

Fig. 2. Newman projection along C(19)–C(20) of the three staggered conformations around this bond.

Therefore, right- and left-handed crystals should occur in approximately equal numbers. This is another example of spontaneous resolution of so-called switchable molecules (Rogers, 1975) into chiral crystals of either chirality.

We have not been able to verify the occurrence of the enantiomorphous crystal forms. The crystals are fine needles along [010] with the forms {001} and  $\bar{1}01$

dominating. No clear top faces enabling the distinction between the enantiomorphous forms were present. Because of the small amount of substance at our disposal, no recrystallization has been attempted.

I thank Professor F. Bickelhaupt and Dr R. J. M. Weusting for suggesting the investigation and providing the crystals.

#### References

- ALLEN, D. W., COPPOLA, J. C., KENNARD, O., MANN, F. G., MOTHERWELL, W. D. S. & WATSON, D. G. (1970). *J. Chem. Soc. C*, pp. 810–815.
- KENNARD, O., WAMPLER, D. K., COPPOLA, J. C., MOTHERWELL, W. D. S., MANN, F. G., WATSON, D. G., MACGILLAVRY, C. H., STAM, C. H. & BENCI, P. (1971). *J. Chem. Soc. C*, pp. 1511–1515.
- ROGERS, D. (1979). *Anomalous Scattering*, edited by S. RAMASESHAN & S. C. ABRAHAMS, p. 233. Copenhagen: Munksgaard.
- WEUSTING, R. J. M. (1977). Thesis, Amsterdam.

*Acta Cryst.* (1980). B36, 457–459

## The Structure of Tetrachlorotetrakis(diethyl sulfide)dimolybdenum(II)

BY F. ALBERT COTTON AND PHILLIP E. FANWICK

*Department of Chemistry, Texas A & M University, College Station, Texas 77843, USA*

(Received 7 August 1979; accepted 25 October 1979)

**Abstract.**  $[\text{Mo}_2(\text{C}_4\text{H}_{10}\text{S})_4\text{Cl}_4]$ ,  $\text{C}_{16}\text{H}_{40}\text{Cl}_4\text{Mo}_2\text{S}_4$ , monoclinic,  $Cc$ ,  $a = 18.769$  (5),  $b = 10.096$  (2),  $c = 15.793$  (6) Å,  $\beta = 109.94$  (2)°,  $V = 2813$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $d_x = 1.640$  Mg m<sup>-3</sup>,  $\mu(\text{Mo } K\alpha) = 1.537$  mm<sup>-1</sup>. The structure was solved using Patterson and Fourier techniques with diffractometer data and refined by least-squares methods to a final  $R$  of 0.041 based on 1567 independent observed reflections. The molecule possesses non-crystallographic symmetry and consists of two *trans* square-planar arrangements of chloride and sulfide ligands bound to each Mo atom and rotated by 90° with respect to each other. The Mo–Mo distance is 2.144 (1) Å.

**Introduction.** In 1974 the synthesis of a series of tetrahalodimolybdenum(II) dimers of the type  $\text{Mo}_2X_4L_n$ , where  $L$  is either a unidentate ( $n = 4$ ) or bidentate ( $n = 2$ ) sulfide or phosphine ligand, was reported (San Filippo, Sniadoch & Grayson, 1974). There has not yet been a structural characterization of any of these compounds. Since molecules of this type are quite

numerous it was considered worthwhile to determine the structure of at least one to serve as a benchmark.

Crystals of  $[\text{Mo}_2\{(\text{C}_2\text{H}_5)_2\text{S}\}_4\text{Cl}_4]$  were prepared by the literature method and were obtained as dark-blue tablets by evaporation of a methylene chloride solution. The crystals grew with prominent (001) faces. They were extremely dichroic, being dark blue when the plane of polarization was parallel to the  $c$  axis and nearly clear for  $a$  polarization. 2221 reflections were measured for  $0^\circ < 2\theta \leq 45^\circ$  on a Syntex  $P\bar{1}$  four-circle diffractometer using graphite-monochromated  $\text{Mo } K\alpha$  radiation and a  $\theta$ – $2\theta$  scan procedure. The crystal had dimensions  $0.5 \times 0.3 \times 0.1$  mm. No absorption correction was applied. The 1567 reflections with  $I > 3\sigma(I)$  were used for all refinements after  $L_p$  corrections were applied. The cell dimensions were based on the centering of 15 strong reflections with  $16^\circ < 2\theta < 30^\circ$ .

Systematic absences indicated space groups  $Cc$  (No. 9) or  $C2/c$  (No. 15). Since a Howells–Phillips–Rogers test strongly indicated that the unit cell was noncentrosymmetric, refinement was commenced with

Table 1. Positional parameters and their estimated standard deviations

	x	y	z
Mo(1)	0.7508 (1)	0.14994 (7)	0.4990 (2)
Mo(2)	0.7500	0.36233 (7)	0.5000
Cl(1)	0.8155 (2)	0.0771 (3)	0.6504 (2)
Cl(2)	0.6821 (2)	0.0695 (3)	0.3493 (2)
Cl(3)	0.6391 (2)	0.4374 (4)	0.5304 (2)
Cl(4)	0.8618 (1)	0.4386 (3)	0.4715 (2)
S(1)	0.8768 (2)	0.1095 (4)	0.4795 (2)
S(2)	0.6247 (2)	0.1182 (3)	0.5219 (2)
S(3)	0.6873 (2)	0.3983 (3)	0.3325 (2)
S(4)	0.8132 (2)	0.4002 (3)	0.6669 (2)
C(1)	0.8691 (7)	0.151 (1)	0.3625 (8)
C(2)	0.9424 (9)	0.133 (2)	0.3434 (11)
C(3)	0.8867 (10)	-0.069 (2)	0.4864 (13)
C(4)	0.8560 (10)	-0.156 (2)	0.4314 (12)
C(5)	0.6350 (7)	0.122 (1)	0.6400 (9)
C(6)	0.5519 (10)	0.159 (2)	0.6400 (12)
C(7)	0.5886 (7)	-0.056 (1)	0.4979 (8)
C(8)	0.6614 (18)	-0.138 (3)	0.5574 (21)
C(9)	0.7061 (8)	0.578 (2)	0.3195 (10)
C(10)	0.6691 (12)	0.668 (2)	0.3703 (15)
C(11)	0.5867 (7)	0.378 (1)	0.3055 (9)
C(12)	0.5511 (10)	0.398 (2)	0.1986 (13)
C(13)	0.8074 (6)	0.573 (1)	0.6910 (7)
C(14)	0.8363 (7)	0.673 (1)	0.6358 (9)
C(15)	0.9159 (7)	0.387 (1)	0.7038 (9)
C(16)	0.9595 (8)	0.431 (2)	0.7940 (10)

Table 2. Bond distances (Å) and angles (°) in  $[\text{Mo}_2\{(\text{C}_2\text{H}_5)_2\text{S}\}_4\text{Cl}_4]$ 

Mo(1)—Mo(2)	2.144 (1)		
Mo(1)—Cl(1)	2.398 (7)	Mo(2)—Cl(3)	2.413 (7)
—Cl(2)	2.415 (7)	—Cl(4)	2.417 (6)
—S(1)	2.522 (7)	—S(3)	2.528 (7)
—S(2)	2.532 (7)	—S(4)	2.523 (7)
S(1)—C(1)	1.85 (2)	S(3)—C(9)	1.87 (2)
S(1)—C(3)	1.81 (2)	S(3)—C(11)	1.80 (2)
C(1)—C(2)	1.52 (2)	C(9)—C(10)	1.53 (4)
C(3)—C(4)	1.23 (3)	C(11)—C(12)	1.60 (3)
S(2)—C(5)	1.81 (2)	S(4)—C(13)	1.80 (2)
S(2)—C(7)	1.87 (1)	S(4)—C(15)	1.82 (2)
C(5)—C(6)	1.61 (3)	C(13)—C(14)	1.55 (2)
C(7)—C(8)	1.60 (4)	C(15)—C(16)	1.45 (3)
Mo(2)—Mo(1)—Cl(1)	107.6 (2)	Mo(1)—Mo(2)—Cl(3)	108.9 (2)
—Cl(2)	109.9 (2)	—Cl(4)	108.0 (2)
—S(1)	99.9 (2)	—S(3)	97.9 (2)
—S(2)	96.7 (2)	—S(4)	99.0 (2)
Cl(1)—Mo(1)—Cl(2)	142.42 (9)	Cl(3)—Mo(2)—Cl(4)	143.10 (8)
—S(1)	84.0 (2)	—S(3)	92.2 (2)
—S(2)	90.0 (2)	—S(4)	82.8 (2)
Cl(2)—Mo(1)—S(1)	92.0 (2)	Cl(4)—Mo(2)—S(3)	83.3 (2)
—S(2)	83.4 (2)	—S(4)	91.0 (2)
S(1)—Mo(1)—S(2)	163.4 (1)	S(3)—Mo(2)—S(4)	163.04 (8)
Mo(1)—S(1)—C(1)	109.1 (6)	Mo(2)—S(3)—C(9)	102.5 (7)
Mo(1)—S(1)—C(3)	103.6 (7)	Mo(2)—S(3)—C(11)	107.9 (7)
Mo(1)—S(2)—C(5)	111.6 (6)	Mo(2)—S(4)—C(13)	109.1 (6)
Mo(1)—S(2)—C(7)	112.4 (5)	Mo(4)—S(4)—C(15)	113.1 (7)
S(1)—C(1)—C(2)	113 (1)	S(3)—C(9)—C(10)	112 (2)
S(1)—C(3)—C(4)	131 (2)	S(3)—C(11)—C(12)	105 (1)
S(2)—C(5)—C(6)	104 (1)	S(4)—C(13)—C(14)	117 (1)
S(2)—C(7)—C(8)	101 (1)	S(4)—C(15)—C(16)	118 (1)

space group *Cc*. The structure was solved with the *Enraf-Nonius Crystal Structure Determination Package*. The weighting scheme used was  $w = Lp^2 / [\sigma(I)^2 + (0.07I)^2]$  and the function minimized during least-squares refinement was  $\sum w(|F_o| - |F_c|)^2$ . Refinement converged with  $R_1 = \sum(|F_o| - |F_c|) / \sum |F_o| = 0.041$ ,  $R_2 = [\sum w(|F_o| - |F_c|)^2 / \sum w F_o^2]^{1/2} = 0.063$  and the estimated standard deviation of an observation of unit weight equal to 1.533. A final difference Fourier map revealed no peaks greater than  $0.73 \text{ e } \text{Å}^{-3}$ .

The positional parameters for  $[\text{Mo}_2\{(\text{C}_2\text{H}_5)_2\text{S}\}_4\text{Cl}_4]$  are listed in Table 1. The bond distances and angles are given in Table 2. Fig. 1 shows the molecule and the numbering scheme used.\*

**Discussion.** The non-crystallographic symmetry of the  $[\text{Mo}_2\{(\text{C}_2\text{H}_5)_2\text{S}\}_4\text{Cl}_4]$  molecule is  $D_{2d}$  as would be expected, although there is no crystallographically required symmetry. This geometry minimizes the steric interference of the bulky diethyl sulfide ligands. The Mo—Mo distance of 2.144 (1) Å is quite similar to those in several salts of  $[\text{Mo}_2\text{Cl}_8]^{4-}$  where distances of 2.139 Å for  $\text{K}_4\text{Mo}_2\text{Cl}_8 \cdot 2\text{H}_2\text{O}$  (Brencic & Cotton,

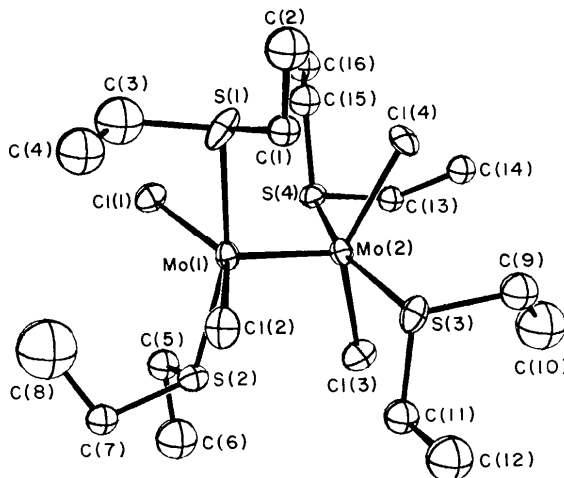


Fig. 1. A computer-drawn representation of the molecule, showing the numbering scheme. Thermal ellipsoids enclose 40% of the electron density.

1969) to 2.150 Å in  $(\text{NH}_4)_4\text{Mo}_2\text{Cl}_8 \cdot (\text{NH}_4)\text{Cl}_4 \cdot \text{H}_2\text{O}$  (Brencic & Cotton, 1970) have been found, and to that in  $[\text{Mo}_2\{(\text{C}_6\text{H}_5)_3\text{P}\}_2(\text{CH}_3\text{OH})_2\text{Cl}_4]$  (McGinnis, Ryan & McCarley, 1978), 2.143 Å. These are among the longest distances observed in quadruply bonded molybdenum dimers. The molybdenum—chlorine angles in  $[\text{Mo}_2\{(\text{C}_2\text{H}_5)_2\text{S}\}_4\text{Cl}_4]$  averaged to  $108.6^\circ$  as compared

\* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34868 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

to  $104.7^\circ$  for  $[\text{Mo}_2\text{Cl}_8]^{4-}$ . The mean S—Cl distance between the eclipsed ligands is  $3.29(5) \text{ \AA}$  which is quite comparable to the  $3.38(2) \text{ \AA}$  Cl—Cl distance in  $[\text{Mo}_2\text{Cl}_8]^{4-}$ .

The structure reported here is probably typical for all of the monodentate sulfide and phosphine compounds of formula  $\text{Mo}_2\text{X}_4\text{L}_4$ . These compounds are all dark blue and have nearly identical electronic spectra and physical properties. Attempts to obtain crystals of the presumed chelated complexes of the blue-green bidentate sulfides were unsuccessful.

*Acta Cryst.* (1980). **B36**, 459–461

### The Structure of Rubidium Hydrogen Bis(dibromoacetate)

BY V. VIDENOVA, J. BARAN, T. GŁOWIAK AND H. RATAJCZAK

*Institute of Chemistry, Wrocław University, 50-383 Wrocław, Joliot Curie 14, Poland*

(Received 23 July 1979; accepted 30 October 1979)

**Abstract.**  $\text{RbH}(\text{C}_2\text{HBr}_2\text{O}_2)_2$ ,  $\text{Rb}^+ \cdot \text{C}_4\text{H}_3\text{Br}_2\text{O}_4^-$ , FW = 520.15, monoclinic,  $P2_1/n$ ,  $a = 6.299(3)$ ,  $b = 20.958(5)$ ,  $c = 4.446(3) \text{ \AA}$ ,  $\beta = 98.05(3)^\circ$ ,  $V = 581.2 \text{ \AA}^3$ ,  $Z = 2$ ,  $\mu = 19.04 \text{ mm}^{-1}$ ,  $\lambda(\text{Mo } K\alpha)$ ,  $D_c = 2.98 \text{ Mg m}^{-3}$ . Final  $R = 0.074$  for 741 reflections with  $I \geq 1.92 \sigma(I)$ . The crystal has a layer structure. Within the layers [parallel to the (010) plane] the hydrogen bis(dibromoacetate) anions are connected by  $\text{Rb}^+$  cations. The two dibromoacetate residues are linked by a symmetric (i) hydrogen bond with  $\text{O} \cdots \text{O} = 2.43(2) \text{ \AA}$ .

**Introduction.** We have determined the crystal structures of caesium (Głowiak, Videnova, Baran & Ratajczak, 1980) and potassium hydrogen bis(dibromoacetate) (Baran, Videnova, Głowiak & Ratajczak, 1979). In these crystals strong hydrogen bonds between two dibromoacetate residues are present with  $\text{O} \cdots \text{O} = 2.50(2)$  and  $2.44(3) \text{ \AA}$  respectively. Now we report the crystal structure of the title compound.

Crystals of  $\text{RbH}(\text{C}_2\text{HBr}_2\text{O}_2)_2$  were prepared by adding dibromoacetic acid to  $\text{Rb}_2\text{CO}_3$  in 4:1 molar ratio in an excess of  $\text{H}_2\text{O}$ . The solution was then evaporated to form colourless crystals.

The cell dimensions and intensities were measured on a Syntex  $P2_1$  four-circle diffractometer with a graphite monochromator and  $\text{Mo } K\alpha$  radiation. The lattice parameters and orientation matrix used for data collection were obtained by least squares from 15 reflections. Intensities of 1081 reflections were collected

- #### References
- BRENCIC, J. V. & COTTON, F. A. (1969). *Inorg. Chem.* **8**, 7–10.  
 BRENCIC, J. V. & COTTON, F. A. (1970). *Inorg. Chem.* **9**, 346–351.  
 MCGINNIS, R. N., RYAN, T. R. & MCCARLEY, R. E. (1978). *J. Am. Chem. Soc.* **100**, 7900–7902.  
 SAN FILIPPO, J., SNIADACH, H. J. & GRAYSON, R. L. (1974). *Inorg. Chem.* **13**, 2121–2130.

by the  $\theta$ – $2\theta$  scan technique to  $2\theta = 60^\circ$ . 741 reflections with  $I \geq 1.92\sigma(I)$  were accepted for further calculations. Standard reflections were measured every 15 reflections and showed no significant change. The data were corrected for Lorentz and polarization factors, but not for absorption. The structure was solved by the heavy-atom technique. The positions of all non-H atoms were found from a Patterson synthesis and from difference maps. The positions of the H atoms were not found. The refinement gave  $R_1 = 0.087$  and  $R_2 = 0.072$ . The function minimized during refinement was  $\sum w(|F_o| - |F_c|)^2$  with  $w = 1/\sigma^2(F)$ . Scattering factors were taken from *International Tables for X-ray*

Table 1. *The atomic coordinates ( $\times 10^4$ ) with e.s.d.'s in parentheses*

	x	y	z
Rb	0	0	0
Br(1)	3209 (4)	3598 (1)	577 (6)
Br(2)	–1058 (5)	2967 (1)	2161 (6)
O(1)	596 (25)	4494 (6)	4071 (30)
O(2)	–2281 (28)	4516 (7)	488 (34)
C(1)	–597 (40)	4292 (10)	1690 (54)
C(2)	201 (36)	3662 (9)	138 (46)
Symmetry code			
(i)	0.00 + x,	0.00 + y,	0.00 + z
(ii)	0.50 – x,	0.50 + y,	0.50 – z
(iii)	0.00 – x,	0.00 – y,	0.00 – z
(iv)	–0.50 + x,	–0.50 – y,	–0.50 + z