of 1·292 (11)–1·309 (11) Å are in agreement with those found in other dithiobisformamidinium salts (Villa, Manfredotti, Nardelli & Tani, 1972; Lis, 1979). Also, the S valency angles |102·9 (4) and 102·4 (3)°| and the torsion angle of the disulphide group |100·2 (5)°| lie in the normal range. The two thiourea parts of the cation are both planar.

The structure is held together by hydrogen bonds utilizing all H atoms. The data on these hydrogen bonds are given in Table 3. Two |O(1)–H(1)···Cl(5) and O(1)–H(10)···O(2)| may be classified as relatively strong.

References

ADMAN, E. & MARGULIS, T. N. (1967). Inorg. Chem. 6, 210–214.

BEYER, H., GLEMSER, O. & KREBS, B. (1968). Angew. Chem. 80, 286–287.
COTTON, F. A., FRENZ, B. A. & SHIVE, L. W. (1975). Inorg. Chem. 14, 649–652.
FRAIS, P. W. & LOCK, C. J. L. (1972). Can. J. Chem. 50, 1811–1818.
GRUNDY, H. D. & BROWN, I. D. (1970). Can. J. Chem. 48, 1151–1154.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
KOZ'MIN, P. A., NOVITSKAIA, G. N. & KUZNETSOV, W. G. (1973). Zh. Strukt. Khim. 14, 680–684.
LIS, T. (1976). Acta Cryst. B32, 2707–2709.
LIS, T. (1977). Acta Cryst. B33, 944–946.
LIS, T. (1979). Acta Cryst. B35, 3041–3044.
LISHER, E. J., COWLAM, N. & GILLOTT, L. (1979). Acta Cryst. B35, 1033–1038.
VILLA, A. C., MANFREDOTTI, A. G., NARDELLI, M. & TANI, M. E. V. (1972). Acta Cryst. B28, 356–360.

Bis(tetraethylammonium) Di-μ-sulphido-bis[oxo(disulphido)molybdenum]

By WILLIAM CLEGG AND GEORGE M. SHELDRICK

Anorganisch-Chemisches Institut der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

AND C. DAVID GARNER AND GEORGE CHRISTOU

Department of Chemistry, Manchester University, Manchester M13 9PL, England

Abstract. $[N(C_2H_5)_4]_2[Mo_2O_2S_2(S_2)_2]$, $2C_8H_{20}N^+.Mo_2O_2S_6^{2-}$, $M_r = 676·6$, monoclinic, $P2_1/c$, $a = 13·485$ (3), $b = 14·354$ (3), $c = 14·749$ (3) Å, $\beta = 97·50$ (2)°, $U = 2830·2$ Å^3, $Z = 4$, $D_x = 1·588$ Mg m^{-3}; final $R = 0·0358$ for 4025 reflexions. The anion contains two Mo^V atoms, each with a terminal O^{2-} and an edge-on $(S_2)^{2-}$ ligand, bridged by two S^{2-} ligands, and has approximate C_{2v} (mm) symmetry.

Introduction. The compound was obtained from a reaction between MoCl$_3$ and NaSPh (1:5 molar ratio) in methanol, followed by treatment with NEt$_4$Cl, and was recrystallized from a methyl cyanide–diethyl ether mixture. Intensities were collected from a crystal of average dimension 0·28 mm by a profile-fitting procedure (Clegg, 1981) with graphite-monochromated Mo Kα radiation ($\lambda = 0·71069$ Å) and a Stoe–Siemens AED four-circle diffractometer. 4025 unique reflexions with $7 \leq 2\theta \leq 60°$ and $I \geq 2\sigma(I)$ were measured and corrected for absorption by an empirical method based on measurements of sets of reflexions at different azimuthal angles ($\mu = 1·31$ mm^{-1}; transmission factors range from 0·588 to 0·654). Cell dimensions were obtained from the diffractometer angles for 50 centred reflexions with $20 \leq 2\theta \leq 25°$.

The structure was solved by Patterson and Fourier methods, and refined to a minimum value of $\sum w\Delta^2$ [$\Delta = |F_o| - |F_c|$; $w^{-1} = \sigma^2(F_o)$]. Anisotropic thermal parameters were refined for all non-H atoms. H atoms were assigned isotropic thermal parameters fixed at 1·3 times the equivalent isotropic value for the corresponding C atom. C–H lengths and H–C–H angles were constrained to 0·96 Å and 109·5°.

The final R is 0·0358, with $R_w = (\sum w\Delta^2/\sum wF_o^2)^{1/2} = 0·0291$. Coordinates and derived bond lengths and angles are given in Tables 1 and 2.*

Table 1. *Atomic coordinates* $(\times 10^5)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^4)$

	x	y	z	U
Mo(1)	24076 (3)	3249 (2)	8933 (2)	406 (1)
Mo(2)	28775 (3)	−4454 (2)	26621 (2)	433 (1)
S(1)	33145 (9)	10157 (8)	21648 (7)	589 (4)
S(2)	15716 (9)	−8594 (7)	15498 (7)	552 (4)
S(3)	18808 (9)	18305 (8)	3490 (8)	619 (5)
S(4)	9076 (9)	7526 (8)	−690 (8)	614 (4)
S(5)	29054 (10)	1615 (8)	41744 (7)	671 (5)
S(6)	19622 (10)	−9572 (8)	38683 (8)	672 (5)
O(1)	32239 (21)	−1264 (20)	2369 (17)	635 (12)
O(2)	38394 (22)	−11767 (21)	26059 (18)	714 (12)
N(1)	4325 (26)	63520 (22)	21493 (24)	606 (14)
C(11)	11934 (36)	68816 (32)	28000 (30)	811 (22)
C(12)	11912 (42)	66394 (38)	38022 (34)	1026 (26)
C(13)	6174 (38)	53110 (29)	22322 (40)	896 (24)
C(14)	16432 (45)	50019 (39)	20238 (51)	1493 (38)
C(15)	5428 (37)	66977 (32)	11931 (31)	764 (21)
C(16)	−1689 (40)	62604 (33)	4428 (32)	877 (23)
C(17)	−6102 (31)	65049 (29)	23707 (30)	654 (18)
C(18)	−9448 (38)	75119 (31)	23260 (33)	844 (23)
N(2)	50485 (24)	24171 (21)	−1921 (20)	449 (12)
C(21)	50567 (35)	15766 (30)	4238 (28)	678 (19)
C(22)	58530 (36)	15606 (33)	12337 (27)	738 (20)
C(23)	60065 (34)	24242 (36)	−6255 (30)	821 (22)
C(24)	61485 (40)	32297 (34)	−12430 (30)	926 (25)
C(25)	41493 (34)	23629 (34)	−9032 (29)	750 (20)
C(26)	40391 (37)	15068 (33)	−14884 (29)	820 (21)
C(27)	49892 (36)	33022 (30)	3455 (28)	710 (20)
C(28)	41702 (34)	33535 (31)	9421 (28)	676 (19)

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

Mo(1)⋯Mo(2)	2·828 (1)		
Mo(1)–S(1)	2·323 (1)	Mo(2)–S(1)	2·323 (1)
Mo(1)–S(2)	2·320 (1)	Mo(2)–S(2)	2·322 (1)
Mo(1) S(3)	2·381 (1)	Mo(2)–S(5)	2·390 (1)
Mo(1)–S(4)	2·394 (1)	Mo(2)–S(6)	2·409 (1)
Mo(1)–O(1)	1·687 (3)	Mo(2)–O(2)	1·679 (3)
S(3)–S(4)	2·070 (2)	S(5)–S(6)	2·062 (2)
N(1)–C(11)	1·515 (5)	N(2)–C(21)	1·509 (5)
N(1)–C(13)	1·517 (5)	N(2)–C(23)	1·514 (6)
N(1)–C(15)	1·520 (6)	N(2)–C(25)	1·498 (5)
N(1)–C(17)	1·501 (6)	N(2)–C(27)	1·505 (5)
C(11)–C(12)	1·519 (7)	C(21)–C(22)	1·498 (6)
C(13)–C(14)	1·522 (8)	C(23)–C(24)	1·499 (7)
C(15)–C(16)	1·504 (6)	C(25)–C(26)	1·498 (7)
C(17)–C(18)	1·513 (6)	C(27)–C(28)	1·501 (7)
S(1)–Mo(1)–S(2)	102·1 (1)	S(1)–Mo(2)–S(2)	102·0 (1)
S(1)–Mo(1)–S(3)	89·3 (1)	S(1)–Mo(2)–S(5)	89·5 (1)
S(2)–Mo(1)–S(3)	131·7 (1)	S(2)–Mo(2)–S(5)	132·1 (1)
S(1)–Mo(1)–S(4)	133·2 (1)	S(1)–Mo(2)–S(6)	133·2 (1)
S(2)–Mo(1)–S(4)	90·9 (1)	S(2)–Mo(2)–S(6)	91·7 (1)
S(3)–Mo(1)–S(4)	51·4 (1)	S(5)–Mo(2)–S(6)	50·9 (1)
S(1)–Mo(1)–O(1)	108·2 (1)	S(1)–Mo(2)–O(2)	108·6 (1)
S(2)–Mo(1)–O(1)	110·3 (1)	S(2)–Mo(2)–O(2)	108·9 (1)
S(3)–Mo(1)–O(1)	110·1 (1)	S(5)–Mo(2)–O(2)	110·8 (1)
S(4)–Mo(1)–O(1)	108·8 (1)	S(6)–Mo(2)–O(2)	108·6 (1)
Mo(1)–S(1)–Mo(2)	75·0 (1)	Mo(1)–S(3)–S(4)	64·6 (1)
Mo(1)–S(2)–Mo(2)	75·1 (1)	Mo(1)–S(4)–S(3)	64·0 (1)
Mo(2)–S(5)–S(6)	65·0 (1)	Mo(2)–S(6)–S(5)	64·1 (1)
C(11)–N(1)–C(13)	110·6 (3)	C(21)–N(2)–C(23)	108·6 (3)
C(11)–N(1)–C(15)	106·5 (3)	C(21)–N(2)–C(25)	108·8 (3)
C(13)–N(1)–C(15)	111·2 (4)	C(23)–N(2)–C(25)	111·2 (3)
C(11)–N(1)–C(17)	111·4 (3)	C(21)–N(2)–C(27)	110·8 (3)
C(13)–N(1)–C(17)	106·0 (3)	C(23)–N(2)–C(27)	108·9 (3)
C(15)–N(1)–C(17)	111·2 (3)	C(25)–N(2)–C(27)	108·6 (3)
N(1)–C(11)–C(12)	114·5 (4)	N(2)–C(21)–C(22)	116·1 (4)
N(1)–C(13)–C(14)	114·5 (4)	N(2)–C(23)–C(24)	116·4 (4)
N(1)–C(15)–C(16)	114·6 (4)	N(2)–C(25)–C(26)	117·2 (4)
N(1)–C(17)–C(18)	114·4 (4)	N(2)–C(27)–C(28)	116·4 (4)

Discussion. We have already described the crystal structure of the bis(tetramethylammonium) salt of this anion (Clegg, Mohan, Müller, Neumann, Rittner & Sheldrick, 1980). The structure determination for the bis(tetraethylammonium) salt is more precise, probably because of better crystal quality. The anion (Fig. 1) has essentially the same geometry in both salts. The major difference is in the degree of asymmetry of binding of the $(S_2)^2$ ligands to the Mo atoms. In the $|NMe_4|^+$ salt, both $(S_2)^{2-}$ ligands are asymmetrically bonded [Mo—S = 2·424 (5), 2·432 (5); 2·384 (6), 2·390 (5) Å] as is also the case in the $|Mo_2(S_2)_6|^{2-}$ anion [2·463 (4)–2·507 (4); 2·382 (4)–2·454 (4) Å] (Müller, Nolte & Krebs, 1978). In the structure described here, the asymmetry is slight |2·409 (1), 2·394 (1); 2·390 (1), 2·381 (1) Å|. In the anion of $[AsPh_4]_2[(S_2)Mo(S)-(\mu\text{-}S)_2Mo(S)(S_4)]$ no asymmetry of binding of the $(S_2)^{2-}$ ligand is detected |2·394 (3), 2·395 (2) Å| (Christou, Garner, Clegg & Sheldrick, 1980).

The two crystallographically independent $|NEt_4|^+$ cations have different arrangements of the ethyl groups
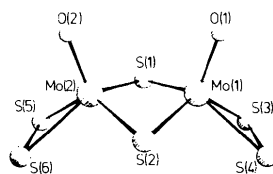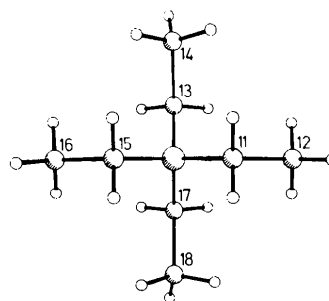
Fig. 2. The N(1) cation.

Fig. 1. The $|Mo_2O_2S_2(S_2)_2|^{2-}$ anion.

Fig. 3. The N(2) cation.

(different CH_2–N–CH_2–CH_3 torsion angles). The N(1) cation (Fig. 2) has approximate D_{2d} ($\overline{4}2m$) symmetry, making all methyl C atoms approximately coplanar with N(1). The N(2) cation (Fig. 3) has approximate S_4 ($\overline{4}$) symmetry, with the methyl groups widely separated.

References

CHRISTOU, G., GARNER, C. D., CLEGG, W. & SHELDRICK, G. M. (1980). Submitted.

CLEGG, W. (1981). *Acta Cryst.* A**37**. In the press.

CLEGG, W., MOHAN, N., MÜLLER, A., NEUMANN, A., RITTNER, W. & SHELDRICK, G. M. (1980). *Inorg. Chem.* **19**, 2066–2069.

MÜLLER, A., NOLTE, W.-O. & KREBS, B. (1978). *Angew. Chem. Int. Ed. Engl.* **17**, 279.

The Structure of Benzamido(phenyl)mercury(II)

BY JOAN HALFPENNY* AND R. W. H. SMALL

Chemistry Department, The University, Lancaster LA1 4YA, England

Abstract. $[Hg(C_6H_5)(C_7H_6NO)]$, $C_{13}H_{11}HgNO$, monoclinic, $P2_1/c$, a = 14·44 (1), b = 9·69 (1), c = 8·60 (1) Å, β = 105·9 (1)°, D_m = 2·22, D_x = 2·28 Mg m^{-3} with Z = 4. The structure was determined from 1112 diffractometer-measured intensities and refined by least squares to R = 0·076. Hg is two-coordinated, Hg–C = 2·02 (2), Hg–N = 2·04 (2) Å, ∠C–Hg–N = 179 (1)°. There are no other contacts to Hg; a weak hydrogen bond N···O = 3·15 (3) Å between screw-axis-related molecules is suggested.

Introduction. Crystals of the title compound, kindly supplied by Dr F. G. Thorpe, were thin plates somewhat elongated parallel to **b** with {100} as the principal form. The space group and cell dimensions were obtained from Weissenberg photographs taken with Cu $K\alpha$ radiation (λ = 1·542 Å). From one crystal of dimensions 0·012 × 0·50 × 0·32 mm, mounted with **b** aligned on the ω axis of the Stoe STADI-2 automatic two-circle diffractometer, 1380 unique intensities were measured using graphite-monochromatized Mo $K\alpha$ radiation (λ = 0·7107 Å). Absorption corrections (μ = 14·36 mm^{-1}) were made with the *SHELX* program (Sheldrick, 1976). The structure determination and refinement were performed using XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972), only 1112 reflexions for which $I \geq 3\sigma(I)$ being included.

Coordinates of Hg, obtained from a Patterson map, were refined together with U_{ij} values and used to phase

* Present address: School of Chemistry, Brunel University, Uxbridge, England.

an ($F_o - F_c$) map which gave the positions of all the remaining non-H atoms. Refinement of atomic parameters proceeded using anisotropic U_{ij} for Hg and isotropic U for C, N and O. At an intermediate stage of refinement individual scale factors for the intensity layers $k = 0$ to 8 were refined with U_{22} restrained at a constant value to avoid excessive correlation. When the shifts on all parameters were less than 0·05 of the e.s.d. and there were no significant features on the ($F_o - F_c$)

Table 1. *Fractional atomic parameters* (×10^3, *for* Hg ×10^4) *and thermal parameters* (×10^3)

	x	y	z	U(Å^2)
N	33 (1)	537 (3)	290 (2)	34 (6)
O	53 (1)	742 (2)	184 (2)	53 (6)
C(1)	4 (2)	630 (4)	182 (4)	40 (8)
C(2)	–91 (2)	619 (3)	56 (3)	26 (6)
C(3)	–113 (2)	723 (4)	–64 (3)	45 (8)
C(4)	–205 (2)	718 (4)	–181 (3)	45 (8)
C(5)	–269 (2)	607 (4)	–175 (3)	48 (8)
C(6)	–245 (2)	516 (4)	–55 (4)	56 (9)
C(7)	–158 (1)	507 (3)	62 (2)	17 (6)
C(8)	288 (2)	581 (3)	632 (3)	37 (7)
C(9)	303 (2)	694 (3)	737 (3)	38 (7)
C(10)	397 (2)	713 (4)	852 (3)	50 (8)
C(11)	473 (2)	624 (4)	852 (3)	45 (8)
C(12)	459 (2)	515 (4)	742 (4)	57 (9)
C(13)	363 (2)	496 (4)	635 (4)	62 (10)
Hg	1617 (1)	5572 (1)	4615 (1)	*

* Anisotropic thermal parameters for Hg (×10^4)

U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
476 (7)	204 (9)	530 (7)	26 (6)	97 (5)	74 (6)