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Bis[benzyl(methyl)ammonium] tetrathiomolybdate(VI)

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Bis[benzyl(methyl)ammonium] tetrathiomolybdate(VI)

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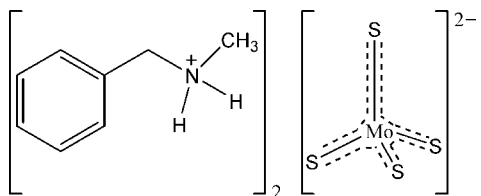
Received 7 October 2007; accepted 9 October 2007

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.050; wR factor = 0.100; data-to-parameter ratio = 17.3.

The title compound, $(\text{C}_8\text{H}_{12}\text{N})_2[\text{MoS}_4]$, was synthesized by the aqueous reaction of ammonium tetrathiomolybdate with benzyl(methyl)amine in a 1:2 molar ratio. The structure consists of a slightly distorted tetrahedral $[\text{MoS}_4]^{2-}$ dianion and two crystallographically independent benzyl(methyl)ammonium cations, with all atoms located in general positions. The cations and anions are linked by weak $\text{N}-\text{H}\cdots\text{S}$ and $\text{C}-\text{H}\cdots\text{S}$ interactions, the strength and number of which can explain the observed $\text{Mo}-\text{S}$ bond distances.

Related literature

Previous reports give details of the structural characterization of several organic ammonium tetrathiomolybdates derived from organic diamines (Srinivasan *et al.*, 2001; Srinivasan, Dhuri *et al.*, 2005; Srinivasan, Näther & Bensch, 2005), chiral amines (Srinivasan, Naik *et al.*, 2007), triamines (Srinivasan, Dhuri *et al.*, 2007), cyclic amines (Srinivasan, Näther & Bensch, 2006), a tetraamine (Srinivasan *et al.*, 2004) and a primary amine (Srinivasan, Näther *et al.*, 2006).



Experimental

Crystal data

 $(\text{C}_8\text{H}_{12}\text{N})_2[\text{MoS}_4]$ $M_r = 468.55$ Triclinic, $P\bar{1}$ $a = 7.1857$ (12) Å $b = 10.7376$ (18) Å $c = 14.881$ (2) Å $\alpha = 110.811$ (2)° $\beta = 90.608$ (3)° $\gamma = 100.504$ (3)° $V = 1051.7$ (3) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 1.02$ mm⁻¹ $T = 298$ (2) K

0.42 × 0.06 × 0.02 mm

Data collection

Bruker SMART APEX CCD
diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 2004)

 $T_{\min} = 0.901$, $T_{\max} = 0.976$

7363 measured reflections

3639 independent reflections

3015 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.035$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.100$ $S = 1.05$

3639 reflections

210 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.68$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.51$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Mo1—S1	2.1582 (13)	Mo1—S3	2.1888 (14)
Mo1—S2	2.1597 (16)	Mo1—S4	2.1908 (14)
S1—Mo1—S2	109.43 (6)	S1—Mo1—S4	109.47 (5)
S1—Mo1—S3	109.32 (6)	S2—Mo1—S4	109.01 (6)
S2—Mo1—S3	110.28 (7)	S3—Mo1—S4	109.31 (6)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
N1—H1A \cdots S3 ⁱ	0.90	2.51	3.308 (4)	149
N1—H1A \cdots S1 ⁱ	0.90	2.78	3.358 (4)	124
N1—H1B \cdots S4	0.90	2.37	3.253 (4)	168
N2—H2A \cdots S4	0.90	2.48	3.305 (5)	153
N2—H2B \cdots S4 ⁱⁱ	0.90	2.54	3.264 (5)	138
N2—H2B \cdots S2 ⁱⁱ	0.90	2.93	3.602 (5)	132
C7—H7B \cdots S3	0.97	2.92	3.727 (6)	141
C15—H15A \cdots S1 ⁱⁱⁱ	0.97	2.91	3.629 (6)	132

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2001); program(s) used to refine structure: SHELXTL; molecular graphics: DIAMOND (Brandenburg 1999); 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: NC2064).

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supplementary materials

Acta Cryst. (2007). E63, m2737-m2738 [doi:10.1107/S1600536807049562]

Bis[benzyl(methyl)ammonium] tetrathiomolybdate(VI)

B. R. Srinivasan, S. V. Girkar and P. Raghavaiah

Comment

As part of an ongoing research programme, we are investigating the synthesis and structural characterization of organic ammonium tetrathiomolybdates of the group VI metals Mo and W (Srinivasan, Naik *et al.*, 2007). In earlier work we have structurally characterized several $[\text{MoS}_4]^{2-}$ compounds derived from organic diamines (Srinivasan *et al.*, 2001; Srinivasan, Dhuri *et al.*, 2005; Srinivasan, Näther & Bensch, 2005), chiral amines (Srinivasan, Naik *et al.*, 2007) triamines (Srinivasan, Dhuri *et al.*, 2007), cyclic amines (Srinivasan, Näther & Bensch, 2006), tetraamine (Srinivasan *et al.*, 2004) and primary amine (Srinivasan, Näther *et al.*, 2006). All the organic ammonium tetrathiomolybdates exhibit several weak hydrogen bonding interactions between the organic cations and $[\text{MoS}_4]^{2-}$ anions. We have also shown that in some organic $[\text{MoS}_4]^{2-}$ compounds the organic amines are partially protonated (Srinivasan, Dhuri *et al.*, 2007). The secondary amine *N*-methyl-1-phenylmethanamine used for the synthesis of the title compound (I) is an isomer of the chiral primary amine 1-phenylethylamine used in our earlier report (Srinivasan, Naik *et al.*, 2007).

The structure of (I) consists of discrete tetrahedral $[\text{MoS}_4]^{2-}$ ions and two crystallographically independent benzyl(methyl)ammonium cations (Fig. 1) with all atoms located in general positions. The $[\text{MoS}_4]$ tetrahedron is slightly distorted with S—Mo—S angles between 109.01 (6) and 110.28 (7)° (Table 1). The Mo—S bond lengths range from 2.1582 (13) to 2.1908 (14) Å with an average value of 2.1744 Å which is comparable to the bond lengths observed in the related chiral $[\text{MoS}_4]^{2-}$ compound synthesized from the isomeric chiral primary amine (Srinivasan, Naik *et al.*, 2007). The Mo1—S1 and Mo1—S2 bond distances are indistinguishable within experimental error as also the Mo1—S3 and Mo1—S4 bonds. The weak H-bonding interactions between the cations and anions can explain the observed short and long Mo—S bond distances. A scrutiny of the structure reveals that the organic cations and tetrathiomolybdate anions are linked with the aid of several N—H⋯S and C—H⋯S hydrogen bonding interactions. Thus each $[\text{MoS}_4]^{2-}$ is hydrogen bonded to five different organic cations with the aid of six N—H⋯S bonds and two weak C—H⋯S interactions (Fig. 2). An examination of the surroundings of the cations reveals that one organic cation (N1) is H-bonded to two different $[\text{MoS}_4]^{2-}$ ions while the second organic cation (N2) is surrounded by three different $[\text{MoS}_4]^{2-}$ ions (Table 2). One H atom on each N atom functions as a singly shared donor with the other functioning as a bifurcated donor. A benzylic H atom from each unique cation is involved in C—H⋯S interaction. S4 atom which makes the longest Mo—S bond at 2.1908 (14) Å is involved in three N—H⋯S bonds, two of which are singly shared. S4 also makes the shortest singly shared N—H⋯S bond at 2.37 Å, which can explain the elongation of this bond. In contrast, S1 atom involved in the shortest Mo—S bond makes a bifurcated N—H⋯S bond at a longer S⋯H distance accompanied by a small NH—S angle. S1 also makes a very weak C—H⋯S contact. The observed difference Δ between the longest and the shortest Mo—S bond of 0.0326 Å in (I) is shorter than the Δ value of 0.0422 Å in the tetrathiomolybdate compound containing the *R*-form of the monoprotonated isomeric chiral primary amine 1-phenylethylamine (Srinivasan, Naik *et al.*, 2007).

supplementary materials

Experimental

(NH₄)₂[MoS₄] (520 mg, 2 mmol) was dissolved in water (15 ml) containing a few drops of liquor ammonia. To this mixture *N*-methyl-1-phenylmethanamine (1 ml) was added and the reaction mixture filtered and left in the refrigerator for crystallization. After two days small needles of the title compound separated. The crystals were filtered, washed with ice-cold water (2 ml), followed by 2-propanol (10 ml) and diethyl ether (10 ml) and dried. Yield: 60%.

Refinement

The H atoms were positioned with idealized geometry (C—H = 0.93 (aromatic), 0.96 (methyl) and 0.97 (benzitic) Å and N—H = 0.90 Å) and were refined using a riding model, with $U_{\text{iso}}(\text{H})$ fixed at $1.5U_{\text{eq}}(\text{CH}_3)$ and $1.2U_{\text{eq}}(\text{NH}_2, \text{benzitic, aromatic})$.

Figures

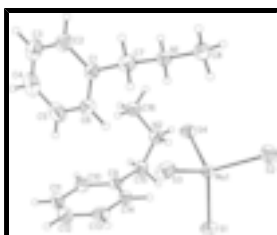


Fig. 1. The crystal structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

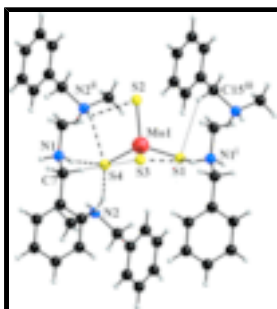


Fig. 2. A view of the surroundings of the [MoS₄]²⁻ anion showing its linking to five different organic cations with the aid of six N—H...S and two C—H...S interactions. N—H...S and C—H...S interactions are shown as dashed lines and dotted lines respectively. Symmetry codes: (i) $x + 1, y, z$ (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x, -y + 1, z - 1$;

Bis[benzyl(methyl)ammonium] tetrathiomolybdate(VI)

Crystal data

(C₈H₁₂N)₂[MoS₄]

$M_r = 468.55$

Triclinic, $P\bar{1}$

Hall symbol: $-P 1$

$a = 7.1857$ (12) Å

$b = 10.7376$ (18) Å

$c = 14.881$ (2) Å

$\alpha = 110.811$ (2)°

$Z = 2$

$F_{000} = 480$

$D_x = 1.480$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4674 reflections

$\theta = 2.9$ – 25.9°

$\mu = 1.02$ mm⁻¹

$T = 298$ (2) K

$\beta = 90.608 (3)^\circ$
 $\gamma = 100.504 (3)^\circ$
 $V = 1051.7 (3) \text{ \AA}^3$

Thin needle, red
 $0.42 \times 0.06 \times 0.02 \text{ mm}$

Data collection

Bruker SMART APEX CCD diffractometer
 Radiation source: fine-focus sealed tube
 Monochromator: graphite
 $T = 298(2) \text{ K}$
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.901, T_{\max} = 0.976$
 7363 measured reflections

3639 independent reflections
 3015 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 25.0^\circ$
 $\theta_{\text{min}} = 1.5^\circ$
 $h = -8 \rightarrow 8$
 $k = -12 \rightarrow 11$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.100$
 $S = 1.05$
 3639 reflections
 210 parameters
 Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 0.9757P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.68 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.51 \text{ e \AA}^{-3}$
 Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5906 (7)	0.4755 (6)	0.1061 (3)	0.0448 (13)
C2	0.7203 (8)	0.4231 (7)	0.0439 (4)	0.0575 (15)

supplementary materials

H2	0.8260	0.4816	0.0361	0.069*
C3	0.6971 (11)	0.2869 (8)	-0.0068 (4)	0.0714 (19)
H3	0.7854	0.2531	-0.0493	0.086*
C4	0.5436 (12)	0.2006 (7)	0.0055 (5)	0.077 (2)
H4	0.5279	0.1075	-0.0283	0.092*
C5	0.4126 (10)	0.2507 (7)	0.0674 (5)	0.075 (2)
H5	0.3075	0.1918	0.0753	0.090*
C6	0.4366 (8)	0.3879 (7)	0.1177 (4)	0.0592 (16)
H6	0.3478	0.4217	0.1600	0.071*
C7	0.6150 (8)	0.6270 (6)	0.1585 (4)	0.0510 (14)
H7A	0.7090	0.6734	0.1287	0.061*
H7B	0.4957	0.6545	0.1521	0.061*
C8	0.6958 (8)	0.8155 (5)	0.3161 (4)	0.0573 (15)
H8A	0.7718	0.8658	0.2829	0.086*
H8B	0.7565	0.8372	0.3791	0.086*
H8C	0.5728	0.8392	0.3223	0.086*
C9	0.1099 (7)	0.1225 (5)	0.2958 (4)	0.0401 (12)
C10	0.0895 (8)	0.0024 (6)	0.2183 (4)	0.0528 (14)
H10	0.1464	-0.0669	0.2221	0.063*
C11	-0.0153 (9)	-0.0155 (7)	0.1348 (5)	0.0677 (18)
H11	-0.0323	-0.0979	0.0831	0.081*
C12	-0.0937 (9)	0.0877 (7)	0.1282 (4)	0.0682 (18)
H12	-0.1609	0.0765	0.0712	0.082*
C13	-0.0739 (8)	0.2073 (6)	0.2050 (4)	0.0583 (16)
H13	-0.1285	0.2773	0.2006	0.070*
C14	0.0269 (8)	0.2240 (5)	0.2887 (4)	0.0494 (14)
H14	0.0389	0.3052	0.3411	0.059*
C15	0.2213 (8)	0.1417 (6)	0.3879 (4)	0.0511 (14)
H15A	0.1415	0.1651	0.4412	0.061*
H15B	0.2585	0.0571	0.3826	0.061*
C16	0.5316 (8)	0.2289 (7)	0.3349 (4)	0.0640 (17)
H16A	0.5654	0.1422	0.3224	0.096*
H16B	0.6432	0.2994	0.3579	0.096*
H16C	0.4767	0.2309	0.2764	0.096*
Mo1	0.16547 (6)	0.66475 (4)	0.38214 (3)	0.03200 (14)
N1	0.6748 (6)	0.6684 (4)	0.2609 (3)	0.0443 (10)
H1A	0.7867	0.6441	0.2662	0.053*
H1B	0.5892	0.6228	0.2875	0.053*
N2	0.3937 (6)	0.2507 (5)	0.4080 (3)	0.0563 (13)
H2A	0.3565	0.3291	0.4152	0.068*
H2B	0.4529	0.2612	0.4645	0.068*
S1	-0.10243 (18)	0.57324 (14)	0.41788 (10)	0.0458 (3)
S2	0.2566 (2)	0.86869 (15)	0.48604 (12)	0.0619 (4)
S3	0.1321 (2)	0.66870 (17)	0.23687 (10)	0.0552 (4)
S4	0.37925 (19)	0.54486 (14)	0.38649 (10)	0.0466 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.045 (3)	0.060 (4)	0.033 (3)	0.018 (3)	-0.001 (2)	0.018 (3)
C2	0.056 (4)	0.068 (4)	0.051 (3)	0.018 (3)	0.006 (3)	0.023 (3)
C3	0.087 (5)	0.083 (5)	0.050 (4)	0.043 (4)	0.008 (4)	0.020 (4)
C4	0.118 (7)	0.055 (4)	0.058 (4)	0.030 (5)	-0.016 (4)	0.014 (4)
C5	0.080 (5)	0.067 (5)	0.064 (4)	-0.006 (4)	-0.009 (4)	0.017 (4)
C6	0.051 (4)	0.070 (5)	0.049 (3)	0.008 (3)	0.008 (3)	0.014 (3)
C7	0.052 (4)	0.061 (4)	0.047 (3)	0.015 (3)	0.001 (3)	0.026 (3)
C8	0.058 (4)	0.044 (4)	0.065 (4)	0.008 (3)	0.010 (3)	0.014 (3)
C9	0.034 (3)	0.044 (3)	0.043 (3)	0.007 (2)	0.008 (2)	0.019 (3)
C10	0.052 (4)	0.038 (3)	0.065 (4)	0.013 (3)	0.005 (3)	0.013 (3)
C11	0.069 (4)	0.055 (4)	0.056 (4)	0.010 (3)	-0.004 (3)	-0.006 (3)
C12	0.061 (4)	0.089 (5)	0.049 (4)	0.019 (4)	-0.008 (3)	0.017 (4)
C13	0.046 (4)	0.063 (4)	0.073 (4)	0.022 (3)	0.003 (3)	0.028 (4)
C14	0.047 (3)	0.038 (3)	0.056 (3)	0.009 (3)	-0.002 (3)	0.008 (3)
C15	0.058 (4)	0.054 (4)	0.045 (3)	0.012 (3)	0.007 (3)	0.022 (3)
C16	0.041 (3)	0.083 (5)	0.070 (4)	0.009 (3)	0.004 (3)	0.031 (4)
Mo1	0.0265 (2)	0.0333 (3)	0.0379 (2)	0.00674 (17)	0.00297 (17)	0.01470 (19)
N1	0.041 (3)	0.044 (3)	0.047 (3)	0.009 (2)	0.002 (2)	0.014 (2)
N2	0.051 (3)	0.068 (3)	0.046 (3)	0.017 (3)	-0.005 (2)	0.015 (2)
S1	0.0340 (7)	0.0519 (9)	0.0577 (8)	0.0090 (6)	0.0102 (6)	0.0271 (7)
S2	0.0690 (11)	0.0358 (8)	0.0698 (10)	0.0050 (8)	-0.0012 (8)	0.0087 (8)
S3	0.0500 (9)	0.0818 (12)	0.0498 (8)	0.0243 (8)	0.0121 (7)	0.0375 (8)
S4	0.0360 (8)	0.0492 (9)	0.0594 (9)	0.0137 (6)	0.0021 (6)	0.0230 (7)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.371 (7)	C10—H10	0.9300
C1—C6	1.372 (7)	C11—C12	1.363 (9)
C1—C7	1.509 (7)	C11—H11	0.9300
C2—C3	1.364 (8)	C12—C13	1.365 (8)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.364 (9)	C13—C14	1.372 (7)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.370 (9)	C14—H14	0.9300
C4—H4	0.9300	C15—N2	1.487 (7)
C5—C6	1.372 (8)	C15—H15A	0.9700
C5—H5	0.9300	C15—H15B	0.9700
C6—H6	0.9300	C16—N2	1.465 (7)
C7—N1	1.463 (6)	C16—H16A	0.9600
C7—H7A	0.9700	C16—H16B	0.9600
C7—H7B	0.9700	C16—H16C	0.9600
C8—N1	1.477 (6)	Mo1—S1	2.1582 (13)
C8—H8A	0.9600	Mo1—S2	2.1597 (16)
C8—H8B	0.9600	Mo1—S3	2.1888 (14)
C8—H8C	0.9600	Mo1—S4	2.1908 (14)

supplementary materials

C9—C14	1.369 (7)	N1—H1A	0.9000
C9—C10	1.372 (7)	N1—H1B	0.9000
C9—C15	1.509 (7)	N2—H2A	0.9000
C10—C11	1.380 (8)	N2—H2B	0.9000
C2—C1—C6	118.7 (6)	C11—C12—C13	120.2 (6)
C2—C1—C7	120.4 (5)	C11—C12—H12	119.9
C6—C1—C7	120.9 (5)	C13—C12—H12	119.9
C3—C2—C1	121.3 (6)	C12—C13—C14	119.8 (6)
C3—C2—H2	119.4	C12—C13—H13	120.1
C1—C2—H2	119.4	C14—C13—H13	120.1
C4—C3—C2	119.6 (6)	C9—C14—C13	120.7 (5)
C4—C3—H3	120.2	C9—C14—H14	119.6
C2—C3—H3	120.2	C13—C14—H14	119.6
C3—C4—C5	120.1 (7)	N2—C15—C9	111.2 (4)
C3—C4—H4	119.9	N2—C15—H15A	109.4
C5—C4—H4	119.9	C9—C15—H15A	109.4
C4—C5—C6	119.9 (7)	N2—C15—H15B	109.4
C4—C5—H5	120.0	C9—C15—H15B	109.4
C6—C5—H5	120.0	H15A—C15—H15B	108.0
C5—C6—C1	120.4 (6)	N2—C16—H16A	109.5
C5—C6—H6	119.8	N2—C16—H16B	109.5
C1—C6—H6	119.8	H16A—C16—H16B	109.5
N1—C7—C1	112.2 (4)	N2—C16—H16C	109.5
N1—C7—H7A	109.2	H16A—C16—H16C	109.5
C1—C7—H7A	109.2	H16B—C16—H16C	109.5
N1—C7—H7B	109.2	S1—Mo1—S2	109.43 (6)
C1—C7—H7B	109.2	S1—Mo1—S3	109.32 (6)
H7A—C7—H7B	107.9	S2—Mo1—S3	110.28 (7)
N1—C8—H8A	109.5	S1—Mo1—S4	109.47 (5)
N1—C8—H8B	109.5	S2—Mo1—S4	109.01 (6)
H8A—C8—H8B	109.5	S3—Mo1—S4	109.31 (6)
N1—C8—H8C	109.5	C7—N1—C8	114.4 (4)
H8A—C8—H8C	109.5	C7—N1—H1A	108.7
H8B—C8—H8C	109.5	C8—N1—H1A	108.7
C14—C9—C10	119.2 (5)	C7—N1—H1B	108.7
C14—C9—C15	120.5 (5)	C8—N1—H1B	108.7
C10—C9—C15	120.3 (5)	H1A—N1—H1B	107.6
C9—C10—C11	120.1 (5)	C16—N2—C15	116.1 (5)
C9—C10—H10	119.9	C16—N2—H2A	108.3
C11—C10—H10	119.9	C15—N2—H2A	108.3
C12—C11—C10	119.9 (6)	C16—N2—H2B	108.3
C12—C11—H11	120.0	C15—N2—H2B	108.3
C10—C11—H11	120.0	H2A—N2—H2B	107.4

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots S3^i$	0.90	2.51	3.308 (4)	149

supplementary materials

N1—H1A…S1 ⁱ	0.90	2.78	3.358 (4)	124
N1—H1B…S4	0.90	2.37	3.253 (4)	168
N2—H2A…S4	0.90	2.48	3.305 (5)	153
N2—H2B…S4 ⁱⁱ	0.90	2.54	3.264 (5)	138
N2—H2B…S2 ⁱⁱ	0.90	2.93	3.602 (5)	132
C7—H7B…S3	0.97	2.92	3.727 (6)	141
C15—H15A…S1 ⁱⁱⁱ	0.97	2.91	3.629 (6)	132

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

Fig. 1

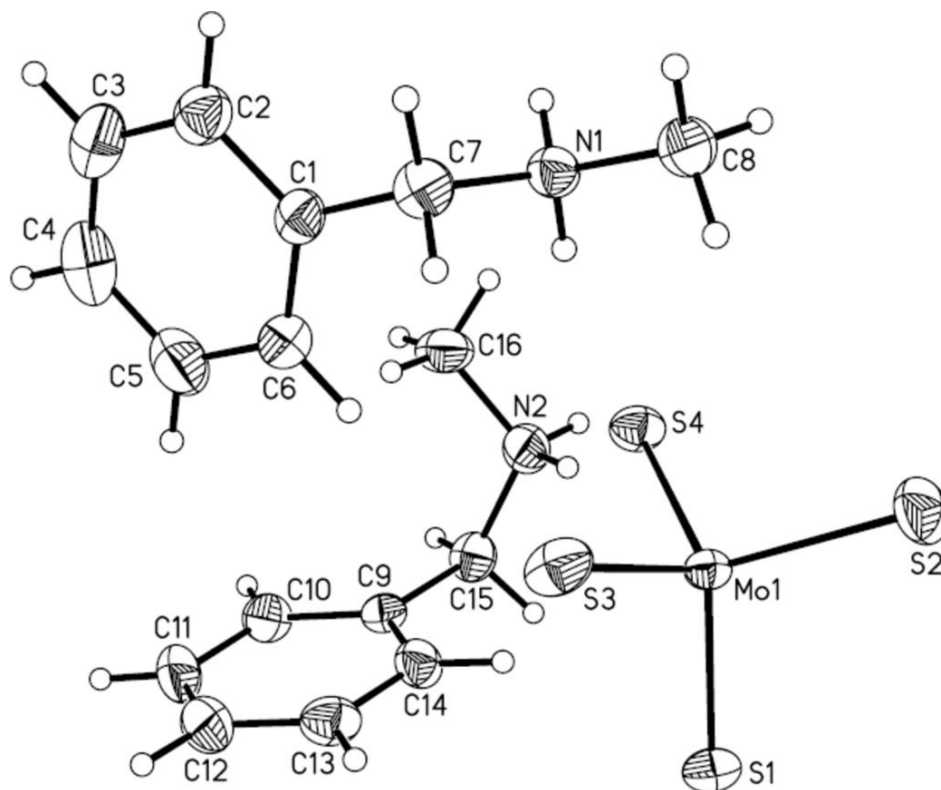


Fig. 2

