# metal-organic compounds

# Bis(isopropylammonium) tetrasulfido-molybdate(VI)

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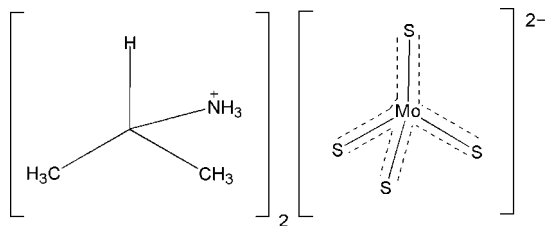
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The title compound, $(C_3H_{10}N)_2[MoS_4]$, was synthesized by passing a rapid stream of $H_2S$ into an aqueous isopropylamine solution of molybdic acid. The title compound is isotypic with the corresponding W analogue $(C_3H_{10}N)_2[WS_4]$; its structure consists of a slightly distorted tetrahedral $[MoS_4]^{2-}$ dianion and two crystallographically independent isopropylammonium cations, with all atoms located in general positions. The cations and anion are linked by weak $N-H\cdots S$ and $C-H\cdots S$ interactions, the strength and number of which can explain the observed Mo—S bond distances.

## Related literature

Previous reports give details of the structural characterization of several organic ammonium tetrasulfidomolybdates derived from chiral amines (Srinivasan, Naik *et al.*, 2007), diamines (Srinivasan *et al.*, 2001; Srinivasan, Dhuri *et al.*, 2005; Srinivasan, Näther & Bensch 2005), triamines (Srinivasan, Dhuri *et al.*, 2007), cyclic amines (Srinivasan, Näther & Bensch 2006), a tetraamine (Srinivasan *et al.*, 2004), a primary amine (Srinivasan, Näther, Naik & Bensch 2006) and a secondary amine (Srinivasan, Girkar & Raghavaiah 2007). The title compound is isotypic with the corresponding W analogue $(C_3H_{10}N)_2[WS_4]$ (Srinivasan, Näther, Dhuri & Bensch 2006).



## Experimental

### Crystal data

| | |
|---|---|
| $(C_3H_{10}N)_2[MoS_4]$ | $V$ = 2937.3 (4) Å³ |
| $M_r$ = 344.42 | $Z$ = 8 |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation |
| $a$ = 20.2640 (14) Å | $\mu$ = 1.43 mm⁻¹ |
| $b$ = 13.9118 (12) Å | $T$ = 170 (2) K |
| $c$ = 11.0933 (8) Å | $0.13 \times 0.1 \times 0.08$ mm |
| $\beta$ = 110.076 (8)° | |

### Data collection

| | |
|---|---|
| Stoe IPDS 1 diffractometer | 10806 measured reflections |
| Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998) | 3134 independent reflections |
| | 2716 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.756, $T_{max}$ = 0.830 | $R_{int}$ = 0.025 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.026 | 125 parameters |
| $wR(F^2)$ = 0.071 | H-atom parameters constrained |
| $S$ = 1.03 | $\Delta\rho_{max}$ = 0.64 e Å⁻³ |
| 3134 reflections | $\Delta\rho_{min}$ = −0.72 e Å⁻³ |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Mo—S2 | 2.1695 (6) | Mo—S1 | 2.2023 (6) |
| Mo—S3 | 2.1769 (5) | Mo—S4 | 2.2085 (6) |
| | | | |
| S2—Mo—S3 | 109.08 (2) | S2—Mo—S4 | 109.01 (2) |
| S2—Mo—S1 | 109.02 (3) | S3—Mo—S4 | 109.78 (2) |
| S3—Mo—S1 | 109.51 (2) | S1—Mo—S4 | 110.42 (2) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| N1—H1N1⋯S4 | 0.91 | 2.43 | 3.310 (2) | 162 |
| N1—H2N1⋯S4ⁱ | 0.91 | 2.47 | 3.359 (2) | 165 |
| N1—H3N1⋯S1ⁱⁱ | 0.91 | 2.54 | 3.4297 (19) | 165 |
| N1—H3N1⋯S3ⁱⁱ | 0.91 | 2.85 | 3.3019 (19) | 112 |
| N2—H1N2⋯S3ⁱⁱⁱ | 0.91 | 2.58 | 3.376 (2) | 147 |
| N2—H1N2⋯S2ⁱⁱⁱ | 0.91 | 2.92 | 3.580 (2) | 131 |
| N2—H2N2⋯S4 | 0.91 | 2.43 | 3.309 (2) | 164 |
| N2—H3N2⋯S1ⁱ | 0.91 | 2.50 | 3.393 (2) | 169 |
| C2—H2A⋯S4ⁱ | 0.98 | 2.97 | 3.759 (3) | 139 |
| C5—H5B⋯S3ⁱⁱ | 0.98 | 2.98 | 3.639 (6) | 126 |

Symmetry codes: (i) $x, -y+1, z-\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *IPDS Program Package* (Stoe & Cie, 1998); cell refinement: *IPDS Program Package*; data reduction: *IPDS Program Package*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *CIFTAB* in *SHELXTL* (Bruker, 1998).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2062).

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**supplementary materials**

## Bis(isopropylammonium) tetrasulfidomolybdate(VI)

### B. R. Srinivasan, C. Näther, A. R. Naik and W. Bensch

### Comment

As part of an ongoing research programme, we are investigating the synthesis and structural aspects of organic ammonium tetrasulfidometalates of the group VI metals Mo and W (Srinivasan, Naik *et al.*, 2007). In earlier work we have structurally characterized several $[MoS_4]^{2-}$ compounds linked to organic cations derived from chiral amines (Srinivasan, Naik *et al.*, 2007), diamines (Srinivasan *et al.*, 2001; Srinivasan, Dhuri *et al.*, 2005; Srinivasan, Näther & Bensch 2005), triamines (Srinivasan, Dhuri *et al.*, 2007), cyclic amines (Srinivasan, Näther & Bensch 2006), a tetraamine (Srinivasan *et al.*, 2004), a primary amine (Srinivasan, Näther, Naik & Bensch 2006) and a secondary amine (Srinivasan, Girkar & Raghavaiah 2007). All the organic ammonium tetrasulfidomolybdates exhibit several weak hydrogen bonding interactions between the organic cations and $[MoS_4]^{2-}$ anions. We have shown that in some organic $[MoS_4]^{2-}$ compounds the organic amines are partially protonated (Srinivasan, Dhuri *et al.*, 2007). In the present work, we have employed isopropylamine ($ipNH_2$) for the synthesis of the title compound, which is isostructural with the corresponding W compound $(C_3H_{10}N)_2[WS_4]$ (Srinivasan, Näther, Dhuri & Bensch 2006).

The structure of the title compound consists of discrete tetrahedral $[MoS_4]^{2-}$ ions and two crystallographically independent isopropylammonium cations $(ipNH_2)^+$ (Fig. 1), with all atoms located in general positions. The geometric parameters of the organic cations are in agreement with those reported for the analogous $[WS_4]^{2-}$ compound. The MoS$_4$ tetrahedron is slightly distorted with S—Mo—S angles between 109.01 (2) and 110.42 (2) ° (Table 1). The Mo—S bond distances range from 2.1695 (6) to 2.2085 (6) Å with an average value of 2.1893 Å and are comparable to the bond lengths observed in several reported tetrathiomolybdates (Srinivasan, Dhuri *et al.*, 2007). Two of the Mo—S bond lengths are shorter than the average Mo—S distance while the other two are longer. 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 (Table 2). Thus each $[MoS_4]^{2-}$ is hydrogen bonded to seven different organic cations with the aid of eight 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 three different $[MoS_4]^{2-}$ ions while the second organic cation (N2) is surrounded by four different $[MoS_4]^{2-}$ ions (Table 2). One H atom on each N atom functions as a bifurcated donor with the other two functioning as singly shared donors. One H atom attached to a methyl group from each unique cation is involved in a weak C—H⋯S interaction. S4 atom which makes the longest Mo—S bond at 2.2085 (6) Å is involved in three singly shared N—H⋯S bonds. S4 also makes the shortest singly shared N—H⋯S bond at 2.43 Å, which can explain the elongation of this bond. In contrast, S2 atom involved in the shortest Mo—S bond at 2.1695 (6) Å makes a bifurcated N—H⋯S bond at a longer S⋯H distance accompanied by a small NH—S angle. As a result of the H-bonding interactions, the cations and anions are organized such that the organic ammonium ions always point towards the S atoms of $[MoS_4]^{2-}$ as is evident in the observed sequence ⋯$(ipNH_2)^+$ ⋯ $[MoS_4]^{2-}$⋯$(ipNH_2)^+$ $(ipNH_2)^+$ ⋯ $[MoS_4]^{2-}$ ⋯$(ipNH_2)^+$ and so on when viewed along the *c* axis (Fig. 3). The

# supplementary materials

observed difference Δ between the longest and the shortest Mo—S bond of 0.0390 Å in the title compound is quite longer than the Δ value of 0.0334 Å in the isostructural tetrathiotungstate compound $(C_3H_{10}N)_2[WS_4]$ (Srinivasan *et al.*, 2007*a*).

## Experimental

A rapid stream of $H_2S$ gas was passed into a solution containing molybdic acid (3 g) dissolved in water (30 ml) and isopropyl amine (10 ml). After 30 min when crystals begin to appear, the gas passing was stopped and the deep red reaction mixture was filtered. The filtrate was left aside for crystallization. After 3 to 4 h the crystalline product was filtered, washed with a little ice-cold water (2 ml), followed by 2-propanol (20 ml) and diethyl ether (10 ml) and dried to obtain 3.2 g of the title compound.

## Refinement

All H atoms were located in difference map but were positioned with idealized geometry ((CH$_3$ and NH$_3$ allowed to rotate but not to tip) with 1.00 Å (C—H), 0.98 Å (methyl) and N—H = 0.91 Å) and were refined using a riding model, with $U_{iso}$(H) fixed at $1.5U_{eq}$(CH$_3$ and NH$_3$) and $1.5U_{eq}$(NH$_3$).
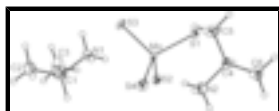
## Figures



Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.
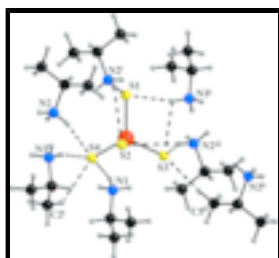


Fig. 2. A view of the surroundings of the $[MoS_4]^{2-}$ anion showing its linking to seven different (ipNH$_2$)$^+$ cations with the aid of eight N—H···S and two C—H···S interactions, shown as broken lines. Symmetry codes: (i) $x$, $-y + 1$, $z - 1/2$ (ii) $-x + 1/2$, $-y + 3/2$, $-z + 1$; (iii) $-x + 1/2$, $y - 1/2$, $-z + 3/2$;
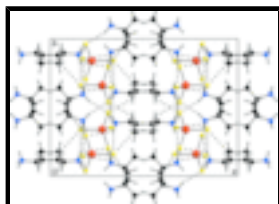


Fig. 3. A view along $c$ axis of the crystallographic packing of the title compound showing the organization of the organic cations and $[MoS_4]^{2-}$ anions. H-bonds are shown as broken lines.

## Bis(isopropylammonium) tetrasulfidomolybdate(VI)

*Crystal data*

| | |
|---|---|
| $(C_3H_{10}N)_2[MoS_4]$ | $F_{000} = 1408$ |
| $M_r = 344.42$ | $D_x = 1.558$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |

Hall symbol: -C 2yc

$a = 20.2640\,(14)$ Å

$b = 13.9118\,(12)$ Å

$c = 11.0933\,(8)$ Å

$\beta = 110.076\,(8)°$

$V = 2937.3\,(4)$ Å$^3$

$Z = 8$

Cell parameters from 8000 reflections

$\theta = 12.5–28.0°$

$\mu = 1.43$ mm$^{-1}$

$T = 170\,(2)$ K

Needle, red

$0.13 \times 0.1 \times 0.08$ mm

*Data collection*

STOE IPDS 1 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 170(2)$ K

Phi Scan scans

Absorption correction: numerical (X-SHAPE; Stoe & Cie, 1998)

$T_{min} = 0.756$, $T_{max} = 0.830$

10806 measured reflections

3134 independent reflections

2716 reflections with $I > 2\sigma(I)$

$R_{int} = 0.025$

$\theta_{max} = 27.1°$

$\theta_{min} = 2.4°$

$h = -23 \rightarrow 25$

$k = -17 \rightarrow 17$

$l = -13 \rightarrow 14$

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.071$

$S = 1.03$

3134 reflections

125 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

$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.8301P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.64$ e Å$^{-3}$

$\Delta\rho_{min} = -0.72$ e Å$^{-3}$

Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00132 (18)

*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 $F^2 > 2sigma(F^2)$ 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 ($Å^2$)*

|  | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Mo | 0.277534 (8) | 0.660872 (11) | 0.763180 (16) | 0.01395 (9) |
| S1 | 0.16255 (3) | 0.66070 (4) | 0.71330 (6) | 0.02148 (14) |
| S2 | 0.32646 (3) | 0.64606 (4) | 0.96951 (5) | 0.02172 (14) |
| S3 | 0.31057 (3) | 0.79621 (4) | 0.70333 (5) | 0.02302 (14) |
| S4 | 0.31079 (3) | 0.53985 (4) | 0.66851 (6) | 0.02458 (14) |
| N1 | 0.34948 (9) | 0.61362 (13) | 0.41696 (19) | 0.0219 (4) |
| H1N1 | 0.3314 | 0.6038 | 0.4805 | 0.033* |
| H2N1 | 0.3318 | 0.5690 | 0.3541 | 0.033* |
| H3N1 | 0.3376 | 0.6735 | 0.3835 | 0.033* |
| C1 | 0.42792 (12) | 0.60477 (18) | 0.4708 (3) | 0.0292 (6) |
| H1 | 0.4404 | 0.5389 | 0.5075 | 0.035* |
| C2 | 0.45832 (14) | 0.61919 (19) | 0.3658 (3) | 0.0346 (6) |
| H2A | 0.4421 | 0.5676 | 0.3023 | 0.052* |
| H2B | 0.5097 | 0.6180 | 0.4027 | 0.052* |
| H2C | 0.4429 | 0.6814 | 0.3241 | 0.052* |
| C3 | 0.45457 (18) | 0.6778 (3) | 0.5776 (3) | 0.0557 (10) |
| H3A | 0.4450 | 0.7428 | 0.5417 | 0.084* |
| H3B | 0.5053 | 0.6695 | 0.6204 | 0.084* |
| H3C | 0.4306 | 0.6684 | 0.6400 | 0.084* |
| N2 | 0.17411 (10) | 0.40310 (14) | 0.51513 (19) | 0.0247 (4) |
| H1N2 | 0.1828 | 0.3534 | 0.5719 | 0.037* |
| H2N2 | 0.2062 | 0.4506 | 0.5478 | 0.037* |
| H3N2 | 0.1773 | 0.3820 | 0.4397 | 0.037* |
| C4 | 0.10154 (11) | 0.44146 (15) | 0.4922 (2) | 0.0197 (4) |
| H4 | 0.0987 | 0.4646 | 0.5756 | 0.024* |
| C5 | 0.08879 (15) | 0.52564 (17) | 0.4005 (3) | 0.0323 (6) |
| H5A | 0.0933 | 0.5043 | 0.3195 | 0.048* |
| H5B | 0.1234 | 0.5761 | 0.4386 | 0.048* |
| H5C | 0.0414 | 0.5510 | 0.3842 | 0.048* |
| C6 | 0.04982 (13) | 0.35982 (18) | 0.4430 (3) | 0.0307 (6) |
| H6A | 0.0022 | 0.3828 | 0.4303 | 0.046* |
| H6B | 0.0616 | 0.3073 | 0.5057 | 0.046* |
| H6C | 0.0522 | 0.3366 | 0.3612 | 0.046* |

*Atomic displacement parameters ($Å^2$)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Mo | 0.01492 (12) | 0.01177 (12) | 0.01591 (12) | −0.00135 (6) | 0.00626 (8) | 0.00189 (6) |
| S1 | 0.0147 (3) | 0.0242 (3) | 0.0243 (3) | −0.00166 (18) | 0.0051 (2) | 0.0059 (2) |
| S2 | 0.0215 (3) | 0.0247 (3) | 0.0167 (3) | −0.0033 (2) | 0.0038 (2) | 0.00321 (19) |
| S3 | 0.0266 (3) | 0.0171 (3) | 0.0275 (3) | −0.0053 (2) | 0.0121 (2) | 0.0054 (2) |
| S4 | 0.0337 (3) | 0.0184 (3) | 0.0257 (3) | 0.0004 (2) | 0.0154 (3) | −0.0027 (2) |
| N1 | 0.0182 (9) | 0.0205 (9) | 0.0258 (10) | −0.0003 (7) | 0.0060 (8) | 0.0033 (7) |
| C1 | 0.0181 (11) | 0.0290 (12) | 0.0371 (14) | 0.0021 (9) | 0.0049 (10) | 0.0151 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0260 (12) | 0.0357 (14) | 0.0461 (16) | −0.0012 (10) | 0.0174 (12) | −0.0046 (12) |
| C3 | 0.0378 (17) | 0.103 (3) | 0.0258 (14) | −0.0309 (17) | 0.0098 (13) | −0.0058 (16) |
| N2 | 0.0184 (9) | 0.0315 (10) | 0.0242 (10) | −0.0023 (8) | 0.0073 (8) | 0.0044 (8) |
| C4 | 0.0190 (10) | 0.0219 (10) | 0.0211 (10) | −0.0015 (8) | 0.0107 (9) | −0.0011 (8) |
| C5 | 0.0462 (15) | 0.0203 (11) | 0.0348 (13) | 0.0051 (10) | 0.0195 (12) | 0.0037 (10) |
| C6 | 0.0205 (11) | 0.0278 (12) | 0.0444 (15) | −0.0041 (9) | 0.0118 (11) | 0.0000 (11) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Mo—S2 | 2.1695 (6) | C3—H3B | 0.9800 |
| Mo—S3 | 2.1769 (5) | C3—H3C | 0.9800 |
| Mo—S1 | 2.2023 (6) | N2—C4 | 1.502 (3) |
| Mo—S4 | 2.2085 (6) | N2—H1N2 | 0.9100 |
| N1—C1 | 1.499 (3) | N2—H2N2 | 0.9100 |
| N1—H1N1 | 0.9100 | N2—H3N2 | 0.9100 |
| N1—H2N1 | 0.9100 | C4—C5 | 1.514 (3) |
| N1—H3N1 | 0.9100 | C4—C6 | 1.514 (3) |
| C1—C2 | 1.506 (4) | C4—H4 | 1.0000 |
| C1—C3 | 1.513 (4) | C5—H5A | 0.9800 |
| C1—H1 | 1.0000 | C5—H5B | 0.9800 |
| C2—H2A | 0.9800 | C5—H5C | 0.9800 |
| C2—H2B | 0.9800 | C6—H6A | 0.9800 |
| C2—H2C | 0.9800 | C6—H6B | 0.9800 |
| C3—H3A | 0.9800 | C6—H6C | 0.9800 |
| | | | |
| S2—Mo—S3 | 109.08 (2) | C1—C3—H3C | 109.5 |
| S2—Mo—S1 | 109.02 (3) | H3A—C3—H3C | 109.5 |
| S3—Mo—S1 | 109.51 (2) | H3B—C3—H3C | 109.5 |
| S2—Mo—S4 | 109.01 (2) | C4—N2—H1N2 | 109.5 |
| S3—Mo—S4 | 109.78 (2) | C4—N2—H2N2 | 109.5 |
| S1—Mo—S4 | 110.42 (2) | H1N2—N2—H2N2 | 109.5 |
| C1—N1—H1N1 | 109.5 | C4—N2—H3N2 | 109.5 |
| C1—N1—H2N1 | 109.5 | H1N2—N2—H3N2 | 109.5 |
| H1N1—N1—H2N1 | 109.5 | H2N2—N2—H3N2 | 109.5 |
| C1—N1—H3N1 | 109.5 | N2—C4—C5 | 108.67 (19) |
| H1N1—N1—H3N1 | 109.5 | N2—C4—C6 | 108.12 (18) |
| H2N1—N1—H3N1 | 109.5 | C5—C4—C6 | 113.6 (2) |
| N1—C1—C2 | 109.9 (2) | N2—C4—H4 | 108.8 |
| N1—C1—C3 | 107.5 (2) | C5—C4—H4 | 108.8 |
| C2—C1—C3 | 112.7 (2) | C6—C4—H4 | 108.8 |
| N1—C1—H1 | 108.9 | C4—C5—H5A | 109.5 |
| C2—C1—H1 | 108.9 | C4—C5—H5B | 109.5 |
| C3—C1—H1 | 108.9 | H5A—C5—H5B | 109.5 |
| C1—C2—H2A | 109.5 | C4—C5—H5C | 109.5 |
| C1—C2—H2B | 109.5 | H5A—C5—H5C | 109.5 |
| H2A—C2—H2B | 109.5 | H5B—C5—H5C | 109.5 |
| C1—C2—H2C | 109.5 | C4—C6—H6A | 109.5 |
| H2A—C2—H2C | 109.5 | C4—C6—H6B | 109.5 |
| H2B—C2—H2C | 109.5 | H6A—C6—H6B | 109.5 |
| C1—C3—H3A | 109.5 | C4—C6—H6C | 109.5 |

| C1—C3—H3B | 109.5 | H6A—C6—H6C | 109.5 |
| H3A—C3—H3B | 109.5 | H6B—C6—H6C | 109.5 |

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
| --- | --- | --- | --- | --- |
| N1—H1N1···S4 | 0.91 | 2.43 | 3.310 (2) | 162 |
| N1—H2N1···S4[i] | 0.91 | 2.47 | 3.359 (2) | 165 |
| N1—H3N1···S1[ii] | 0.91 | 2.54 | 3.4297 (19) | 165 |
| N1—H3N1···S3[ii] | 0.91 | 2.85 | 3.3019 (19) | 112 |
| N2—H1N2···S3[iii] | 0.91 | 2.58 | 3.376 (2) | 147 |
| N2—H1N2···S2[iii] | 0.91 | 2.92 | 3.580 (2) | 131 |
| N2—H2N2···S4 | 0.91 | 2.43 | 3.309 (2) | 164 |
| N2—H3N2···S1[i] | 0.91 | 2.50 | 3.393 (2) | 169 |
| C2—H2A···S4[i] | 0.98 | 2.97 | 3.759 (3) | 139 |
| C5—H5B···S3[ii] | 0.98 | 2.98 | 3.639 (6) | 126 |

Symmetry codes: (i) $x$, $-y+1$, $z-1/2$; (ii) $-x+1/2$, $-y+3/2$, $-z+1$; (iii) $-x+1/2$, $y-1/2$, $-z+3/2$.
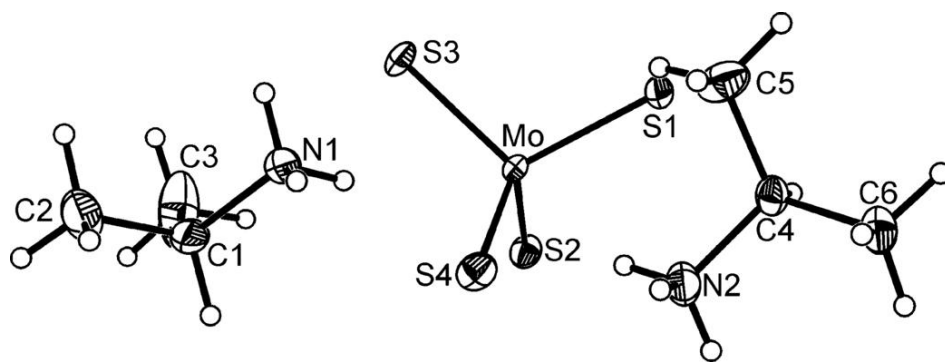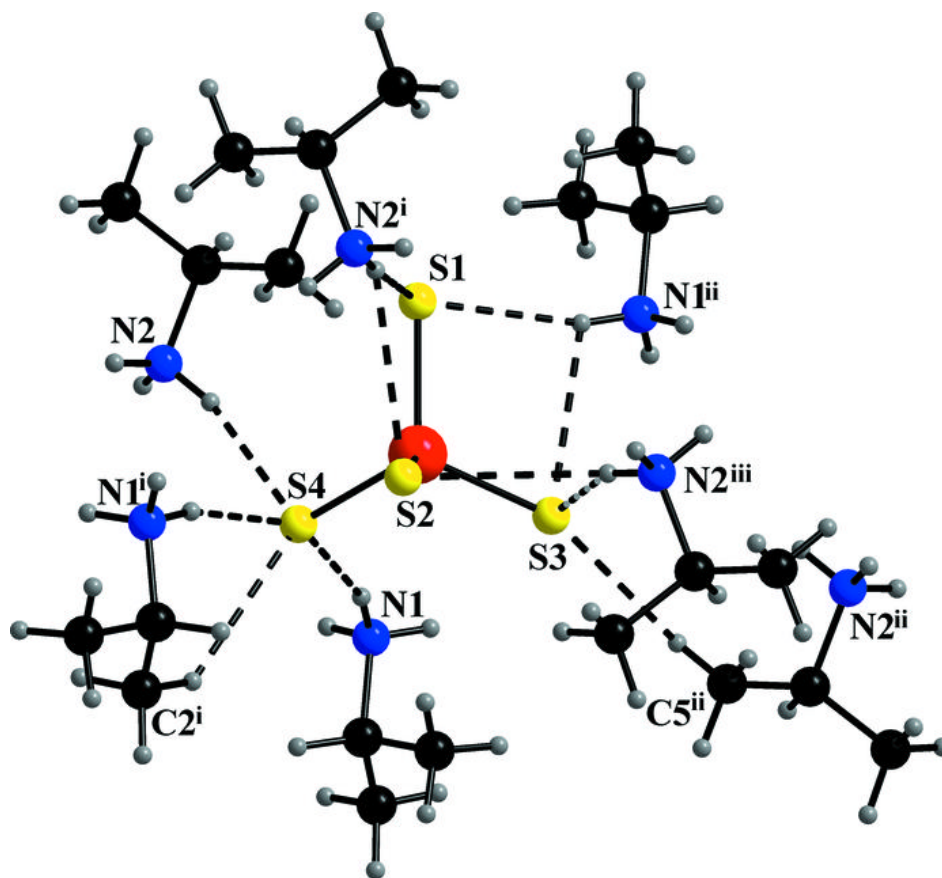
**Fig. 1**

**Fig. 2**

**Fig. 3**