

S(1)—Cr—S(2)	81.66 (7)	S(3)—Cr—S(4)	81.39 (6)
S(5)—Cr—S(6)	81.50 (6)	S(1)—Cr—S(3)	96.98 (6)
S(2)—Cr—S(6)	95.37 (6)	S(4)—Cr—S(5)	96.01 (7)
S(1)—P(1)—S(2)	105.77 (9)	S(3)—P(2)—S(4)	105.30 (9)
S(5)—P(3)—S(6)	105.22 (9)	O(1)—P(1)—O(2)	96.6 (2)
O(3)—P(2)—O(4)	96.0 (2)	O(5)—P(3)—O(6)	96.6 (2)

Data were collected with *CONTROL* (Molecular Structure Corporation, 1986) software. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983); the Cr heavy atom was located in an *E* map and the remaining non-H atoms were located using *DIRDIF* (Beurskens, 1984). H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all non-H atoms. Calculations were performed on a VAX 3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

This work is supported by a grant for a Major Project from the State Science and Technology Commission, and the National Science Foundation of China, as well as the State Key Laboratory of Tribology for Tsinghua University.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1031). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A *cis*-Oxo[diphenylhydrazido(2-)]-molybdenum Complex: (Et<sub>3</sub>NH)<sub>2</sub>[MoO(NNPh<sub>2</sub>)(SC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>)<sub>2</sub>]

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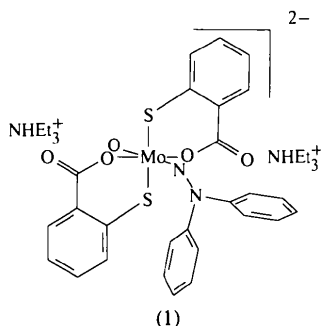
(Received 22 November 1994; accepted 9 May 1995)

## Abstract

The structure of bis(triethylammonium) (diphenylhydrazido)bis(2-mercaptobenzoato-*O,S*)oxomolybdate (VI) consists of two triethylammonium cations and a dianionic molybdenum complex. The structure exhibits the anticipated *cis*-oxo[diphenylhydrazido(2-)] geometry with two dianionic mercaptobenzoate ligands whose S donor atoms are mutually *trans*.

## Comment

Transition metal complexes containing the organohydrazido(2-) ligand have been studied extensively because the NNRR' ligand (RR' = alkyl and/or aryl) is of interest as a potential model of the NNH<sub>2</sub> intermediate detected in the chemical and enzymatic conversion of dinitrogen into ammonia (Henderson, Leigh & Pickett, 1992; Leigh, 1992). As part of our current research (Bustos *et al.*, 1991; Bustos, Manzur, Carrillo, Robert & Gouzerh, 1994), the study of the title compound (1) was undertaken.



The structure is closely related to that of the recently reported (Li-Kao, González, Baggio, Garland & Carrillo, 1995) compound bis(triethylammonium) bis-(2-mercaptobenzoato-*O,S*)dioxomolybdate(VI), (2), the coordination around the metallic centre being quite similar, though slightly distorted probably due to the steric hindrance introduced by the bulky  $\text{NNPh}_2$  anion replacing an O atom in (2).

As for (2), the structure of (1) comprises (Fig. 1) monomeric units consisting of two mercaptobenzoato ligands coordinated to a *cis*- $\text{MoO}(\text{N}-\text{N}'\text{Ph}_2)$  core *via* their respective mercapto S and carboxylate O atoms (S1, O2, S2 and O4). The ways in which the organic ions approach the metal are dissimilar: while in (1) the ligand binds in a 'slanted' way, thus leaving the metal centre 1.80 Å away from planar core of the ligand and forcing the  $\text{CO}_2$  group to rotate *ca* 30° out of the plane, in (2) the ligand binds in a rather 'straight' fashion, with the Mo atom displaced 0.002 (1) Å and the carboxylate group rotated 10° with respect to the least-squares plane of the ligand. These different geometries are also reflected in the differences found in the chelate angles

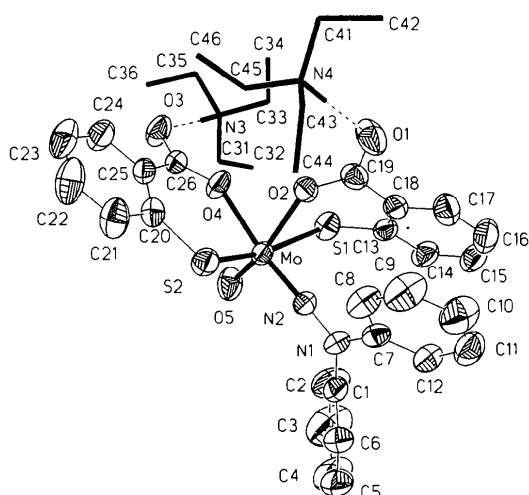


Fig. 1. View of the molecule showing the numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. H atoms are omitted for clarity.  $\text{NHEt}_3$  groups are presented in a schematic way, showing only one of the two almost overlapping disordered units.

subtended by each ligand to the metal centre: S1—Mo—O2 78.12 (6) and S2—Mo—O4 84.72 (6)° in (1), with the homologous values in (2) being 81.5 (1) and 82.0 (1)°, respectively.

As for (2), the coordination sphere around the  $\text{Mo}^{\text{VI}}$  ion is well described as a slightly distorted octahedron, with the basal plane now defined by Mo—N2 [1.774 (2) Å], Mo—O5 [1.701 (2) Å], Mo—O2 [2.250 (2) Å] and Mo—O4 [2.152 (2) Å]; r.m.s. deviations from the least-squares plane through the metal centre average 0.09 Å. The slightly more distorted character of the Mo environment in (1) is evident from the deviation from the vertical of the apical sites (occupied by S1 and S2 of the organic anion) by 8°, as opposed to 5° in (2).

The two triethylammonium units in the formula appear to be disordered in two slightly displaced positions, each with almost equal occupancy of 0.5 (attempts to refine an ordered model in *P1* proved unsuccessful as the refinement became highly unstable). As in (2), they interact with the ion through  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds (Figs. 1 and 2) ( $\text{O1}\cdots\text{H4}-\text{N4}$  estimated length/angle 1.73 Å/174°,  $\text{O3}\cdots\text{H3}-\text{N3}$  1.80 Å/174°).

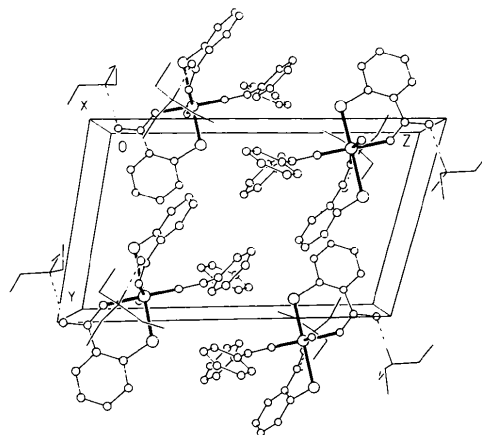


Fig. 2. Packing viewed along (100).

## Experimental

The title complex was synthesized by reacting  $[\text{MoO}(\text{NNPh}_2)(\text{acac})_2]$  (Carrillo, Robert & Gouzerh, 1992) (2.867 g in 15 ml of MeOH), where acac is acetylacetonato, and triethylammonium mercaptobenzoate (1.750 g of mercaptobenzoic acid and 3.50 ml of triethylamine in 15 ml of MeOH). The mixture was refluxed for 1 h and the red oil formed was collected, dispersed with diethyl ether and recrystallized from a  $\text{CH}_2\text{Cl}_2$ /hexane mixture.

### Crystal data

$(\text{C}_6\text{H}_{16}\text{N})_2[\text{Mo}(\text{C}_{12}\text{H}_{10}\text{N}_2)(\text{C}_7\text{H}_4\text{O}_2\text{S})_2(\text{O})]$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å

$M_r = 802.88$   
 Triclinic  
 $P\bar{1}$   
 $a = 11.357(4) \text{ \AA}$   
 $b = 11.643(4) \text{ \AA}$   
 $c = 17.638(9) \text{ \AA}$   
 $\alpha = 98.49^\circ$   
 $\beta = 96.44^\circ$   
 $\gamma = 117.360(10)^\circ$   
 $V = 2005.2(14) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.330 \text{ Mg m}^{-3}$

Cell parameters from 25 reflections  
 $\theta = 7.5\text{--}12.5^\circ$   
 $\mu = 0.476 \text{ mm}^{-1}$   
 $T = 293(2) \text{ K}$   
 Prism  
 $0.60 \times 0.32 \times 0.28 \text{ mm}$   
 Orange

C15	-0.0351(4)	-0.6288(3)	0.3153(2)	0.0659(10)
C16	0.0990(4)	-0.5932(3)	0.3346(2)	0.0691(10)
C17	0.1938(3)	-0.4897(3)	0.3090(2)	0.0594(9)
C18	0.1555(3)	-0.4183(3)	0.2645(2)	0.0439(7)
C19	0.2664(3)	-0.3082(3)	0.2387(2)	0.0489(8)
C20	0.3783(3)	0.1695(3)	0.2308(2)	0.0442(7)
C21	0.4881(3)	0.2967(3)	0.2635(2)	0.0670(10)
C22	0.5613(4)	0.3775(4)	0.2173(3)	0.0795(12)
C23	0.5273(4)	0.3356(4)	0.1376(2)	0.0749(11)
C24	0.4180(3)	0.2129(3)	0.1040(2)	0.0602(9)
C25	0.3412(3)	0.1278(3)	0.1492(2)	0.0426(7)
C26	0.2233(3)	-0.0011(3)	0.1039(2)	0.0430(7)
N3X†	-0.0054(6)	-0.2584(7)	-0.0440(3)	0.043(3)
C31X†	-0.1281(9)	-0.2684(11)	-0.0178(6)	0.062(3)
C32X†	-0.2540(11)	-0.4022(16)	-0.0389(15)	0.150(12)
C33X†	0.0358(9)	-0.3532(7)	-0.0195(4)	0.041(3)
C34X†	0.1767(12)	-0.3250(17)	-0.0281(12)	0.072(4)
C35X†	-0.0049(8)	-0.2542(6)	-0.1274(3)	0.039(2)
C36X†	-0.0420(20)	-0.1558(16)	-0.1543(8)	0.059(4)
N3Y†	-0.0220(9)	-0.2527(9)	-0.0418(4)	0.060(4)
C31Y†	-0.1360(11)	-0.2735(12)	-0.0014(6)	0.074(4)
C32Y†	-0.2549(7)	-0.4045(14)	-0.0458(11)	0.117(10)
C33Y†	0.0290(13)	-0.3467(13)	-0.0295(9)	0.117(7)
C34Y†	0.1791(14)	-0.2929(18)	-0.0242(14)	0.093(6)
C35Y†	-0.0598(13)	-0.2592(12)	-0.1263(4)	0.133(7)
C36Y†	-0.0051(20)	-0.1264(15)	-0.1482(9)	0.068(5)
N4X†	0.5675(8)	-0.1167(7)	0.2018(3)	0.046(3)
C41X†	0.6784(7)	-0.1492(7)	0.1970(4)	0.064(2)
C42X†	0.6393(13)	-0.2785(10)	0.1408(7)	0.107(5)
C43X†	0.6036(8)	0.0029(6)	0.2629(4)	0.075(2)
C44X†	0.652(2)	-0.0043(18)	0.3444(4)	0.197(13)
C45X†	0.4999(8)	-0.1150(8)	0.1253(4)	0.083(3)
C46X†	0.5795(13)	-0.0005(11)	0.0895(6)	0.109(4)
N4Y†	0.5830(9)	-0.1240(7)	0.1990(4)	0.065(4)
C41Y†	0.6417(13)	-0.1846(11)	0.1456(5)	0.110(4)
C42Y†	0.6366(13)	-0.2991(9)	0.1778(7)	0.092(4)
C43Y†	0.6768(8)	-0.0482(10)	0.2755(4)	0.112(3)
C44Y†	0.6121(16)	-0.0089(16)	0.3373(6)	0.105(5)
C45Y†	0.5325(8)	-0.0454(8)	0.1603(6)	0.095(4)
C46Y†	0.6415(11)	0.0698(10)	0.1361(8)	0.116(5)

† Occupancy = 0.5.

**Data collection**

Siemens R3m diffractometer  
 $\theta/2\theta$  scans (4.19–29.3° min<sup>-1</sup>)  
 Absorption correction: none  
 5710 measured reflections  
 5272 independent reflections  
 4688 observed reflections  
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0094$   
 $\theta_{\text{max}} = 22.55^\circ$   
 $h = 0 \rightarrow 12$   
 $k = -12 \rightarrow 11$   
 $l = -19 \rightarrow 18$   
 2 standard reflections monitored every 98 reflections  
 intensity decay: none

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.0285$   
 $wR(F^2) = 0.0702$   
 $S = 1.065$   
 5270 reflections  
 566 parameters  
 H atoms not refined; riding on host atoms  
 $w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 1.4374P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.026$   
 $\Delta\rho_{\text{max}} = 0.295 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.220 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

**Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )**

$U_{\text{iso}}$  for C31Y, C33Y, C35Y;  $U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$  for others.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}/U_{\text{iso}}$
Mo	0.10852(2)	-0.13424(2)	0.24672(1)	0.03728(10)
S1	-0.04385(8)	-0.37611(8)	0.18426(5)	0.0499(2)
S2	0.30370(9)	0.08272(8)	0.30143(4)	0.0509(2)
O1	0.3675(3)	-0.3219(2)	0.2283(2)	0.0857(9)
O2	0.2590(2)	-0.2061(2)	0.23002(12)	0.0452(5)
O3	0.1922(3)	-0.0135(2)	0.03139(12)	0.0651(7)
O4	0.1574(2)	-0.0948(2)	0.13567(11)	0.0526(5)
O5	-0.0232(2)	-0.1033(2)	0.22814(14)	0.0620(6)
N1	0.0900(2)	-0.1966(2)	0.41022(13)	0.0439(6)
N2	0.1031(2)	-0.1682(2)	0.34145(13)	0.0412(6)
C1	-0.0259(3)	-0.2061(3)	0.4401(2)	0.0509(8)
C2	-0.1502(3)	-0.2664(4)	0.3903(2)	0.0847(12)
C3	-0.2614(4)	-0.2744(6)	0.4198(3)	0.127(2)
C4	-0.2460(5)	-0.2222(6)	0.4980(3)	0.119(2)
C5	-0.1217(5)	-0.1598(5)	0.5470(3)	0.095(2)
C6	-0.0109(4)	-0.1521(4)	0.5180(2)	0.0682(10)
C7	0.1851(3)	-0.2273(3)	0.4501(2)	0.0435(7)
C8	0.3191(3)	-0.1640(3)	0.4420(2)	0.0603(9)
C9	0.4101(3)	-0.1975(4)	0.4777(2)	0.0794(12)
C10	0.3689(4)	-0.2930(4)	0.5206(2)	0.0810(12)
C11	0.2362(4)	-0.3557(4)	0.5288(2)	0.0684(10)
C12	0.1430(3)	-0.3237(3)	0.4936(2)	0.0535(8)
C13	0.0183(3)	-0.4537(3)	0.2442(2)	0.0420(7)
C14	-0.0762(3)	-0.5609(3)	0.2700(2)	0.0548(8)

**Table 2. Selected geometric parameters ( $\text{\AA}$ , °)**

Mo—O5	1.701(2)	C7—C12	1.386(4)
Mo—N2	1.774(2)	C7—C8	1.388(4)
Mo—O4	2.152(2)	C8—C9	1.384(4)
Mo—O2	2.250(2)	C9—C10	1.371(5)
Mo—S2	2.4248(11)	C10—C11	1.376(5)
Mo—S1	2.5131(11)	C11—C12	1.387(6)
S1—C13	1.770(3)	C13—C14	1.401(4)
S2—C20	1.763(3)	C13—C18	1.403(4)
O1—C19	1.259(4)	C14—C15	1.386(4)
O2—C19	1.260(3)	C15—C16	1.367(5)
O3—C26	1.256(3)	C16—C17	1.376(4)
O4—C26	1.261(3)	C17—C18	1.394(4)
N1—N2	1.310(3)	C18—C19	1.501(4)
N1—C7	1.431(4)	C20—C25	1.399(4)
N1—C1	1.436(4)	C20—C21	1.404(4)
C1—C2	1.374(4)	C21—C22	1.378(5)
C1—C6	1.381(6)	C22—C23	1.367(7)
C2—C3	1.389(7)	C23—C24	1.374(5)
C3—C4	1.379(8)	C24—C25	1.404(4)
C4—C5	1.366(8)	C25—C26	1.498(4)
C5—C6	1.381(7)		
O5—Mo—N2	103.20(11)	C8—C7—N1	119.4(3)
O5—Mo—O4	89.70(10)	C9—C8—C7	119.6(3)
N2—Mo—O4	167.00(10)	C10—C9—C8	120.5(3)
O5—Mo—O2	161.53(10)	C9—C10—C11	119.9(3)
N2—Mo—O2	91.62(9)	C10—C11—C12	120.7(3)
O4—Mo—O2	76.07(8)	C7—C12—C11	119.2(3)
O5—Mo—S2	104.58(9)	C14—C13—C18	118.1(3)
N2—Mo—S2	90.34(8)	C14—C13—S1	117.8(2)
O4—Mo—S2	84.72(6)	C18—C13—S1	124.0(2)
O2—Mo—S2	86.03(6)	C15—C14—C13	120.9(3)
O5—Mo—S1	90.54(8)	C16—C15—C14	120.4(3)

N2—Mo—S1	90.86 (8)	C15—C16—C17	120.0 (3)
O4—Mo—S1	90.67 (6)	C16—C17—C18	120.9 (3)
O2—Mo—S1	78.12 (6)	C17—C18—C13	119.7 (3)
S2—Mo—S1	164.14 (3)	C17—C18—C19	116.8 (3)
C13—S1—Mo	102.18 (10)	C13—C18—C19	123.5 (3)
C20—S2—Mo	114.22 (10)	O1—C19—O2	122.2 (3)
C19—O2—Mo	134.8 (2)	O1—C19—C18	116.7 (3)
C26—O4—Mo	141.8 (2)	O2—C19—C18	121.1 (3)
N2—N1—C7	119.1 (2)	C25—C20—C21	118.1 (3)
N2—N1—C1	118.5 (2)	C25—C20—S2	128.5 (2)
C7—N1—C1	122.2 (2)	C21—C20—S2	113.5 (2)
N1—N2—Mo	176.1 (2)	C22—C21—C20	121.6 (3)
C2—C1—C6	120.7 (3)	C23—C22—C21	120.4 (3)
C2—C1—N1	119.5 (3)	C22—C23—C24	119.2 (3)
C6—C1—N1	119.8 (3)	C23—C24—C25	122.0 (3)
C1—C2—C3	118.9 (4)	C20—C25—C24	118.7 (3)
C4—C3—C2	120.0 (4)	C20—C25—C26	125.8 (2)
C5—C4—C3	120.8 (4)	C24—C25—C26	115.5 (3)
C4—C5—C6	119.4 (4)	O3—C26—O4	120.4 (3)
C5—C6—C1	120.1 (4)	O3—C26—C25	117.0 (2)
C12—C7—C8	120.1 (3)	O4—C26—C25	122.6 (3)
C12—C7—N1	120.4 (3)		

The structure was solved through a combination of direct methods and difference Fourier synthesis. Refinement was on  $F^2$  using the whole data set. H atoms were included at their expected positions, with fixed thermal parameters, and allowed to ride on their host atoms. The two disordered  $\text{NHET}_3$  groups were subject to a mild similarity restraint in homologous bond distances.

Data collection: *P3/P4-PC* (Siemens, 1991). Cell refinement: *P3/P4-PC*. Data reduction: *XDISK* in *SHELXTL/PC* (Sheldrick, 1991). Program(s) used to solve structure: *XS* in *SHELXTL/PC*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB*.

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Lists of structure factors, anisotropic displacement parameters, least-squares-planes data, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1181). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## (*N,N*-Dimethylthiocarbamoylthioacetato)-triphenyltin

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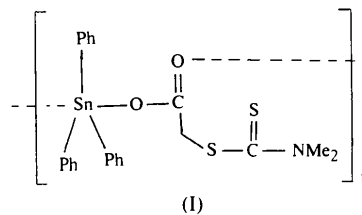
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## Abstract

Carboxylate bridges link the two independent molecules in the asymmetric unit of the title compound,  $[\text{Sn}(\text{C}_5\text{H}_8\text{NO}_2\text{S}_2)(\text{C}_6\text{H}_5)_3]$ , into a helical chain parallel to **b** {*i.e.* *catena*-poly[triphenyltin- $\mu$ -(*N,N*-dimethylthiocarbamoylthioacetato)-*O:O'*]}. Both five-coordinate Sn atoms show *trans*- $\text{C}_3\text{SnO}_2$  trigonal bipyramidal coordination geometry.

## Comment

The bond lengths around the Sn atoms in this fungicidal compound, (I) (Kumar Das, Kuthubutheen, Ng & Ng, 1987), compare well with those found in other carboxylate-bridged triorganotin carboxylates (Ng, Chen



& Kumar Das, 1988). The repeat distance of the chain (4.33 Å) is much shorter than that found for  $(\text{C}_4\text{H}_9)_3\text{SnO}_2\text{CCH}_2\text{SC}(\text{S})\text{N}(\text{CH}_3)(\text{C}_6\text{H}_5)$  (5.12 Å; Ng, Chen, Kumar Das, Yap & Butcher, 1992), and is in agreement with the flexible chain structure previously inferred from variable-temperature Mössbauer spectroscopy ( $a = -0.0152 \text{ K}^{-1}$ ) (Ng & Kumar Das,