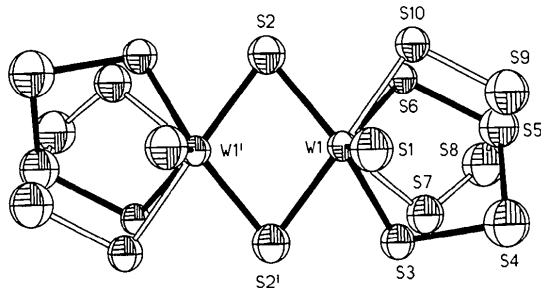


Fig. 1. SHELTLP (Sheldrick, 1994) view of the two orientations (*A* and *B*) of the anion showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Orientation *A* comprises atoms S7–S10, shown by hollow bonds, and orientation *B* atoms S3–S6, indicated by solid bonds.

Experimental

To the orange–red solution obtained by mixing sodium tungstate ($Na_2WO_4 \cdot 2H_2O$; 3.0 g, 0.009 mol), tetraethylammonium bromide (Et_4NBr ; 3.8 g, 0.018 mol) and aqueous ammonium polysulfide $[(NH_4)_2S_x$; 75 ml], 6 *M* hydrochloric acid was added dropwise with constant stirring. A vigorous reaction started with evolution of H_2S gas and in the pH range 8–6, the colour of the solution changed from orange–red to deep green, with a red compound separating out along with elemental sulfur. Addition of HCl was stopped when the reaction solution reached pH 6, but stirring was continued for another 15 min. The separated compound was filtered off, washed thoroughly with water, ethanol, CS_2 and diethyl ether, and dried *in vacuo*. Maroon crystals were obtained by recrystallization from an acetone–light petroleum (313–333 K) mixture. Yield 2.5 g (55%). Elemental analysis found: C 19.4, H 4.2, N 2.9, S 37.2, W 36.5%; $[(C_2H_5)_4N]_2[W_2S_2(\mu-$

$S)_2(S_4)_2]$ requires: C 19.0, H 4.0, N 2.8, S 38.0, W 36.3%. The IR spectrum (Perkin Elmer 597 IR spectrometer) showed a strong $\nu(S-S)$ vibration at 505 cm^{-1} (corresponding to $\eta^2-S_4^{2-}$ ligands) and a weak $\nu(WS_2W)$ asymmetric vibration at 440 cm^{-1} . Two other weak bands at 330 and 310 cm^{-1} were assigned to a $\nu(W-S_x)$ vibration. The complex was diamagnetic and behaved as expected for a 2:1 electrolyte ($\Lambda = 250\text{ W}^{-1}\text{ cm}^2\text{ M}^{-1}$) in CH_3CN . The diamagnetism of the complex indicated the existence of a metal–metal bond in the dimeric $W^V d^1$ system, subsequently confirmed by X-ray crystallographic analysis.

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

$$U_{iso} \text{ for W1, S1, S2, N1, C1 and C2, } U_{eq} = (1/3)\sum_i \sum_j U^{ij} a_i^* a_j^* a_i \cdot a_j \text{ for others.}$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}/U_{eq}
S3†	0.2441 (6)	0.4129 (5)	0.1267 (7)	0.080 (3)
S6†	0.2259 (4)	0.4096 (4)	0.3368 (7)	0.060 (2)
S4†	0.1916 (8)	0.4996 (8)	0.1486 (11)	0.140 (6)
S5†	0.1542 (6)	0.4682 (6)	0.2700 (9)	0.114 (4)
S7‡	0.2283 (4)	0.4336 (4)	0.1607 (6)	0.094 (2)
S10‡	0.2433 (4)	0.4365 (4)	0.3775 (5)	0.081 (2)
S8‡	0.1445 (5)	0.4419 (5)	0.2211 (6)	0.130 (3)
S9‡	0.1844 (5)	0.4991 (5)	0.3136 (7)	0.130 (3)
W1	0.31686 (2)	0.41089 (2)	0.25057 (11)	0.0667 (2)
S1	0.3730 (2)	0.4930 (2)	0.2459 (8)	0.0935 (15)
S2	0.3483 (5)	0.3488 (5)	0.3682 (2)	0.0787 (9)
N1	0	0	0	0.061 (4)
C1	0.0567 (8)	−0.0021 (7)	−0.0594 (10)	0.080 (4)
C2	0.1206 (7)	−0.004 (2)	−0.011 (3)	0.103 (8)
N2	1/2	0	0	0.094 (6)
C3A§	0.540 (2)	−0.0467 (17)	−0.055 (2)	0.137 (15)
C3B§	0.5754 (12)	−0.1013 (12)	−0.014 (2)	0.075 (8)
C4A¶	0.4654 (17)	−0.0401 (17)	−0.070 (2)	0.100 (11)
C4B¶	0.419 (2)	−0.085 (2)	−0.023 (3)	0.115 (15)
N3	0.1957 (6)	0.1957 (6)	0.2500	0.098 (4)
C5	0.1990 (13)	0.2569 (11)	0.3038 (16)	0.146 (9)
C6	0.151 (3)	0.248 (3)	0.379 (4)	0.28 (3)
C7	0.1941 (13)	0.1355 (12)	0.3048 (17)	0.154 (9)
C8	0.254 (2)	0.128 (2)	0.361 (3)	0.216 (19)

† Site occupancy = 0.39 (1). ‡ Site occupancy = 0.61 (1). § Site occupancy = 0.52 (3). ¶ Site occupancy = 0.48 (3).

Table 2. Selected geometric parameters (Å , °)

S3—S4	2.17 (2)	S10—S9	2.061 (11)
S3—W1	2.426 (10)	S10—W1	2.531 (7)
S6—S5	2.206 (14)	S8—S9	2.035 (12)
S6—W1	2.326 (8)	W1—S1	2.106 (4)
S4—S5	2.11 (2)	W1—S2	2.308 (8)
S7—S8	2.003 (11)	W1—S2'	2.332 (9)
S7—W1	2.366 (8)	W1—W1'	2.8157 (11)
S4—S3—W1	102.8 (6)	S1—W1—S7	105.1 (3)
S5—S6—W1	107.8 (5)	S2—W1—S7	141.9 (3)
S3—S4—S5	93.3 (8)	S1—W1—S3	108.5 (4)
S4—S5—S6	108.5 (7)	S2—W1—S3	142.0 (3)
S8—S7—W1	117.3 (5)	S2'—W1—S3	66.4 (4)
S9—S10—W1	98.9 (4)	S6—W1—S3	84.7 (5)
S7—S8—S9	89.9 (6)	S1—W1—S10	101.3 (3)
S8—S9—S10	101.0 (6)	S2—W1—S10	73.5 (3)
S1—W1—S2	109.4 (3)	S2'—W1—S10	152.2 (2)
S2—W1—S2'	100.69 (11)	S7—W1—S10	84.5 (3)
S1—W1—S6	119.7 (3)	W1—S2—W1'	74.71 (9)
S2—W1—S6	78.5 (3)		
S7—W1—S10—S9	−28.4 (4)	S3—W1—S6—S5	−35.4 (5)
W1—S10—S9—S8	64.1 (5)	W1—S6—S5—S4	3.4 (9)
S10—S9—S8—S7	−67.4 (6)	S6—S5—S4—S3	34.9 (9)
S9—S8—S7—W1	47.4 (6)	S5—S4—S3—W1	−62.3 (8)
S8—S7—W1—S10	−11.9 (5)	S4—S3—W1—S6	60.5 (6)

Symmetry code: (i) $y, x, \frac{1}{2} - z$.

Table 3. A comparison of the geometric parameters[†] (Å, °) of the {W₂S₂(μ-S)₂} fragment of some dinuclear tungsten complexes

Complex	W...W	W—S ^m	W—S _β ^m	W—S _γ ^m	S _b —M—S _b	Reference
(Et ₄ N) ₂ [W ₂ S ₄ (S ₄) ₂], (II)	2.816 (1)	2.106 (4)	2.320 (9)	2.412 (8)	100.7 (1)	(a)
(Et ₄ N) ₂ [W ₂ S ₄ (S ₄) ₂], (I)	2.823 (1)	2.100 (8)	2.32 (1)	2.38 (2)	100.8 (2)	(b)
(Et ₄ N) ₂ [W ₂ S ₄ (S ₄) ₂]	2.836 (2)	2.116 (5)	2.320 (6)	2.387 (11)	100.1 (2)	(c)
W ₂ S ₄ [S ₂ P(OEt) ₂] ₂	2.819 (1)	2.114 (5)	2.314 (5)	2.482 (5)	102.0 (2)	(d)
[W ₂ S ₄ (S ₂ N(CEt) ₂) ₂]	2.795 (1)	2.09 (1)	2.31 (1)	2.44 (1)	101.9 (3)	(d)
(PPh ₄)[W ₂ S ₄ (S ₄) ₂].0.5DMF	2.834 (2)	2.103 (10)	2.321 (8)	2.415 (9)	101.5 (3)	(e)
(PPh ₄)[W ₂ S ₄ (S ₂ S ₄)] ₂ .0.5DMF	2.840 (2)	2.125 (8)	2.309 (7)	2.374 (9)	101.9 (2)	(e)
(PPh ₄)[W ₂ S ₄ {S ₂ (S)PPh} ₂]	2.828 (1)	2.118 (3)	2.326 (3)	2.446 (3)	101.2	(f)
(PPh ₄)[W ₂ S ₄ (S ₂ C ₂ H ₄) ₂]	2.862 (1)	2.144 (2)	2.328 (2)	2.404 (2)	100.7	(g)
[W ₂ S ₄ Br ₂ PPh ₃].C ₆ H ₆	2.813 (3)	2.11 (1)	2.31 (1)	—	101.6 (5)	(h)

References: (a) this work; (b) Chakraborty *et al.* (1996); (c) Cohen & Stiefel (1985); (d) Drew, Hobson, Mumba, Rice & Trup (1987); (e) Müller *et al.* (1985); (f) Slater, Garner & Clegg (1990); (g) Pan, Chandler, Enemark & Stiefel (1984); (h) Feddin, Mironov, Virovets, Podberezskaya & Fedorov (1990).

[†] The average values of chemically equivalent and crystallographically independent bond lengths and angles are given. S_β^m, S_γ^m and S_γ^m represent bridging, η² bonded and multiply bonded terminal sulfur, respectively.

Crystal data

(C ₈ H ₂₀ N) ₂ [W ₂ S ₁₂]	Mo Kα radiation
<i>M_r</i> = 1012.92	λ = 0.71073 Å
Tetragonal	Cell parameters from 30 reflections
<i>I</i> 4̄c2	θ = 7.5–15.0°
<i>a</i> = 21.174 (4) Å	μ = 7.534 mm ⁻¹
<i>c</i> = 15.119 (2) Å	<i>T</i> = 293 (2) K
<i>V</i> = 6778 (2) Å ³	Parallelepiped
<i>Z</i> = 8	0.30 × 0.22 × 0.10 mm
<i>D_x</i> = 1.985 Mg m ⁻³	Maroon
<i>D_m</i> = 1.97 Mg m ⁻³	
<i>D_m</i> measured by flotation in ethyl iodide/1,3-dibromopropane	

Data collection

Enraf–Nonius CAD-4 diffractometer	2190 reflections with <i>I</i> > 2σ(<i>I</i>)
ω–2θ scans	<i>R</i> _{int} = 0.0344
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)	θ _{max} = 24.97°
<i>T</i> _{min} = 0.151, <i>T</i> _{max} = 0.471	<i>h</i> = –24 → 0
5322 measured reflections	<i>k</i> = –14 → 25
2828 independent reflections	<i>l</i> = –17 → 14
	3 standard reflections every 100 reflections
	intensity decay: 3%

Refinement

Refinement on <i>F</i> ²	Δρ _{max} = 0.80 e Å ⁻³
<i>R</i> (<i>F</i>) = 0.0484	Δρ _{min} = –0.66 e Å ⁻³
<i>wR</i> (<i>F</i> ²) = 0.1244	Extinction correction: none
<i>S</i> = 1.044	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
2828 reflections	Absolute configuration: Flack (1983)
115 parameters	Flack parameter = 0.47 (4)
<i>w</i> = 1/[σ ² (<i>F</i> _o ²) + (0.07 <i>P</i>) ² + 74.7 <i>P</i>]	
where <i>P</i> = (<i>F</i> _o ² + 2 <i>F</i> _c ²)/3	
(Δ/σ) _{max} = 0.610	

Intensity distributions indicated 4/*mmm* Laue symmetry with the following systematic absences: *hkl* (*h* + *k* + *l* ≠ 2*n*), *hk0* (*h* + *k* ≠ 2*n*) and *0kl* (*k* + *l* ≠ 2*n*). Of the three possible space groups, *I*4*cm*, *I*4/*mcm* and *I*4̄c2, the last was chosen

and a successful structure refinement confirmed the space group assignment. The W atom was located from Patterson interpretation and the remaining atoms were found through successive difference Fourier calculations. Two orientations for the terminal S₄ ligands in the anion and the (Et₄N)⁺ cation occupying the 222 site were apparent in the structure solution. The occupancy factors for the two orientations of the S₄-ligands in the anion and the disordered cation refined as least-squares parameters, with the sum constrained to be 1.0, were 0.61 (1)/0.39 (1) (for the S₄ ligand) and 0.52 (3)/0.48 (3) (for the disordered cation). Iterative refinement of atomic positions and anisotropic displacement parameters for all atoms, except those of the disordered S₄ ligand and cations, which were modeled with isotropic displacement parameters, was performed using *SHELXL93* (Sheldrick, 1993). Only the H atoms of the ordered cations were included in the structure-factor calculations at idealized positions, but they were not refined.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990).

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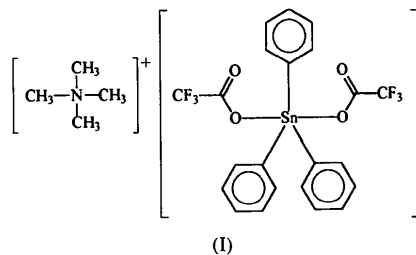
Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1055). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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phenyltin alkanoates, with rare exceptions (Ng, 1995), do not yield complexes with Lewis bases. The appearance of Lewis acidity in triphenyltin trifluoroacetate (Ford & Sams, 1971), which forms a hydrate that is hydrogen bonded to 1,10-phenanthroline (Ng, Kumar Das & Kennard, 1996), is unambiguously proven in tetramethylammonium triphenylbis(trifluoroacetato)stannate, (I). The Lewis acidity of (I) is comparable to that of triphenyltin chloride as it can accept an essentially non-basic trifluoroacetato anion (Brown, 1980). The triphenylbis(trifluoroacetato)stannate ion constitutes the first example of a di(carboxylato)triorganostannate (Tiekink, 1991, 1994).



The discrete nature of this triphenylbis(trifluoroacetato)stannate contrasts with that of (dicarboxylato)triphenylstannates, such as the oxalato (Ng, Kumar Das, Luo & Mak, 1994) and succinato (Ng, Kumar Das, Xiao, van der Helm, Holecek & Lycka, 1991) derivatives, which adopt linear polyanionic chain structures.

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The First Example of a Discrete Di(carboxylato)triorganostannate: Tetramethylammonium Triphenylbis(trifluoroacetato)stannate

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Abstract

Tetramethylammonium triphenylbis(trifluoroacetato)-stannate crystallizes as non-interacting tetrahedral $[(CH_3)_4N]^+$ cations and *trans*-trigonal bipyramidal $[Sn(C_2F_3O_2)_2(C_6H_5)_3]^-$ anions. The anion is the first example of a discrete triorganostannate covalently bonded to two unidentate monocarboxylato anions.

Comment

As a result of their preference for forming carboxyl bridges that link adjacent molecules into five-coordinate polymeric chains (Ng, Chen & Kumar Das, 1988), tri-

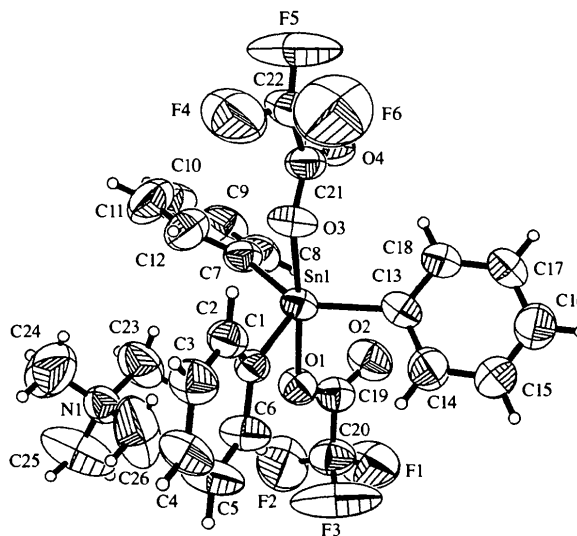


Fig. 1. ORTEP (Johnson, 1976) plot of tetramethylammonium triphenylbis(trifluoroacetato)stannate with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Experimental

Tetramethylammonium hydroxide, triphenyltin hydroxide and trifluoroacetic acid (molar ratio 1:1:2) were heated in a small volume of ethanol. The filtered solution when cooled slowly