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Di-μ-sulfido-bis[(2-aminoethanethiolato-κ^2N,S)oxidomolybdate(V)]

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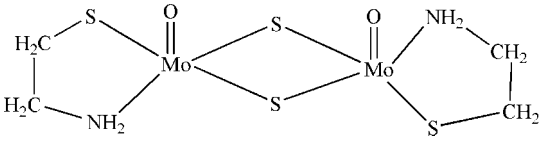
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.015 Å; R factor = 0.057; wR factor = 0.115; data-to-parameter ratio = 17.9.

The title compound, $[Mo_2O_2S_2(C_2H_6NS)_2]$, was obtained by the reaction of sodium molybdate, H_2S, sodium hydrosulfite and 2-aminoethanethiol. In the crystal structure, each Mo^V atom, in a square-pyramidal geometry, is coordinated by two μ-S atoms, one terminal O atom, one S atom and one N atom of the 2-aminoethanethiolate ligand. A three-dimensional hydrogen-bonding network is constructed by intermolecular N—H···O and N—H···S hydrogen bonds.

Related literature

For related structures, see: Bunzey *et al.* (1977); Drew & Kay (1971); Dulebohn *et al.* (1991); Howlader *et al.* (1984); Li *et al.* (2005); Llusar *et al.* (2005); Müller *et al.* (1982); Spivack & Dori (1970).



Experimental

Crystal data

$[Mo_2O_2S_2(C_2H_6NS)_2]$
M_r = 440.28
Monoclinic, $P2_1/c$
a = 10.2359 (9) Å
b = 12.5935 (11) Å
c = 10.2596 (9) Å
β = 104.166 (2)°
V = 1282.30 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.59 mm^{-1}
T = 293 (2) K
0.30 × 0.22 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.492, T_{max} = 0.633
6415 measured reflections
2268 independent reflections
1753 reflections with $I > 2\sigma(I)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.115
S = 1.18
2268 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mo1—O1	1.676 (7)	Mo2—O2	1.683 (7)
Mo1—N1	2.209 (7)	Mo2—N2	2.211 (8)
Mo1—S4	2.306 (3)	Mo2—S2	2.312 (3)
Mo1—S2	2.349 (3)	Mo2—S4	2.329 (3)
Mo1—S1	2.391 (3)	Mo2—S3	2.383 (3)
Mo1—Mo2	2.8197 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O1^{i}	0.90	2.25	3.025 (11)	144
N1—H1D···S3^{ii}	0.90	2.53	3.400 (8)	161
N2—H2C···S2^{iii}	0.90	2.81	3.704 (9)	173
N2—H2D···S1^{iv}	0.90	2.50	3.403 (8)	178

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x, -y+1, -z$; (iv) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Siemens, 1996); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2065).

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supplementary materials

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Di-μ-sulfido-bis[(2-aminoethanethiolato-κ^2N,S)oxidomolybdate(V)]

Y.-M. Li, J.-L. Zhang and X.-W. Zhao

Comment

Molybdenum chalcogenides have been extensively studied due to their applications in modelling of molybdoenzymes in biological systems. During the course of exploring polynuclear molybdenum clusters, dinuclear molybdenum complexes are always considered as excellent starting materials. Many compounds containing a $[Mo_2O_2(\mu\text{-}S)_2]$ structural unit have been isolated and structurally characterized (Bunzey *et al.*, 1977; Drew & Kay, 1971; Dulebohn *et al.*, 1991; Howlader *et al.*, 1984; Li *et al.*, 2005; Llusar *et al.*, 2005; Müller *et al.*, 1982; Spivack & Dori,1970). Herein, we report the crystal structure of a new dinuclear molybdenum complex, (I).

In the structure of (I) (Fig. 1), the two Mo atoms are not crystallographically equivalent, which are linked by two μ-S atoms. Each Mo atom is chelated by one 2-aminoethanethiolate (aet) ligand in the equatorial plane. The two aet ligands are in a *trans* form. Each Mo atom is also bonded to one terminal O atom in the axial position, resulting in a five-coordinated square-pyramidal coordination geometry. In virtue of weak intermolecular N—H···O and N—H···S hydrogen bonds, a three-dimensional hydrogen-bonding network is constructed (Fig. 2).

Experimental

The compound (I) was synthesized by bubbling H_2S gas into a 20 ml aqueous solution of $Na_2MoO_4{\cdot}2H_2O$ (4.84 g, 20 mmol). The solids of $Na_2S_2O_4$ (0.469 g, 2.7 mmol) and 2-aminoethanethiol (2 g, 26 mmol) were added to the red solution with stirring for about 4 h at 358 K. After cooling to room temperature, the solution was filtered and the residue was dissolved in 10 ml DMF. Orange prism crystals of (I) were obtained by slow evaporation of the orange solution for several weeks.

Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.97 and N—H = 0.90 Å and U_{iso}(H) = 1.2U_{eq}(C, N).

Figures

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. A view of the crystal packing along the *c* axis. Hydrogen bonds are shown as dashed lines.

Di-μ-sulfido-bis[(2-aminoethanethiolato-κ^2*N*,*S*)oxidomolybdate(V)](Mo—Mo)

Crystal data

$[Mo_2O_2S_2(C_2H_6NS)_2]$	F_{000} = 856
M_r = 440.28	D_x = 2.281 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 245 reflections
a = 10.2359 (9) Å	θ = 2.1–25.1°
b = 12.5935 (11) Å	μ = 2.59 mm^{-1}
c = 10.2596 (9) Å	T = 293 (2) K
β = 104.166 (2)°	Prism, orange
V = 1282.30 (19) Å^3	0.30 × 0.22 × 0.18 mm
Z = 4	

Data collection

Siemens SMART CCD area-detector diffractometer	2268 independent reflections
Radiation source: fine-focus sealed tube	1753 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.073
T = 293(2) K	θ_{max} = 25.1°
φ and ω scans	θ_{min} = 2.1°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −11→12
T_{min} = 0.492, T_{max} = 0.633	k = −14→15
6415 measured reflections	l = −11→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 11.6194P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.18	$(\Delta/\sigma)_{max} < 0.001$
2268 reflections	$\Delta\rho_{max}$ = 0.75 e Å^{-3}
127 parameters	$\Delta\rho_{min}$ = −0.77 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}

Mo1	0.24088 (8)	0.28563 (7)	0.23707 (8)	0.0308 (2)
Mo2	0.26812 (8)	0.50741 (7)	0.21672 (8)	0.0314 (2)
S1	0.0614 (2)	0.1740 (2)	0.1200 (3)	0.0390 (6)
S2	0.0689 (2)	0.4136 (2)	0.1873 (3)	0.0398 (6)
S3	0.3706 (3)	0.5979 (2)	0.0634 (3)	0.0428 (6)
S4	0.4155 (2)	0.3758 (2)	0.1811 (3)	0.0446 (7)
O1	0.2747 (8)	0.2657 (6)	0.4035 (7)	0.057 (2)
O2	0.3266 (8)	0.5483 (6)	0.3766 (7)	0.0517 (19)
N1	0.3497 (7)	0.1462 (6)	0.1863 (8)	0.0345 (18)
H1C	0.3588	0.1535	0.1017	0.041*
H1D	0.4329	0.1445	0.2416	0.041*
N2	0.1333 (8)	0.6454 (6)	0.1567 (8)	0.041 (2)
H2C	0.0776	0.6332	0.0755	0.050*
H2D	0.0826	0.6544	0.2162	0.050*
C1	0.1444 (9)	0.0480 (8)	0.1021 (10)	0.038 (2)
H1A	0.0911	−0.0105	0.1224	0.046*
H1B	0.1522	0.0400	0.0103	0.046*
C2	0.2825 (9)	0.0463 (8)	0.1971 (10)	0.042 (2)
H2A	0.2747	0.0362	0.2886	0.050*
H2B	0.3347	−0.0121	0.1743	0.050*
C3	0.2878 (11)	0.7277 (9)	0.0442 (10)	0.049 (3)
H3A	0.3548	0.7833	0.0516	0.059*
H3B	0.2262	0.7327	−0.0441	0.059*
C4	0.2115 (12)	0.7426 (8)	0.1509 (11)	0.048 (3)
H4A	0.2737	0.7553	0.2373	0.058*
H4B	0.1515	0.8031	0.1293	0.058*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.0347 (5)	0.0266 (5)	0.0306 (4)	0.0024 (4)	0.0070 (3)	0.0003 (4)
Mo2	0.0363 (4)	0.0267 (5)	0.0305 (4)	−0.0013 (4)	0.0068 (3)	−0.0026 (3)
S1	0.0306 (12)	0.0306 (14)	0.0560 (16)	−0.0003 (10)	0.0108 (11)	0.0003 (12)
S2	0.0340 (13)	0.0301 (14)	0.0586 (16)	0.0021 (11)	0.0176 (12)	0.0041 (12)
S3	0.0438 (14)	0.0433 (16)	0.0441 (15)	−0.0091 (12)	0.0159 (12)	−0.0018 (12)
S4	0.0309 (13)	0.0344 (16)	0.0696 (18)	−0.0044 (11)	0.0142 (12)	−0.0091 (13)
O1	0.083 (5)	0.054 (5)	0.036 (4)	0.014 (4)	0.016 (4)	0.001 (4)
O2	0.076 (5)	0.040 (5)	0.037 (4)	0.000 (4)	0.008 (4)	−0.005 (3)
N1	0.029 (4)	0.025 (4)	0.047 (5)	0.004 (3)	0.005 (3)	−0.001 (4)
N2	0.050 (5)	0.034 (5)	0.043 (5)	0.004 (4)	0.017 (4)	0.001 (4)
C1	0.043 (6)	0.025 (5)	0.050 (6)	−0.001 (4)	0.015 (5)	−0.003 (5)
C2	0.041 (6)	0.036 (6)	0.046 (6)	−0.002 (5)	0.008 (5)	0.005 (5)
C3	0.054 (6)	0.046 (7)	0.050 (6)	−0.005 (5)	0.015 (5)	0.011 (5)
C4	0.073 (7)	0.018 (5)	0.050 (6)	0.000 (5)	0.007 (6)	0.002 (4)

Geometric parameters (Å, °)

Mo1—O1	1.676 (7)	N1—H1D	0.9000
Mo1—N1	2.209 (7)	N2—C4	1.471 (12)

Mo1—S4	2.306 (3)	N2—H2C	0.9000
Mo1—S2	2.349 (3)	N2—H2D	0.9000
Mo1—S1	2.391 (3)	C1—C2	1.508 (13)
Mo1—Mo2	2.8197 (12)	C1—H1A	0.9700
Mo2—O2	1.683 (7)	C1—H1B	0.9700
Mo2—N2	2.211 (8)	C2—H2A	0.9700
Mo2—S2	2.312 (3)	C2—H2B	0.9700
Mo2—S4	2.329 (3)	C3—C4	1.505 (14)
Mo2—S3	2.383 (3)	C3—H3A	0.9700
S1—C1	1.829 (10)	C3—H3B	0.9700
S3—C3	1.830 (11)	C4—H4A	0.9700
N1—C2	1.451 (12)	C4—H4B	0.9700
N1—H1C	0.9000		
O1—Mo1—N1	97.5 (3)	C2—N1—H1C	108.9
O1—Mo1—S4	110.4 (3)	Mo1—N1—H1C	108.9
N1—Mo1—S4	82.3 (2)	C2—N1—H1D	108.9
O1—Mo1—S2	106.4 (3)	Mo1—N1—H1D	108.9
N1—Mo1—S2	152.7 (2)	H1C—N1—H1D	107.7
S4—Mo1—S2	101.13 (10)	C4—N2—Mo2	110.9 (6)
O1—Mo1—S1	111.4 (3)	C4—N2—H2C	109.5
N1—Mo1—S1	77.5 (2)	Mo2—N2—H2C	109.5
S4—Mo1—S1	135.38 (10)	C4—N2—H2D	109.5
S2—Mo1—S1	81.46 (9)	Mo2—N2—H2D	109.5
O1—Mo1—Mo2	103.0 (3)	H2C—N2—H2D	108.1
N1—Mo1—Mo2	134.8 (2)	C2—C1—S1	109.5 (7)
S4—Mo1—Mo2	52.90 (7)	C2—C1—H1A	109.8
S2—Mo1—Mo2	52.17 (6)	S1—C1—H1A	109.8
S1—Mo1—Mo2	128.55 (7)	C2—C1—H1B	109.8
O2—Mo2—N2	94.6 (3)	S1—C1—H1B	109.8
O2—Mo2—S2	112.1 (3)	H1A—C1—H1B	108.2
N2—Mo2—S2	84.0 (2)	N1—C2—C1	109.0 (8)
O2—Mo2—S4	106.3 (3)	N1—C2—H2A	109.9
N2—Mo2—S4	154.1 (2)	C1—C2—H2A	109.9
S2—Mo2—S4	101.55 (9)	N1—C2—H2B	109.9
O2—Mo2—S3	112.8 (3)	C1—C2—H2B	109.9
N2—Mo2—S3	77.6 (2)	H2A—C2—H2B	108.3
S2—Mo2—S3	132.44 (10)	C4—C3—S3	110.0 (7)
S4—Mo2—S3	80.21 (10)	C4—C3—H3A	109.7
O2—Mo2—Mo1	104.4 (3)	S3—C3—H3A	109.7
N2—Mo2—Mo1	137.1 (2)	C4—C3—H3B	109.7
S2—Mo2—Mo1	53.37 (7)	S3—C3—H3B	109.7
S4—Mo2—Mo1	52.15 (7)	H3A—C3—H3B	108.2
S3—Mo2—Mo1	126.15 (8)	N2—C4—C3	108.1 (8)
C1—S1—Mo1	104.0 (3)	N2—C4—H4A	110.1
Mo2—S2—Mo1	74.46 (8)	C3—C4—H4A	110.1
C3—S3—Mo2	103.4 (3)	N2—C4—H4B	110.1
Mo1—S4—Mo2	74.95 (8)	C3—C4—H4B	110.1
C2—N1—Mo1	113.3 (6)	H4A—C4—H4B	108.4

O1—Mo1—Mo2—O2	6.2 (4)	N1—Mo1—S2—Mo2	−116.2 (4)
N1—Mo1—Mo2—O2	−108.5 (4)	S4—Mo1—S2—Mo2	−21.39 (10)
S4—Mo1—Mo2—O2	−99.7 (3)	S1—Mo1—S2—Mo2	−156.15 (9)
S2—Mo1—Mo2—O2	107.0 (3)	O2—Mo2—S3—C3	−76.3 (5)
S1—Mo1—Mo2—O2	137.7 (3)	N2—Mo2—S3—C3	13.5 (4)
O1—Mo1—Mo2—N2	−107.2 (4)	S2—Mo2—S3—C3	83.3 (4)
N1—Mo1—Mo2—N2	138.1 (4)	S4—Mo2—S3—C3	−180.0 (4)
S4—Mo1—Mo2—N2	146.9 (3)	Mo1—Mo2—S3—C3	153.8 (4)
S2—Mo1—Mo2—N2	−6.5 (3)	O1—Mo1—S4—Mo2	−91.1 (3)
S1—Mo1—Mo2—N2	24.3 (3)	N1—Mo1—S4—Mo2	173.7 (2)
O1—Mo1—Mo2—S2	−100.8 (3)	S2—Mo1—S4—Mo2	21.17 (10)
N1—Mo1—Mo2—S2	144.5 (3)	S1—Mo1—S4—Mo2	110.25 (12)
S4—Mo1—Mo2—S2	153.34 (13)	O2—Mo2—S4—Mo1	95.8 (3)
S1—Mo1—Mo2—S2	30.75 (12)	N2—Mo2—S4—Mo1	−121.7 (5)
O1—Mo1—Mo2—S4	105.9 (3)	S2—Mo2—S4—Mo1	−21.56 (10)
N1—Mo1—Mo2—S4	−8.8 (3)	S3—Mo2—S4—Mo1	−153.14 (10)
S2—Mo1—Mo2—S4	−153.34 (13)	O1—Mo1—N1—C2	72.4 (7)
S1—Mo1—Mo2—S4	−122.59 (13)	S4—Mo1—N1—C2	−177.9 (6)
O1—Mo1—Mo2—S3	139.3 (3)	S2—Mo1—N1—C2	−78.5 (8)
N1—Mo1—Mo2—S3	24.6 (3)	S1—Mo1—N1—C2	−38.0 (6)
S4—Mo1—Mo2—S3	33.46 (12)	Mo2—Mo1—N1—C2	−170.9 (5)
S2—Mo1—Mo2—S3	−119.88 (12)	O2—Mo2—N2—C4	70.5 (7)
S1—Mo1—Mo2—S3	−89.13 (12)	S2—Mo2—N2—C4	−177.7 (6)
O1—Mo1—S1—C1	−83.6 (4)	S4—Mo2—N2—C4	−73.7 (9)
N1—Mo1—S1—C1	9.7 (4)	S3—Mo2—N2—C4	−41.9 (6)
S4—Mo1—S1—C1	74.9 (4)	Mo1—Mo2—N2—C4	−172.5 (5)
S2—Mo1—S1—C1	172.2 (3)	Mo1—S1—C1—C2	15.9 (7)
Mo2—Mo1—S1—C1	148.0 (3)	Mo1—N1—C2—C1	59.9 (9)
O2—Mo2—S2—Mo1	−91.9 (3)	S1—C1—C2—N1	−46.7 (9)
N2—Mo2—S2—Mo1	175.6 (2)	Mo2—S3—C3—C4	13.0 (8)
S4—Mo2—S2—Mo1	21.20 (10)	Mo2—N2—C4—C3	62.8 (9)
S3—Mo2—S2—Mo1	108.43 (12)	S3—C3—C4—N2	−46.8 (11)
O1—Mo1—S2—Mo2	93.9 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1C···O1^{i}	0.90	2.25	3.025 (11)	144
N1—H1D···S3^{ii}	0.90	2.53	3.400 (8)	161
N2—H2C···S2^{iii}	0.90	2.81	3.704 (9)	173
N2—H2D···S1^{iv}	0.90	2.50	3.403 (8)	178

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*, −*y*+1, −*z*; (iv) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

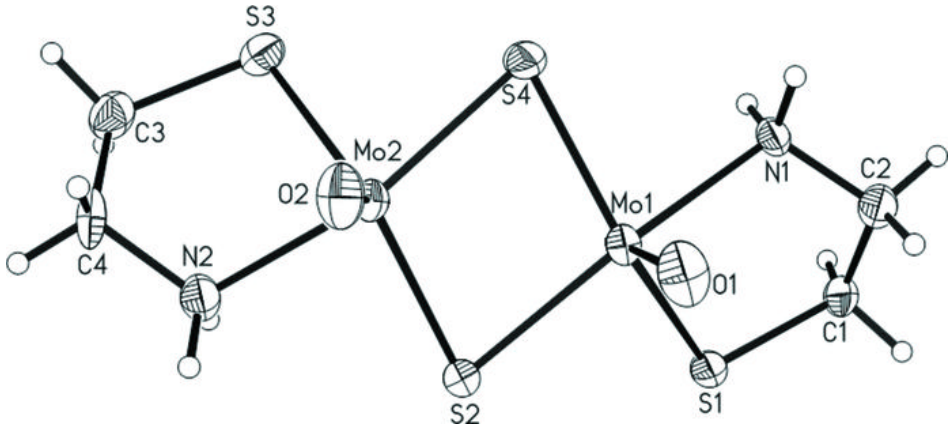

Fig. 2