Discussion. Positional parameters and isotropic *B* values are listed in Table 1;* the numbering of the atoms is given in Fig. 1; intramolecular bond distances and valency angles are listed in Table 2. The Pb atom is five-coordinated in a distorted trigonal bipyramidal geometry, I and N being in apical and C(1), C(10) and C(17) in the equatorial positions. The Pb atom lies slightly [0·275 (5) Å] below the plane of these C atoms on the side of the I atom. The 4-methoxyphenyl and 4-methylphenyl groups make angles of 21·8 (3) and 44·8 (3)° respectively with the plane through C(1), C(10) and C(17); the benzylamine group is rotated out of this plane by 73·9 (2)° (Fig. 2).

All these features are also found in the tin compound mentioned with the exception of the 4-methylphenyl

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* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42040 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(1), C(10), C(17) dihedral angle, which is 8·3° in the tin compound. The I—Pb—N angle at 168·8 (1)° in the lead compound is slightly smaller than the Br—Sn—N angle (171·0°) in the tin compound.

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Structure of μ-Oxo-bis{[N-(2-mercaptoethyl)-N',N'-dimethylethylenediamino]dioxomolybdenum(VI)}, [Mo2O5{(CH3)2NCH2CH2NHCH2CH2S}2]

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Abstract. $M_r = 565.9$, monoclinic, *C*2/*c*, $a = 11.801$ (2), $b = 8.521$ (2), $c = 21.319$ (4) Å, $\beta = 106.72$ (1)°, $V = 2053.0$ (8) Å³, $Z = 4$, $D_x = 1.82$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 14.22$ cm⁻¹, $F(000) = 1144$, room temperature, final $R = 0.055$ for 1060 reflections. The Mo atom displays distorted octahedral geometry with a facial stereochemistry for the bridging and terminal oxo groups. The angle at the bridging oxo group Mo—O(3)—Mo' is non-linear, 147.0 (5)°, possibly as a consequence of weak intramolecular hydrogen bonding between N(2)···O(2') and N(2')···O(2).

Introduction. Oxo complexes of Mo^{VI} have been under intense study (Bruce, Corbin, Dahlstrom, Hyde, Minelli, Stiefel, Spence & Zubieta, 1982) as potential model systems for Mo sites in enzymes (Stiefel, 1977). Ligands containing S donors are most relevant as there is strong spectroscopic evidence for mercapto coordi-

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nation to the enzymic Mo site. As part of a continuing investigation of the structural and synthetic chemistry of molybdenum–thiolate systems, we have been studying the chemistry of Mo–oxo moieties with tridentate ligands (Hyde, Magin & Zubieta, 1980; Hyde & Zubieta, 1982). Tridentate ligands are capable of stabilizing complexes with vacant coordination sites, as in [Mo2O3(SCH2CH2SCH2CH2S)2], which may potentially bind substrate or inhibitor molecules. Furthermore, our interest in the reactivity of the oxo functionality toward substituted hydrazines prompted the preparation and characterization of new Mo–oxo complexes as synthetic precursors. In this paper, we report the structure of one of these complexes, [Mo2O5(Me2NCH2CH2NHCH2CH2S)2].

Experimental. *N*-(2-Mercaptoethyl)-*N'*,*N'*-dimethylethylenediamine, (CH3)2NCH2CH2NHCH2CH2SH, was prepared from the reaction of ethylene sulfide and *N,N*-dimethylethylenediamine in toluene (Karlin & Lippard, 1976). A solution of the ligand (30 mmol) in

methanol (30 ml) was added dropwise to a solution of [MoO₂(CH₃CHOCHOHCH₃)₂].2(2,3-butanediol) (26 mmol) in methanol (75 ml). The resulting yellow solution was cooled gradually, whereupon bright-yellow crystals deposited. The crystals were washed with 2-propanol and ether and recrystallized from dichloromethane/2-propanol (yield, 72%). D_m not determined. Crystal dimensions: $0 \cdot 15 \times 0 \cdot 18 \times 0 \cdot 20$ mm. Nicolet $R3m$ diffractometer equipped with a highly oriented graphite monochromator (equatorial mode). 25 reflections in the range $20° \leq 2\theta \leq 25°$ used to generate lattice parameters. Reflections in the quadrant $\pm h$, $+k$, $+l$ collected using coupled θ(crystal)–2θ(counter) scan mode, $0° \leq 2\theta \leq 45°$, variable scan speed 3 to $30°$ min⁻¹, scan width $[2\theta(K\alpha_1)-1\cdot 0]°$ to $[2\theta(K\alpha_2)+1\cdot 0]°$, backgrounds measured in the stationary-crystal–stationary-counter mode at each end of the 2θ scan range for half the time taken for the 2θ scan.

Standard reflections measured every 197 data reflections showed no significant deviations over the 74 h data-collection time. A total of 1753 reflections collected; 1060 with $F_o \geq 6\sigma(F_o)$ used in subsequent refinement. Data corrected for background, Lorentz and polarization effects and absorption ($T_{max}/T_{min} = 1\cdot 05$) in the usual fashion (Bruce et al., 1982). Coordinates of the Mo atom found from a three-dimensional Patterson synthesis, and a series of least-squares refinements followed by three-dimensional difference Fourier syntheses revealed all non-hydrogen atoms. Refinement of positional and anisotropic thermal parameters for all non-hydrogen atoms with H atoms included in their idealized positions with d(C–H) = $0\cdot 96$ and d(N–H) = $1\cdot 02$ Å resulted in convergence with $R_F = 5\cdot 50\%$ and $wR_F = 5\cdot 74\%$, $w = 1/\sigma^2$, with a goodness of fit of $1\cdot 61$. $(\Delta/\sigma)_{max} = 0\cdot 42$. Max. and min. heights on final $\Delta\rho$ map $1\cdot 1$ and $-0\cdot 6$ e Å⁻³. No reflections affected significantly by secondary extinction. The average value of the function $\sum w(|F_o|-|F_c|)^2$ showed no major variations as a function of $|F_o|$ or of $(\sin\theta)/\lambda$. The weighting scheme is thus satisfactory and the structure complete. All calculations performed on a Nova 3 computer using local versions of SHELXTL programs (Sheldrick, 1978) as supplied by Nicolet XRD.

Table 1. *Atom coordinates* ($\times 10^4$) *and temperature factors* (Å² $\times 10^3$)

U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Mo	−334 (1)	6971 (1)	3287 (1)	25 (1)
S(1)	130 (3)	7856 (3)	4422 (1)	35 (1)
O(1)	−1307 (7)	5589 (9)	3364 (4)	43 (3)
O(2)	−1103 (6)	8679 (9)	2986 (3)	33 (3)
O(3)	0	6334 (11)	2500	27 (3)
N(1)	1209 (7)	4944 (10)	3754 (4)	32 (3)
N(2)	1503 (8)	8212 (9)	3456 (4)	30 (3)
C(1)	2353 (9)	5730 (13)	3966 (6)	39 (4)
C(2)	2438 (10)	7017 (11)	3497 (6)	33 (4)
C(3)	1842 (9)	9346 (13)	4030 (5)	36 (4)
C(4)	805 (12)	9696 (12)	4281 (6)	46 (5)
C(5)	1188 (10)	3722 (12)	3248 (6)	38 (4)
C(6)	970 (11)	4100 (14)	4327 (6)	46 (4)

Table 2. *Bond lengths* (Å) *and angles* (°)

Mo–S(1)	2·442 (3)	Mo–O(1)	1·685 (8)
Mo–O(2)	1·737 (7)	Mo–O(3)	1·908 (3)
Mo–N(1)	2·499 (8)	Mo–N(2)	2·345 (9)
S(1)–C(4)	1·821 (12)		
N(1)–C(1)	1·457 (13)	N(1)–C(5)	1·495 (15)
N(1)–C(6)	1·512 (17)	N(2)–C(2)	1·485 (14)
N(2)–C(3)	1·521 (14)	C(1)–C(2)	1·506 (17)
C(3)–C(4)	1·500 (19)		

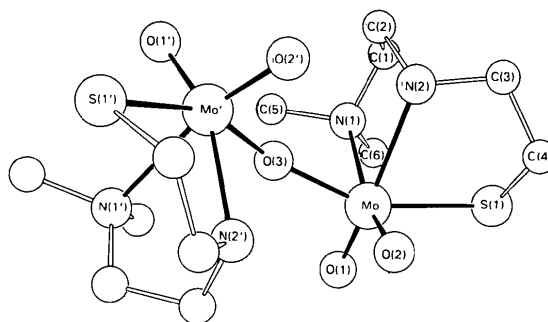| S(1)–Mo–O(1)	94·8 (3)	S(1)–Mo–O(2)	92·9 (2)
O(1)–Mo–O(2)	108·9 (4)	S(1)–Mo–O(3)	156·2 (1)
O(1)–Mo–O(3)	101·9 (3)	O(2)–Mo–O(3)	97·6 (3)
S(1)–Mo–N(1)	83·6 (2)	O(1)–Mo–N(1)	85·3 (3)
O(2)–Mo–N(1)	165·7 (3)	O(3)–Mo–N(1)	81·0 (3)
S(1)–Mo–N(2)	77·0 (3)	O(1)–Mo–N(2)	157·8 (3)
O(2)–Mo–N(2)	92·3 (3)	O(3)–Mo–N(2)	81·3 (3)
N(1)–Mo–N(2)	73·4 (3)	Mo–S(1)–C(4)	95·0 (4)
Mo–O(3)–Mo'	147·0 (5)	Mo–N(1)–C(1)	108·1 (6)
Mo–N(1)–C(5)	109·5 (5)	C(1)–N(1)–C(5)	111·4 (9)
Mo–N(1)–C(6)	111·5 (7)	C(1)–N(1)–C(6)	109·9 (8)
C(5)–N(1)–C(6)	106·5 (8)	Mo–N(2)–C(2)	109·8 (6)
Mo–N(2)–C(3)	115·3 (7)	C(2)–N(2)–C(3)	111·7 (8)
N(1)–C(1)–C(2)	111·2 (8)	N(2)–C(2)–C(1)	110·4 (10)
N(2)–C(3)–C(4)	111·2 (8)	S(1)–C(4)–C(3)	109·1 (8)

Fig. 1. Perspective view of the molecule [Mo₂O₅(Me₂NCH₂CH₂NHCH₂CH₂S)₂], showing the atom-labelling scheme.
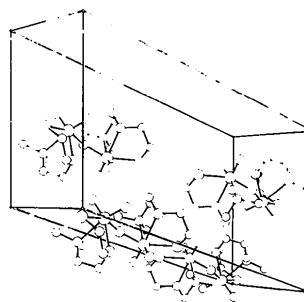

Fig. 2. A view of the packing in the unit cell.

Discussion. The atomic parameters are listed in Table 1,* interatomic distances and angles in Table 2. Fig. 1 is a view of the molecule and Fig. 2 shows the packing.

The structure consists of binuclear units, with the bridging oxo group O(3) located on the twofold axis passing through $0, y, \frac{1}{4}$. The symmetry-related halves of the molecule display pseudo-octahedral geometry at Mo atoms, with a *fac* stereochemistry adopted by the bridging and terminal oxo groups. The remaining positions are occupied by the amine-N and mercapto-S donors of the tridentate ligands. In common with most structures of this type, the N donors are located pseudo-*trans* to the terminal oxo groups, while the S donor occupies a position *trans* to the less strongly π-bonding bridging oxo group.

Two significant features of the structure are the Mo—O(3)—Mo′ angle of 147·0 (5)° and the N(2)···O(2′)$(x,y,\frac{1}{2}-z)$ close contact of 3·00 (1) Å, suggesting some degree of intramolecular hydrogen bonding. Short intermolecular contacts C(3)—H···O(1)$(\frac{1}{2}+x, \frac{1}{2}+y, z)$ of 2·44 (1) Å may conform to criteria for hydrogen bonding and thus make a significant contribution to the overall molecular packing and conformation (Berkovitch-Yellin & Leiserowitz, 1984).

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* List of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42042 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The structure is similar to that determined for [Mo₂O₅{Me₂NCH₂CH₂NHCH₂C(Me₂)S}₂], which was reported during the course of our investigations (Marabella, Enemark, Miller, Bruce, Pariyadath, Corbin & Stiefel, 1983). This analogue exhibits an angle of 143·8 (3)° at the bridging oxo group, with average Mo—O(bridging) and Mo—O(terminal) distances of 1·923 (8) and 1·713 (9) Å, respectively. Other structural parameters correspond closely to those observed for the title structure.

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Synthesis and Structure of *cis*-Tetrakis(acetamidine)carbonatocobalt(III) Chloride Dihydrate, [Co(CO₃)(C₂H₆N₂)₄]Cl.2H₂O at 117 K

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Abstract. $M_r = 422·76$, monoclinic, $C2$, $a = 16·475$ (4), $b = 9·176$ (2), $c = 12·421$ (3) Å, $\beta = 101·52$ (2)°, $V = 1839·9$ (8) Å³, $Z = 4$, $D_x = 1·526$ (1) Mg m⁻³, Cu $K\alpha$, $\lambda = 1·54184$ Å, $\mu = 9·3$ mm⁻¹, $F(000) = 839·8$, $T = 117$ (2) K, final $R = 0·038$ for 1257 reflections. The crystals of the title compound formed by evaporation in air of a methanolic solution of CoCl₂ and acetamidine contain complexes of *cis*-tetrakis(acetamidine)carbonatocobalt(III) ions. The Co—O distances within the complex ion, 1·918 (6)

and 1·933 (5) Å, and the Co—N distances ranging from 1·913 (6) to 1·942 (6) Å agree with those found in related *cis*-carbonato complexes of Co^III.

Introduction. The present study is part of a research project on synthetic and structural studies of acetamidine metal complexes (see also Norrestam, 1984a). So far, the study by Stephenson (1962) on a compound expected to be bis(acetonitrile)tetraammineplatinum(II) chloride monohydrate but shown by the