

$S = 1.133$   
 2054 reflections  
 172 parameters  
 H atoms treated by a  
 mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 3.7035P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)  
 Absolute structure:  
 Flack (1983)  
 Flack parameter =  $-0.01$  (2)

Tsukazaki, M., Tinkl, M., Roglans, A., Chapell, B. J., Taylor, N. J. & Snieckus, V. (1996). *J. Am. Chem. Soc.* **118**, 685.  
 Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

*Acta Cryst.* (1999). **C55**, 2047–2049

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C11	1.412 (3)	C1—C11	1.486 (4)
O1—C12	1.450 (3)	C12—C121	1.500 (4)
O2—C11	1.413 (3)	C12—C13	1.510 (4)
O2—C14	1.435 (4)	C13—C14	1.513 (4)
O3—C121	1.427 (4)		
C11—O1—C12	110.3 (2)	O1—C12—C121	107.9 (2)
C11—O2—C14	109.5 (2)	O1—C12—C13	109.2 (2)
C2—C1—C11	124.6 (3)	C121—C12—C13	114.9 (3)
C5—C1—C11	127.6 (3)	C12—C13—C14	110.5 (3)
O1—C11—O2	111.2 (2)	O2—C14—C13	110.8 (2)
O1—C11—C1	110.1 (2)	O3—C121—C12	111.9 (2)
O2—C11—C1	109.5 (2)		
C11—O1—C12—C13	57.5 (3)	C13—C14—O2—C11	-56.9 (3)
O1—C12—C13—C14	-51.1 (3)	C14—O2—C11—O1	64.1 (3)
C12—C13—C14—O2	51.6 (4)	O2—C11—O1—C12	-65.1 (3)

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O3—H3 $\cdots$ O3 <sup>i</sup>	0.76	2.01	2.720 (3)	156

Symmetry code: (i)  $-y, x, z$ .

The assignment of absolute configuration was confirmed by a Flack (1983) parameter of  $-0.01$  (2) from the final refinement.

Data collection: *IPDS Manual* (Stoe, 1996b). Cell refinement: *IPDS Manual*. Data reduction: *X-RED* (Stoe, 1996c). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *CAMERON* (Watkin *et al.*, 1996).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1052). Services for accessing these data are described at the back of the journal.

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## A long-chain alkyl-tethered $\text{Mo}_2\text{S}_2$ core, $[\text{Bu}_4\text{N}]_2[\text{Mo}_2(\text{CO})_8(n\text{-C}_6\text{H}_{13}\text{S})_2]$

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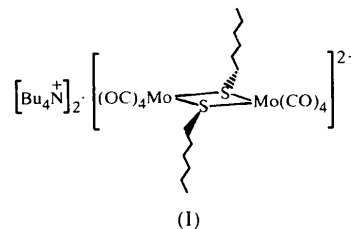
(Received 9 April 1999; accepted 27 August 1999)

## Abstract

The title compound, bis(tetrabutylammonium) bis( $\mu$ -hexanethiolato- $S:S$ ) bis [tetracarbonylmolybdenum(0)],  $(\text{C}_{16}\text{H}_{36}\text{N})_2[\text{Mo}_2(\text{C}_6\text{H}_{13}\text{S})_2(\text{CO})_8]$ , contains two  $\text{Bu}_4\text{N}^+$  cations and a centrosymmetric  $[\text{Mo}_2(\text{CO})_8(n\text{-C}_6\text{H}_{13}\text{S})_2]^{2-}$  anion. The structure of the anion can be viewed as a planar  $\text{Mo}_2\text{S}_2$  unit tethered by two long alkyl chains.

## Comment

Since 1984 low-valent dinuclear molybdenum thiolate complexes containing the  $\text{Mo}_2\text{S}_2$  unit and having two-electron transfer character, *e.g.*  $[\text{Mo}_2(\text{CO})_8(\text{SR})_2]^{2-}$  ( $R = \text{Ph}, \text{tBu}$ ), have been synthesized (Zhuang *et al.*, 1984; Smith *et al.*, 1987). Different  $R$  groups have been introduced into the  $\text{Mo}_2\text{S}_2$  unit, such as *m*-tolyl (Zhuang *et al.*, 1996), *p*-tolyl,  $\text{EtCO}_2\text{CH}_2$  (Zhuang *et al.*, 1986), to investigate the effect of the  $R$  group on the  $\text{Mo}_2\text{S}_2$  unit. The effects of long-chain purely alkyl  $R$  groups have interested us recently and the structure of the title compound, (I), which contains the *n*-hexyl  $[\text{CH}_3(\text{CH}_2)_4\text{CH}_2-]$  group, has been determined.



Compound (I) (Fig. 1) consists of three discrete structure fragments, namely two  $\text{Bu}_4\text{N}^+$  cations and the dianion  $[\text{Mo}_2(\text{CO})_8(n\text{-C}_6\text{H}_{13}\text{S})_2]^{2-}$ . The dianion comprises two equivalent Mo atoms, each of which is coordinated by four terminal carbonyls and two bridging *n*-hexane-

thiolate ligands forming a centrosymmetric  $Mo_2S_2$  core. The geometry of each  $Mo^0$  atom is a distorted octahedron. The bimetallic core is planar, with an  $Mo-S-Mo$  angle of  $99.7(2)^\circ$  and an  $Mo-S$  distance of  $2.602(9)$  Å. The long  $Mo \cdots Mo$  distance of  $3.9788(6)$  Å implies no metal-metal bonding. The two alkyl chains extend from the  $Mo_2S_2$  plane in opposite directions. The structure of the anion can be viewed as a planar  $Mo_2S_2$  unit tethered by two long chains. Compared with  $[Mo_2(CO)_8(SCH_2COOEt)_2]^{2-}$ , (II) (Zhuang *et al.*, 1986), and  $[Mo_2(CO)_8(PhS)_2]^{2-}$ , (III) (Zhuang *et al.*, 1989), the dimensions of the  $Mo_2S_2$  unit in (I) are similar to those in (II) [ $Mo-S-Mo$   $99.2(5)^\circ$ ,  $Mo-S$   $2.587(1)$  Å and  $Mo \cdots Mo$   $3.939(1)$  Å], and are slightly contracted relative to those in (III) [ $Mo-S-Mo$   $102.5(5)^\circ$ ,  $Mo-S$   $2.608(1)$  Å and  $Mo \cdots Mo$   $4.069(1)$  Å]. This implies that the length of the alkyl chain does not influence the conformation of the  $Mo_2S_2$  unit.

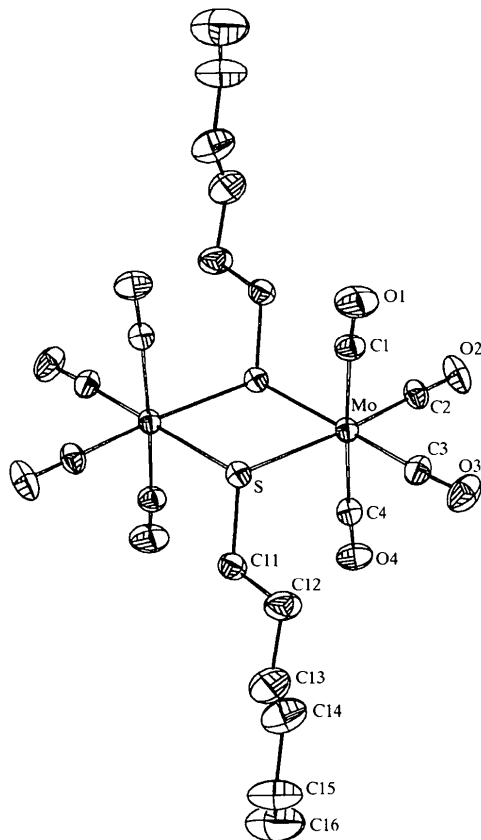


Fig. 1. View of the anion structure of the title compound with displacement ellipsoids shown at the 20% probability level and H atoms omitted for clarity.

## Experimental

The title compound was synthesized by the reaction of  $Mo(CO)_6$ ,  $Bu_4NBr$  and  $NaSCH_2(CH_2)_4CH_3$  (molar ratio 1:1:1) in MeCN, and was crystallized from MeCN/PrOH at a temperature below 273 K over a period of several days.

## Crystal data

$(C_{16}H_{36}N)_2[Mo_2(C_6H_{13}S)_2(CO)_8]$   
 $M_r = 1135.32$   
 Monoclinic  
 $P2_1/c$   
 $a = 12.4702(2)$  Å  
 $b = 15.8641(3)$  Å  
 $c = 16.9102(3)$  Å  
 $\beta = 109.0330(10)^\circ$   
 $V = 3162.43(8)$  Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.192$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 7613 reflections  
 $\theta = 1.73-25.10^\circ$   
 $\mu = 0.507$  mm<sup>-1</sup>  
 $T = 293(2)$  K  
 Needle  
 $0.76 \times 0.36 \times 0.36$  mm  
 Brown

## Data collection

Siemens SMART CCD diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (SADABS; Sheldrick, 1996)  
 $T_{min} = 0.650$ ,  $T_{max} = 0.875$   
 11 821 measured reflections  
 5415 independent reflections

4240 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.028$   
 $\theta_{max} = 25.10^\circ$   
 $h = -8 \rightarrow 14$   
 $k = -18 \rightarrow 11$   
 $l = -20 \rightarrow 15$   
 Intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.105$   
 $S = 1.143$   
 5415 reflections  
 298 parameters  
 H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.7794P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.406$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.338$  e Å<sup>-3</sup>  
 Extinction correction: none  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Mo—C2	1.925 (4)	Mo—S'	2.6000 (9)
Mo—C3	1.951 (4)	Mo—S	2.6045 (9)
Mo—C4	2.017 (4)	Mo $\cdots$ Mo'	3.9788 (6)
Mo—C1	2.046 (4)		
C2—Mo—C3	87.3 (2)	C4—Mo—S	90.76 (11)
C2—Mo—C4	89.4 (2)	C1—Mo—S	89.37 (12)
C3—Mo—C4	87.8 (2)	S'—Mo—S	80.28 (3)
C2—Mo—C1	91.0 (2)	C2—Mo $\cdots$ Mo'	133.46 (12)
C3—Mo—C1	86.9 (2)	C3—Mo $\cdots$ Mo'	139.28 (14)
C4—Mo—C1	174.6 (2)	C4—Mo $\cdots$ Mo'	92.02 (11)
C2—Mo—S'	93.28 (12)	C1—Mo $\cdots$ Mo'	91.55 (12)
C3—Mo—S'	179.45 (13)	S'—Mo $\cdots$ Mo'	40.18 (2)
C4—Mo—S'	92.33 (11)	S—Mo $\cdots$ Mo'	40.10 (2)
C1—Mo—S	93.00 (12)	C11—S—Mo'	108.35 (13)
C2—Mo—S	173.55 (12)	C11—S—Mo	110.94 (14)
C3—Mo—S	99.19 (14)	Mo'—S—Mo	99.72 (3)

Symmetry code: (i)  $1-x, 1-y, -z$ .

Data collection: SMART (Siemens, 1996). Cell refinement: SMART and SAINT (Siemens, 1994). Data reduction: XPREP in SHELXTL. Program(s) used to solve structure: SHELXTL (Sheldrick, 1994). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JA1002). Services for accessing these data are described at the back of the journal.

## References

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## Hexaaquazinc(II) bis[*N*-(4-amino-1-methyl-5-nitroso-6-oxo-1,6-dihydropyrimidin-2-yl)glycinate] dihydrate

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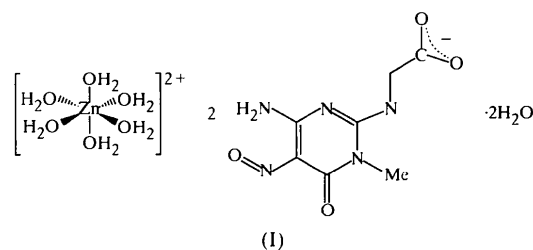
## Abstract

In the title compound,  $[\text{Zn}(\text{H}_2\text{O})_6](\text{C}_7\text{H}_8\text{N}_5\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ , the crystal structure consists of a stacking along the *b*

axis of hexaaquazinc(II) cations which lie on inversion centres, glycinate anions and water molecules.

## Comment

The title compound, (I), was obtained during our continuing research on the reactivity of metal ions with *N*-protected amino acids. The introduction of a substituent at the amino group in an  $\alpha$ -amino acid gives rise to *N*-protected amino acids, which are of interest for the following reasons: (i) the *N*-substituent (4-amino-1-methyl-5-nitroso-6-oxo-1,6-dihydropyrimidine) is an analogue of the potent antitumour agent 2,4-diamino-6-benzyloxy-5-nitrosopyrimidine (Pegg *et al.*, 1995; Loktinova & Pegg, 1996; Roy *et al.*, 1996); (ii) they are especially useful in directing peptide synthesis (Haurowitz, 1963) with markedly reduced racemization (Goldberg & Dice, 1974; Photaki, 1974); (iii) the involvement of these compounds in biological processes has been proved in many cases (Bonamartini Corradi, 1992); (iv) the possibility of modifying the chelating behaviour of the amino acid depending on the nature of the substituent (Arranz Mascarós *et al.*, 1999). We have found in previous studies that in the case of glycine with 4-amino-1-methyl-5-nitroso-6-oxo-1,6-dihydropyrimidine as the *N*-substituent, the amino acid residue acts as a monodentate ligand towards metal ions ( $\text{Mn}^{\text{II}}$ ,  $\text{Cu}^{\text{II}}$ ,  $\text{Zn}^{\text{II}}$  and  $\text{Cd}^{\text{II}}$ ) through the carboxylate group in mono- or bidentate mode (Arranz Mascarós, 1997). These compounds have tremendous versatility in the formation of hydrogen-bonded complexes because of the presence of numerous hydrogen-bond donor and acceptor groups, and it is well known that in the case of nucleic acid bases, these interactions determine the specificity of recognition between nucleic acid bases and DNA.



The crystal structure of the title complex consists of a stack along the *b* axis consisting of hexaaquazinc(II) cations, glycinate anions and water molecules (Fig. 1). The  $\text{Zn}^{\text{II}}$  atom is positioned on a centre of inversion and is hexacoordinated by water molecules with  $D_{2h}$  geometry.

The  $\text{Zn—O}$  bond lengths vary between 2.048 (1) and 2.143 (1) Å, and the  $\text{O—Zn—O}$  angles are close to 90° or 180°. These results are in agreement with usual values (Mak & Huang, 1987).

On the other hand, certain bond lengths differ from those found (values in brackets) in neutral *N*-(4-amino-