[with Si(3)]. Most Si–O bond distances are normal, with an average value of 1·65 Å. Also the O–Si–O bond angles (see Table 4) are quite normal with an average value of 109°4′. The Si(1)–O(8)–Si(2) bond angle is 133°. In the tetrahedron containing Si(1) there is some deviation from the normal value: the Si(1)–O(8) distance is somewhat higher (1·69–0·02 Å) and Si(1)–O(1) lower (1·60 ± 0·03 Å).

The experimental error alone seems an inadequate explanation for this deviation. To obtain a regular tetrahedron around Si(1) it would be necessary to admit a 2σ error in both Si–O distances, and this is highly improbable. We think that the rather short distance Ca(1‴)–Si(1) (3·10 Å), the shortest distance Ca–Si in this structure, could explain the shortening of the Si(1)–O(1) bond. It should be noted also that the distance Al″–O(1) is the shortest of its kind (see Fig. 2). Besides, a lateral shift of Si(1) towards O(8) would be prohibited by two atoms Ca(2) and by Ca(1‴).

The coordination of seven oxygen atoms around the calcium atoms is quite simple: Ca(1) is at the centre of a square [O(2), O(2^{v}), O(3), O(3^{v})] parallel to **b**; two other oxygen atoms, O(4) and O(8‴), lie close to the calcium atom at one side, and one oxygen atom, O(11), at the other.

The same situation holds for Ca(2); it is placed in a square [O(1), O(1^{v}), O(2), O(2^{v})] with two oxygen atoms at one side [O(6), O(9)], and one at the other [O(10″)].

The 14 Ca–O distances average 2·43 ± 0·12 Å which is smaller than usual.

It is worth noting that, although the whole refinement was carried out with isotropic temperature factors, some trials with difference syntheses revealed qualitatively strong anisotropic movements of calcium atoms in the direction normal to the coordination square. The low precision of intensity measurement mentioned above discouraged us from attempting any quantitative measurement of this anisotropic temperature factor.

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The Crystal and Molecular Structure of μ-Oxobis[bis(diethyldithiophosphato)oxomolybdenum(V)] 1,2-Dichlorobenzene, $Mo_2O_3[S_2P(OC_2H_5)_2]_4.2C_6H_4Cl_2$

By J. R. Knox* and C. K. Prout

Chemical Crystallography Laboratory, South Parks Road, Oxford, England

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$Mo_2O_3[S_2P(OC_2H_5)_2]_4.2C_6H_4Cl_2$ crystallizes in space group $P\bar{1}$ with $a = 11{\cdot}36 \pm 0{\cdot}01$, $b = 13{\cdot}96 \pm 0{\cdot}01$, $c = 8{\cdot}66 \pm 0{\cdot}01$ Å, $\alpha = 94{\cdot}9 \pm 0{\cdot}2°$, $\beta = 103{\cdot}8 \pm 0{\cdot}2°$, $\gamma = 81{\cdot}7 \pm 0{\cdot}2°$ and with $Z = 1$. Least-squares refinement with anisotropic temperature factors gave an R index of 0·117 for 3200 non-zero photographic data. The diamagnetic binuclear molecule consists of two distorted octahedra which share a linear bridging oxygen atom situated at an inversion centre. The molybdenum to bridging oxygen atom bond length is 1·863 Å and to the terminal oxygen atom, 1·647. The Mo–S bond *trans* to the terminal oxygen atom is 2·801 Å and the other Mo–S bonds have a mean length of 2·496 Å.

Introduction

Oxidation of square-pyrimidal $MoO(S_2P(OC_2H_5)_2]_2$ (Jowitt & Mitchell, 1966) gives $Mo_2O_3[S_2P(OC_2H_5)_4]_4$, the structure of which we now report.

Experimental

A compound with the formulation $MoO(dtp)_2$, where $dtp = S_2P(OC_2H_5)_2$, diethyldithiophosphate anion was supplied by R. N. Jowitt and P. C. H. Mitchell of Reading University. Recrystallization under nitrogen with 1,2-dichlorobenzene produced ligth-brown transparent crystals. When dried in air they became opaque and

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* Present address: Molecular Biophysics Department, Yale University, New Haven, Connecticut, U.S.A.

their colour and diffraction pattern changed. Photography of a crystal which was in contact with dichlorobenzene while sealed in a glass capillary gave the following triclinic cell dimensions:
$a=11{\cdot}36\pm0{\cdot}01$, $b=13{\cdot}96\pm0{\cdot}01$, $c=8{\cdot}66\pm0{\cdot}01$ Å, $\alpha=94\pm0{\cdot}2$, $\beta=103{\cdot}8\pm0{\cdot}2$, $\gamma=81{\cdot}7\pm0{\cdot}2°$.

In spite of precautions oxidation had apparently occurred during recrystallization, for the crystal data established by the structure determination are: $Mo_2O_3(dtp)_4.2C_6H_4Cl_2$, $M=1275$, $F(000)=644$; D_m (dried crystals) $=1{\cdot}63$ g.cm^{-3}, $Z=1$, $D_c=1{\cdot}61$ g.cm^{-3}; $\mu=106$ cm^{-1} for Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å), $\mu=$

Table 1. *Fractional atomic coordinates* (×10⁴) *and thermal parameters* (Å² × 10³) *with standard deviations in parentheses*

Anisotropic coefficients have the form:

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{23}klb^*c^*+2U_{31}lhc^*a^*+2U_{12}hka^*b^*)\right].$$

	x	y	z
Mo	736 (1·1)	4171 (0·9)	3601 (1·5)
S(1)	−520 (4)	2846 (4)	3606 (6)
S(2)	1854 (4)	3201 (4)	6380 (6)
S(3)	2283 (4)	2999 (3)	2448 (6)
S(4)	2503 (4)	5063 (3)	4277 (5)
P(1)	548 (5)	2392 (4)	5681 (6)
P(2)	3367 (4)	4029 (3)	3018 (5)
O(1)	−254 (22)	2354 (19)	6859 (23)
O(2)	900 (19)	1303 (11)	5545 (23)
O(3)	4706 (10)	3676 (10)	3931 (16)
O(4)	3697 (10)	4448 (9)	1563 (14)
O(5)	0	5000	5000
O(6)	−76 (11)	4498 (10)	1839 (15)
C(1)	−148 (28)	2741 (20)	8387 (29)
C(2)	−851 (32)	2205 (28)	9229 (39)
C(3)	1585 (33)	863 (22)	4334 (52)
C(4)	1867 (39)	−182 (23)	4462 (55)
C(5)	4958 (19)	3246 (19)	5436 (29)
C(6)	6056 (24)	2479 (19)	5513 (42)
C(7)	2713 (16)	4809 (16)	264 (22)
C(8)	3261 (23)	5253 (21)	−848 (27)
C(9)	4325 (18)	−667 (15)	11253 (26)
C(10)	3319 (21)	−702 (17)	10044 (30)
C(11)	3188 (22)	−1519 (17)	8966 (31)
C(12)	4167 (26)	−2255 (19)	9186 (34)
C(13)	5235 (23)	−2173 (18)	10368 (32)
C(14)	5361 (24)	−1417 (19)	11428 (36)
Cl(1)	4507 (11)	313 (9)	12648 (14)
Cl(2)	2100 (13)	248 (10)	9944 (17)

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Mo	42 (1)	51 (1)	46 (1)	−6 (1)	27 (1)	−8 (1)
S(1)	70 (3)	72 (3)	81 (3)	−14 (5)	25 (5)	−60 (5)
S(2)	66 (3)	73 (3)	57 (3)	9 (4)	29 (4)	−29 (4)
S(3)	63 (2)	52 (2)	72 (3)	−17 (4)	58 (4)	−10 (4)
S(4)	52 (2)	55 (2)	63 (3)	−14 (4)	37 (4)	−27 (3)
P(1)	100 (4)	65 (3)	59 (3)	−21 (4)	69 (5)	−54 (5)
P(2)	43 (2)	63 (2)	49 (2)	11 (4)	30 (3)	0 (3)
O(1)	192 (20)	251 (25)	108 (14)	−129 (31)	193 (29)	−293 (38)
O(2)	165 (17)	70 (10)	133 (15)	18 (19)	81 (26)	−50 (21)
O(3)	43 (6)	102 (10)	82 (9)	63 (15)	19 (11)	10 (12)
O(4)	50 (6)	86 (8)	63 (7)	33 (12)	35 (10)	−10 (11)
O(5)	46 (8)	53 (9)	102 (13)	33 (17)	41 (16)	−2 (14)
O(6)	56 (7)	92 (9)	75 (8)	34 (14)	−2 (12)	−12 (12)
C(1)	165 (25)	124 (21)	78 (17)	−47 (30)	156 (34)	−48 (36)
C(2)	145 (28)	181 (34)	130 (24)	−17 (46)	73 (46)	−146 (50)
C(3)	160 (29)	89 (19)	242 (42)	10 (45)	221 (60)	41 (38)
C(4)	210 (37)	88 (20)	278 (46)	−85 (51)	242 (70)	−961 (45)
C(5)	68 (13)	128 (20)	106 (18)	90 (30)	25 (24)	53 (26)
C(6)	95 (18)	95 (17)	216 (32)	156 (39)	78 (38)	26 (28)
C(7)	58 (10)	116 (16)	49 (11)	46 (21)	−6 (16)	−11 (20)
C(8)	104 (17)	148 (22)	73 (15)	98 (29)	−15 (25)	−66 (31)

	U_{iso}		U_{iso}		U_{iso}
Cl(1)	183 (4)	C(9)	107 (8)	C(12)	137 (11)
Cl(2)	213 (5)	C(10)	135 (10)	C(13)	124 (9)
		C(11)	132 (10)	C(14)	163 (13)

102 cm^{-1} for Mo $K\alpha$ radiation ($\lambda=0\cdot7107$ Å). Space group: $P\bar{1}$ (C_i^1, No. 2). Molecule symmetry: $\bar{1}$.

From a crystal $0\cdot15+0\cdot30\times0\cdot60$ mm equi-inclination Weissenberg photography of the *hk*0 to *hk*7 layers with Cu $K\alpha$ radiation produced 3363 observable reflexions. Precession photography of the same crystal with Mo $K\alpha$ radiation gave 129 *h*0*l* and 151 0*kl* reflexions. The visually-estimated data were corrected for Lorentz-polarization effects but not for absorption. The 163 common reflexions were used to obtain layer scale factors by the method of Hamilton, Rollett & Sparks (1965) in which weights proportional to F_o^2 were used. The merging R index $\Sigma|F_i^2-\overline{F^2}|/\Sigma\overline{F^2}$ was 0·083; 3200 independent non-zero reflexions were obtained.

Solution and refinement of the structure

Square-pyramidal coordination around molybdenum was immediately ruled out by the Patterson function, which gave vectors consistent with an oxo-bridged binuclear molecule. The structure factor calculation based on the molybdenum, sulphur and phosphorus atoms led to an R index $\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$ of 0·25. Remaining ligand atoms, together with a molecule of 1,2-dichlorobenzene, were found in Fourier and difference syntheses.

In the least-squares refinement the quantity minimized was $\Sigma w(F_o-F_c)^2$ where $w=[1+[(F_o-a)/b]^2]^{-1}$ with $a=18$ and $b=20$ electrons. Scattering curves were taken from *International Tables for X-ray Crystallography* (1962); the curve for neutral molybdenum was corrected for the real part of the dispersion effect ($f'=-0\cdot5\,e$).

Unobserved reflexions were omitted from the refinement. After four cycles of full-matrix refinement with isotropic temperature factors, parameter shifts were less than their estimated standard errors, and the R index was 0·14. At this stage the C–C bond distances in the dichlorobenzene molecule ranged from 1·26 to 1·56 Å. An F_o map calculated in the plane of the ring confirmed that the solvent molecule was ill-defined. A semi-rigid configuration was therefore imposed on the molecule and the calculated C–C and C–Cl distances, d were fitted to pre-set values d' (1·395 and 1·776 Å) in a conditional least-squares procedure (Waser, 1963; Ford & Rollett, personal communication; Knox & Prout, 1969*a*) which minimized $\Sigma w(F_o-F_c)^2+w'(d-d')^2$. After anisotropic temperature factors were assigned to all but dichlorobenzene atoms, the block-diagonal refinement converged in six cycles to an R index of 01·17 for all 3200 reflexions. A table of structure amplitudes and calculated structure factors may be obtained from C.K.P. Layer scale factors were not varied during the anisotropic refinement. Final atomic coordinates and temperature parameters are given in Table 1. A difference map showed no unusual features. The distances d were held to within $1\cdot5\sigma$ of d' by the weights w' which were $(0\cdot035)^{-2}$ for C–C distances and $(0\cdot030)^{-2}$ for C–Cl distances.

Discussion

The molecule consists of two octahedra which share a common oxygen atom situated on the space group inversion centre (Fig. 1). The Mo–O–Mo bridge is linear and the Mo–O (terminal) bonds are *trans*, as required by crystal symmetry. A molecular orbital treatment (Blake, Cotton & Wood, 1964) shows that the magnetic properties of linear-bridged compounds depends on the angle of twist of the two octahedra about the Mo–O–Mo axis. Only if the angle between the two

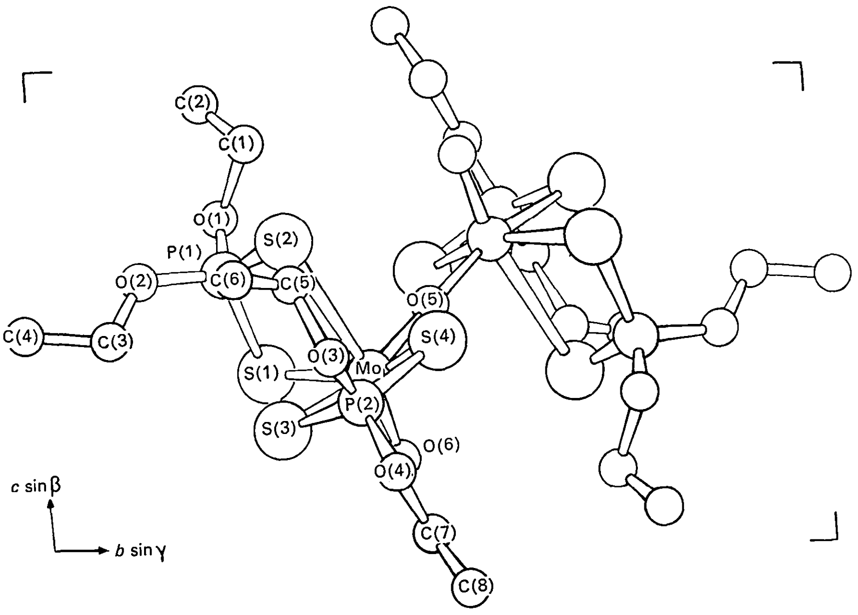

Fig. 1. The $Mo_2O_3[S_2P(OC_2H_5)_2]_4$ molecule viewed along **a**.

Mo–O (terminal) bonds, as viewed down the bridge, is near 0° (or 180°) can electron spin coupling occur to give the observed diamagnetism.

The Mo–O (bridge) bond is 1·86 Å and equals the average distance in the structurally similar xanthate compound $Mo_2O_3(S_2COC_2H_5)_4$ (Blake, Cotton & Wood, 1964), but it is shorter than the 1·91–1·95 Å bonds in the doubly bridged cysteinate

$$Na_2Mo_2O_4[SCH_2CH(NH_2)CO_2]_2$$

(Knox & Prout, 1969*b*) because the linear bridge allows better π-bonding. The second type of Mo–O bond, to the terminal oxygen atom O(6), involves considerable multiple-bond character. Its length (1·65 Å) equals the distance in the xanthate compound. The obtuse O–Mo–O angle (103°) is a result of repulsion between electrons in the short Mo=O and Mo–O bonds; the Mo=O bond makes its largest O–Mo–S angle (108°) with the shortest Mo–S bond.

Only weak bonding is possible to donors which are *trans* to the Mo=O bond (Cotton & Wing, 1965), and here the Mo–S(2) distance is exceptionally longer (2·80 Å) than the other three Mo–S distances. The donor strength of the phosphate ligand is less than that of the xanthate ligand because a part of the anionic charge of the former can move into the *d* orbitals of the phosphorus to avoid the charge build-up on the metal which results from $p\pi\rightarrow d\pi$ bonding in the opposite Mo=O bond. Consequently, the Mo–S(2) bond is 0·1 Å longer than the corresponding bond in

$$Mo_2O_3(S_2COC_2H_5)_4.$$

The next longest bond (2·55 Å) is to the sulphur atom which is opposite the bridge oxygen atom. The mutually *trans* bonds to S(1) and to S(4) are 2·50 and 2·44 Å, the smaller being equivalent to the bond lengths in $(C_5H_5)_2MoS_2C_6H_3CH_3$ (Knox & Prout, 1969*a*) and $(C_5H_5)_2MoS(CH_2)_2HN_2^+I^-$ (Knox & Prout, 1969*c*).

Table 2. *Interatomic distances* (Å, $\sigma\times10^3$) *and angles* (deg, $\sigma\times10$)

Mo–S(1)	2·497 (6)	O(6)–Mo–S(1)	92·0 (5)
Mo–S(2)	2·801 (5)	O(6)–Mo–S(3)	93·7 (5)
Mo–S(3)	2·547 (5)	O(6)–Mo–S(4)	107·5 (5)
Mo–S(4)	2·442 (5)	O(6)–Mo–O(5)	103·0 (10)
Mo–O(5)	1·863 (12)	S(2)–Mo–S(1)	74·7 (2)
Mo–O(6)	1·647 (14)	S(2)–Mo–S(3)	82·6 (2)
		S(2)–Mo–S(4)	85·0 (2)
S(1)–Mo–S(4)	158·6 (2)	S(2)–Mo–O(5)	82·5 (5)
S(2)–Mo–O(6)	166·1 (5)	S(1)–Mo–S(3)	91·0 (2)
S(3)–Mo–O(5)	162·2 (5)	S(3)–Mo–S(4)	79·2 (2)
		S(4)–Mo–O(5)	89·9 (5)
		O(6)–Mo–S(1)	94·4 (5)

P(1)—S(1)	2·010 (8)	P(2)—S(3)	1·983 (7)
P(1)—S(2)	1·947 (8)	P(2)—S(4)	2·017 (6)
P(1)—O(1)	1·530 (27)	P(2)—O(3)	1·568 (15)
P(1)—O(2)	1·517 (22)	P(2)—O(4)	1·576 (14)
O(1)—C(1)	1·370 (42)	O(3)—C(5)	1·430 (30)
O(2)—C(3)	1·491 (50)	O(4)—C(7)	1·451 (26)
C(1)—C(2)	1·508 (50)	C(5)—C(6)	1·516 (45)
C(3)—C(4)	1·456 (66)	C(7)—C(8)	1·483 (37)
S(1)—P(1)—S(2)	109·2 (4)	S(3)—P(2)—S(4)	105·3 (3)
O(1)—P(1)—O(2)	94·9 (13)	O(3)—P(2)—O(4)	97·4 (7)
Mo —S(1)—P(1)	91·7 (3)	Mo —S(3)—P(2)	86·3 (2)
Mo —S(2)—P(1)	84·4 (2)	Mo —S(4)—P(2)	88·4 (2)
P(1)—O(1)—C(1)	130·3 (24)	P(2)—O(3)—C(5)	121·7 (14)
P(1)—O(2)—C(3)	120·3 (23)	P(2)—O(4)—C(7)	118·8 (12)
O(1)—C(1)—C(2)	108·9 (28)	O(3)—C(5)—C(6)	107·8 (22)
O(2)—C(3)—C(4)	110·8 (35)	O(4)—C(7)—C(8)	107·8 (19)
C(9)—Cl(1)	1·748 (26)	Cl(1)–C(9)—C(10)	122·9 (19)
C(10)–Cl(2)	1·765 (30)	Cl(1)–C(9)—C(14)	114·8 (19)
C(9)—C(10)	1·358 (34)	C(14)–C(9)—C(10)	122·2 (23)
C(10)–C(11)	1·412 (37)	Cl(2)–C(10)–C(11)	119·8 (20)
C(11)–C(12)	1·390 (40)	Cl(2)–C(10)–C(9)	118·6 (20)
C(12)–C(13)	1·401 (41)	C(9)—C(10)–C(11)	121·3 (23)
C(13)–C(14)	1·339 (42)	C(10)–C(11)–C(12)	115·9 (25)
C(14)–C(9)	1·446 (38)	C(11)–C(12)–C(13)	121·8 (27)
		C(12)–C(13)–C(14)	123·0 (27)
		C(13)–C(14)–C(9)	115·5 (26)

C(6)···C(4) $(1-x, \bar{y}, 1-z)$	3·70
C(8)···C(8) $(1-x, 1-y, \bar{z})$	3·86
C(2)···C(11) $(\bar{x}, \bar{y}, 2-z)$	3·66
C(6)···C(14) $(1-x, \bar{y}, 2-z)$	3·90
C(8)···C(12) $(x, 1+y, z-1)$	3·87
C(9)···C(9) $(1-x, \bar{y}, 2-z)$	3·69

The bond lengths of the four P–S bonds vary inversly with the lengths of the adjoining Mo–S bonds, as required by conservation of bond order around the sulphur atom. The P–S distances range from 1·95 to 2·02 Å and differ significantly within the same ligand. Averaging of the four distances gives 1·99 Å, which is the distance in the square-planar Ni(dtp)$_2$ structure (McConnell & Kastralsky, 1967). Metal coordination through sulphur has lengthened by 0·06 Å the P–S bond associated with the shortest Mo–S bond, for the distance is 1·96 Å in K[$S_2P(OCH_3)_2$] (Coppens, MacGillavry, Hovenkamp & Douwes, 1962). The length of the P–S bond adjoining the longest Mo–S bond is equal to the distance in the potassium salt. In both ligands the plane of the PO_2 atoms is perpendicular (89·6°) to the PS_2 plane. The average P–O bond length is 1·55 Å, comparable with 1·58 Å in the Ni(dtp)$_2$ compound but below the 1·64 and 1·61 Å values in K[$S_2P(OCH_3)_2$] and Ba[$O_2P(OC_2H_5)_2$] (Kyogoku, & Iitaka, 1966). The individual O–C and C–C distances in the ethoxy groups are equivalent within 3σ. The average values (1·44 and 1·49 Å) are equal to those in Ni(dtp)$_2$ and Ba[$O_2P(OC_2H_5)_2$].

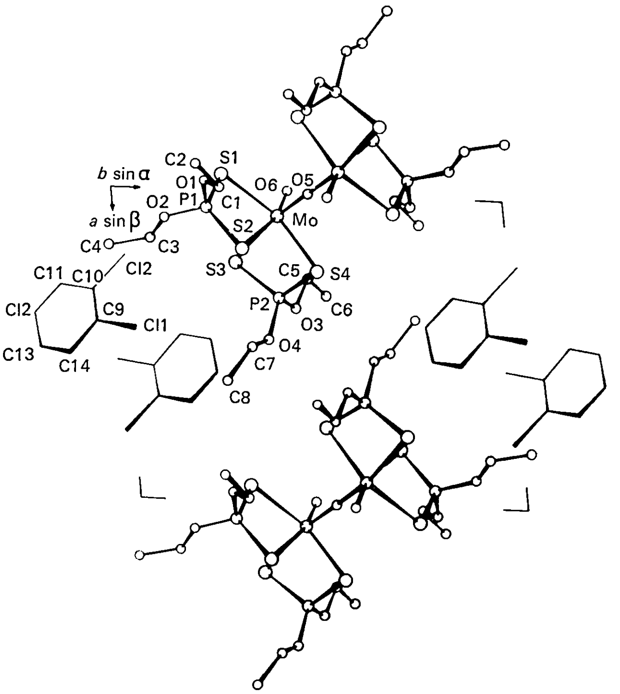

Fig. 2. The crystal structure viewed along **c**.

Because of its weak attachment to the metal, the P(1) ligand subtends a smaller S–Mo–S angle (75°) than does the P(2) ligand (79°). The S–P(2)–S angle in the more tightly-bound ligand (105°) is nearer the angle in Ni(dtp)$_2$ (103°) than is the angle in the P(1) ligand (109°), which is somewhat closer to the 118° value in the potassium salt.

The C–C distances in the $C_6H_4Cl_2$ ring range from 1·34 to 1·45 Å (see *Refinement*) and average 1·39 Å. The mean C–Cl bond length is 1·76 Å. The root mean square displacement of atoms from the least-squares plane is 0·021 Å, and the equation of the plane (referred to orthorgonal axes a, b^* and c' where c' lies on the same side of the ab^* plane as c) is:

$$-0·6291X' - 0·4882Y' + 0·6049Z' = 4·8512.$$

The closest intermolecular repulsions are between ethyl groups (Table 2) and are directed so as to tilt the P(2)S_2 plane 0·33 Å away from Mo. The $C_6H_4Cl_2$ molecules occur as pairs in the cavities formed by the ethyl groups (Fig. 2). The nearest approach of the two dichlorobenzene molecules is 3·69 Å between C(9) atoms.

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