O(3)	0.4894 (6)	0.2592 (6)	0.1629 (3)	0.040 (4)
O(4)	0.5634 (7)	0.4094 (7)	0.1025 (3)	0.051 (4)
C(3)	0.5621 (9)	0.3049 (10)	0.2321 (4)	0.042 (5)
C(4)	0.4821 (10)	0.2252 (10)	0.2052 (4)	0.046 (6)
C(5)	0.4446 (11)	0.3220 (10)	0.1563 (5)	0.050 (7)
C(6)	0.4577 (10)	0.3509 (11)	0.1121 (5)	0.052 (6)
C(7)	0.5842 (13)	0.4374 (12)	0.0603 (4)	0.058 (7)
C(8)	0.5899 (13)	0.3577 (14)	0.0345 (4)	0.061 (8)

† Atom C(1) was refined at 90% occupancy.

Table 2. *Selected geometric parameters* (Å, °)

Ta—Ta^{i}	2.978 (1)	O(1)—C(1)	1.39 (2)
Ta—Ta^{iv}	2.987 (1)	O(2)—C(2)	1.39 (2)
Ta—Cl(1)	2.447 (3)	O(1)—O(2)	2.68 (2)
Ta—Cl(1^{iv})	2.449 (3)	Na—N(1)	2.73 (2)
Ta—Cl(2)	2.449 (3)	Na—N(2)	2.76 (2)
Ta—Cl(2^{ii})	2.447 (3)	Na—O(3)	2.508 (9)
Ta—O(1)	2.049 (10)	Na—O(4)	2.557 (12)
Cl(1)—Ta—Cl(2^{ii})	165.0 (1)	O(3)—Na—O(4)	66.2 (3)
Ta—Cl(1)—Ta^{v}	75.2 (1)	O(3)—Na—O(3^{vi})	106.8 (3)
Ta—O(1)—C(1)	131.3 (9)	O(4)—Na—O(3^{vi})	170.3 (2)
N(1)—Na—O(3)	67.9 (2)	O(4)—Na—O(3^{vii})	82.1 (2)
N(1)—Na—O(4)	113.2 (3)	O(4)—Na—O(4^{vi})	105.4 (3)

Symmetry codes: (i) $1 - y, 1 - y + x, z$; (ii) $-x + y, 1 - x, z$; (iii) $\frac{2}{3} - x, \frac{4}{3} - y, \frac{1}{3} - z$; (iv) $y - \frac{1}{3}, \frac{1}{3} - x + y, \frac{1}{3} - z$; (v) $\frac{2}{3} + x - y, \frac{1}{3} + x, \frac{1}{3} - z$; (vi) $1 - y, -y + x, z$; (vii) $1 + y - x, 1 - x, z$.

The brown crystals are sensitive to moisture and air and so crystals were selected under dry petroleum, fixed with grease and sealed in glass capillaries (0.5 mm diameter) under an atmosphere of argon saturated with methanol. Despite these precautions, the crystals decomposed at room temperature within one day. Another crystal was sealed and immediately transferred to a four-circle diffractometer and cooled to 173 K at a rate of 15 K h^{-1}. Rhombohedral lattice type and Laue class $\bar{3}$ followed from a rapid data collection. The positions of the Ta and Cl atoms were determined by the Patterson method, all other non-H atoms being located in difference Fourier syntheses. The H atoms were located easily after refinement with anisotropic temperature coefficients. All methyl and methylene H atoms were refined with the C—H distance set to 0.96 Å while the isotropic displacement parameter was kept at a common value within the methyl and methylene groups. The acidic H(9) atom of the methanol molecule was refined with an isotropic displacement parameter. Final calculations were performed with the H atoms riding on their associated C atoms.

Because of the high water content of the starting material and oxidation in an oxygen atmosphere, we suspected partial substitution of OCH_3^- by OH^-. Indeed, the refined site occupancy of the C(1) atom was only 0.90 (5). To check the significance of the deviation from unity we refined the occupancy of atoms O(1) and C(3). The values of 1.00 (3) and 1.01 (6) for O(1) and C(3), respectively, support our result for C(1). Therefore, C(1) and the connected H atoms were refined at 90% occupancy in the final least-squares cycles. All calculations were performed on a VAX 3200 computer using the *SHELXTL-Plus* program package (Sheldrick, 1990).

The Ta—Ta distances and their e.s.d.'s did not change when the refinement was performed with 2174 $F_o > 1\sigma(F_o)$ data or with all 2230 $(F_o)^2$ data. Nevertheless, the significance of the slight difference needs to be checked carefully with more precise and extensive data.

We wish to thank the unknown referee for valuable suggestions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71635 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1052]

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Acta Cryst. (1994). C**50**, 697–700

Di-μ-chlorobis[hydrogen bis(diphenylphosphinito)(1 −)-*P*,*P*′]dipalladium, $[Pd_2Cl_2\{(C_6H_5)_2PO\cdots H\cdots OP(C_6H_5)_2\}_2]$

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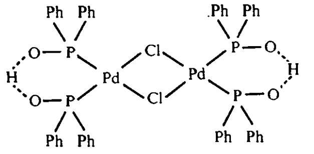
Abstract

The title compound consists of dinuclear chloro-bridged palladium units with two coordinated diphenylphosphinito groups per Pd atom, linked by a hydrogen bridge. The average O···O distance in the O···H···O bridges of the two independent molecules is 2.406 (4) Å, which is comparable to values in similar phosphinato groups.

Comment

As part of a study on the reactions of secondary phosphines with palladium complexes, we reacted an orthometallated azobenzenepalladium chloride dimer (Bruce, Liddell & Pain, 1989) with diphenylphosphine in toluene. The initial product was orange in colour; recrystallization in air using dichloromethane/methanol gave a small quantity of pale yellow material. A second recrystallization from nitromethane gave pale yellow needles, which surprisingly contained no nitrogen. In order to establish the structure, an X-ray diffraction study was performed and this revealed the complex to be the title compound, (I). The preparation of (I) from diphenylphosphine oxide and sodium chloropalladate has been reported previously (Dixon & Rattray, 1971).



(I)

The structure (Figs. 1 and 2) consists of two independent centrosymmetric molecules per unit cell. The coordination geometry is similar in both molecules, but the orientation of the phenyl rings is different. If the phenyl groups are excluded, the $P_4Pd_2Cl_2$ core of each molecule has symmetry fairly close to D_{2h}. The O···H···O distances in the two molecules are very similar: 2.404 (8) in molecule *A* and 2.408 (8) Å in molecule *B*. The structures of several (phosphinito)palladium complexes have been reported. The O···H···O distances are given in Table 3 and are slightly shorter than those observed in the enols of *β*-diketones, for which a distance greater than 2.45 Å is usually found (Emsley, 1984).

In (I) the P—O distances are all within one e.s.d. of 1.541 Å and so it can be suggested that the hydrogen bond is symmetrical, as was found for the iodide and thiocyanate (Table 3). The H atoms in the bridges were found in the difference map and their positions were refined; the experimentally determined differences in O—H bond lengths are thought to be of only marginal significance.

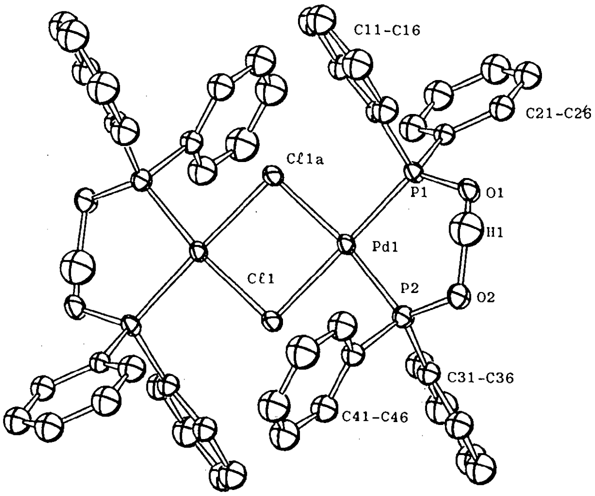


Fig. 1. *ORTEP* (Johnson, 1965) diagram of molecule *A* showing the atomic numbering scheme. The molecule is centrosymmetric and thus numbers are not shown for all atoms.

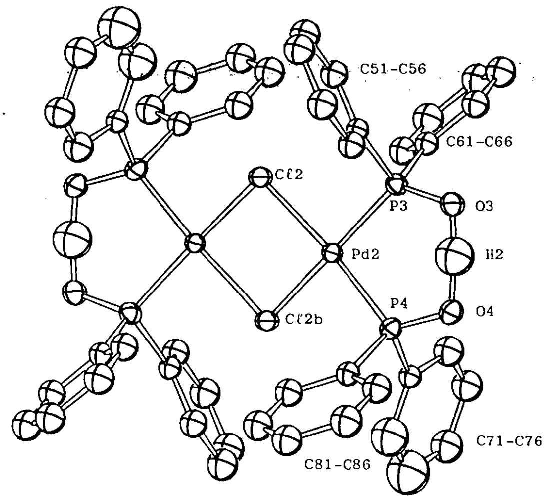


Fig. 2. *ORTEP* (Johnson, 1965) diagram of molecule *B* showing the atomic numbering scheme. The molecule is centrosymmetric and thus numbers are not shown for all atoms.

Experimental

Crystal data

$[Pd_2Cl_2(C_{12}H_{10}OP)_2$-
$(C_{12}H_{11}OP)_2]$
M_r = 1090.45
Triclinic
$P\bar{1}$
a = 11.920 (12) Å
b = 13.299 (13) Å
c = 17.407 (13) Å
α = 100.79 (8)°
β = 100.88 (8)°
γ = 116.60 (8)°
V = 2304 (3) Å^3

Z = 2
D_x = 1.572 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 16 reflections
θ = 22.5-25°
μ = 1.06 mm^{-1}
T = 298 K
Needles
0.55 × 0.35 × 0.10 mm
Pale yellow

Data collection

Picker diffractometer
$\theta/2\theta$ scans
Absorption correction: by integration from crystal shape
$T_{\min}$ = 0.703, $T_{\max}$ = 0.891
6033 measured reflections
6033 independent reflections

4849 observed reflections [$I > 2.5\sigma(I)$]
$\theta_{\max}$ = 22.5°
$h = 0 \rightarrow 12$
$k = -14 \rightarrow 12$
$l = -18 \rightarrow 18$
1 standard reflection monitored every 100 reflections
intensity variation: 0.50%

Refinement

Refinement on F
R = 0.047
wR = 0.048
S = 2.23
4849 reflections
117 parameters
H atom parameters refined for H1 and H2

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max}$ = 0.14
$\Delta\rho_{\max}$ = 1.13 e Å^{-3}
$\Delta\rho_{\min}$ = −0.77 e Å^{-3}
Atomic scattering factors from *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Molecule *A*				
Pd1	0.04153 (5)	0.14591 (5)	0.99275 (3)	2.51 (3)
Cl1	0.1544 (2)	0.0390 (2)	1.0264 (1)	3.45 (11)
P1	−0.0704 (2)	0.2400 (2)	0.9640 (1)	2.86 (11)
P2	0.2353 (2)	0.3020 (2)	1.0035 (1)	3.29 (12)
O1	0.0081 (6)	0.3633 (5)	0.9551 (4)	3.8 (3)
O2	0.2319 (5)	0.4053 (5)	0.9771 (4)	4.4 (4)
C11	−0.2018 (4)	0.1521 (4)	0.8673 (3)	3.1 (1)
C12	−0.1798 (5)	0.1859 (4)	0.7979 (3)	4.5 (2)
C13	−0.2766 (6)	0.1214 (6)	0.7214 (3)	5.9 (2)
C14	−0.3955 (5)	0.0231 (5)	0.7142 (3)	5.5 (2)
C15	−0.4176 (4)	−0.0106 (4)	0.7836 (4)	5.1 (2)
C16	−0.3207 (5)	0.0538 (5)	0.8601 (3)	3.9 (2)
C21	−0.1554 (5)	0.2572 (5)	1.0376 (3)	2.9 (1)
C22	−0.2024 (6)	0.1790 (4)	1.0824 (4)	4.8 (2)
C23	−0.2729 (6)	0.1954 (5)	1.1342 (4)	5.7 (2)
C24	−0.2964 (6)	0.2899 (6)	1.1414 (4)	5.5 (2)
C25	−0.2494 (6)	0.3681 (5)	1.0967 (4)	4.9 (2)
C26	−0.1789 (6)	0.3517 (4)	1.0448 (3)	4.0 (2)
C31	0.3499 (5)	0.3622 (5)	1.1060 (3)	3.6 (2)
C32	0.3073 (5)	0.3231 (5)	1.1696 (4)	5.2 (2)
C33	0.3944 (7)	0.3733 (6)	1.2496 (3)	6.9 (3)
C34	0.5242 (6)	0.4626 (6)	1.2659 (3)	6.5 (3)
C35	0.5667 (5)	0.5017 (5)	1.2023 (4)	6.1 (2)
C36	0.4796 (6)	0.4515 (5)	1.1223 (3)	4.9 (2)
C41	0.3066 (6)	0.2447 (5)	0.9367 (3)	3.5 (2)
C42	0.2600 (6)	0.2267 (6)	0.8527 (4)	5.6 (2)
C43	0.3081 (7)	0.1790 (7)	0.7979 (3)	7.4 (3)
C44	0.4027 (7)	0.1492 (7)	0.8271 (4)	6.6 (3)
C45	0.4493 (6)	0.1672 (6)	0.9111 (4)	5.9 (2)
C46	0.4012 (6)	0.2149 (6)	0.9659 (3)	4.7 (2)
H1	0.09 (1)	0.38 (1)	0.96 (1)	7.9 (28)
Molecule *B*				
Pd2	0.55867 (5)	0.64271 (5)	0.48389 (4)	2.82 (3)
Cl2	0.3498 (2)	0.4608 (2)	0.4495 (1)	3.60 (11)
P3	0.4551 (2)	0.7368 (2)	0.4419 (1)	3.21 (11)
P4	0.7573 (2)	0.8001 (2)	0.5076 (1)	3.43 (11)
O3	0.5374 (6)	0.8612 (5)	0.4347 (4)	4.4 (4)
O4	0.7632 (5)	0.9125 (5)	0.4924 (4)	4.7 (4)
C51	0.3362 (5)	0.6497 (5)	0.3414 (3)	3.3 (2)
C52	0.3830 (4)	0.6641 (5)	0.2742 (4)	4.7 (2)
C53	0.2967 (6)	0.6009 (6)	0.1943 (3)	5.9 (2)
C54	0.1636 (6)	0.5232 (6)	0.1815 (3)	6.3 (2)
C55	0.1168 (4)	0.5088 (5)	0.2486 (4)	6.0 (2)
C56	0.2031 (5)	0.5720 (6)	0.3286 (3)	4.8 (2)
C61	0.3655 (6)	0.7530 (5)	0.5121 (3)	3.5 (2)
C62	0.3510 (6)	0.6961 (5)	0.5723 (4)	4.9 (2)
C63	0.2832 (7)	0.7129 (6)	0.6260 (4)	6.0 (2)
C64	0.2299 (7)	0.7865 (6)	0.6196 (4)	5.9 (2)
C65	0.2444 (7)	0.8434 (6)	0.5594 (4)	5.9 (2)
C66	0.3122 (6)	0.8266 (5)	0.5057 (3)	4.8 (2)
C71	0.8475 (6)	0.8419 (6)	0.6145 (3)	3.3 (2)
C72	0.9592 (7)	0.8324 (7)	0.6416 (5)	9.3 (4)
C73	1.0247 (7)	0.8694 (9)	0.7257 (5)	11.9 (5)
C74	0.9786 (7)	0.9159 (7)	0.7826 (3)	6.6 (3)
C75	0.8669 (7)	0.9254 (6)	0.7554 (3)	6.0 (2)
C76	0.8014 (6)	0.8884 (6)	0.6714 (4)	5.9 (2)
C81	0.8463 (6)	0.7589 (5)	0.4457 (3)	3.7 (2)
C82	0.9073 (7)	0.8354 (4)	0.4028 (4)	5.5 (2)
C83	0.9734 (7)	0.8078 (6)	0.3518 (4)	6.7 (3)
C84	0.9784 (7)	0.7037 (6)	0.3435 (4)	5.9 (2)
C85	0.9174 (7)	0.6271 (5)	0.3864 (4)	5.7 (2)
C86	0.8513 (6)	0.6548 (5)	0.4374 (4)	5.0 (2)
H2	0.65 (1)	0.89 (1)	0.47 (1)	9.9 (33)

Table 2. *Selected geometric parameters* (Å, °)

Molecule *A*		Molecule *B*	
Pd1—Cl1	2.430 (3)	Pd2—Cl2	2.432 (4)
Pd1—Cl1*a*	2.423 (4)	Pd2—Cl2*b*	2.428 (3)
Pd1—P1	2.252 (3)	Pd2—P3	2.238 (3)
Pd1—P2	2.253 (4)	Pd2—P4	2.242 (4)
P1—O1	1.538 (6)	P3—O3	1.545 (6)
P2—O2	1.543 (5)	P4—O4	1.539 (6)
O1—O2	2.404 (8)	O3—O4	2.408 (8)
O1—H1	0.91 (10)	O3—H2	1.18 (13)
O2—H1	1.50 (10)	O4—H2	1.23 (13)
Cl1—Pd1—Cl1*a*	83.40 (10)	Cl2—Pd2—Cl2*b*	84.11 (11)
Cl1—Pd1—P1	177.31 (7)	Cl2—Pd2—P3	91.41 (11)
Cl1—Pd1—P2	89.96 (10)	Cl2—Pd2—P4	174.34 (8)
Cl1*a*—Pd1—P1	94.33 (10)	Cl2*b*—Pd2—P3	168.68 (7)
Cl1*a*—Pd1—P2	171.90 (7)	Cl2*b*—Pd2—P4	93.52 (11)
P1—Pd1—P2	92.43 (10)	P3—Pd2—P4	91.84 (11)
Pd1—Cl1—Pd1*a*	96.60 (10)	Pd2—Cl2—Pd2*b*	95.89 (11)
Pd1—P1—O1	117.08 (23)	Pd2—P3—O3	119.05 (24)
Pd1—P2—O2	117.84 (23)	Pd2—P4—O4	118.30 (24)
P1—O1—H1	111 (6)	P3—O3—H2	106 (5)
P2—O2—H1	107 (4)	P4—O4—H2	107 (5)
O1—H1—O2	171 (10)	O3—H2—O4	173 (11)

Table 3. $O\cdots H\cdots O$ *distances* (Å) *in* (*phosphinito*)-*palladium complexes*

	$O\cdots H\cdots O$ distance	Reference
$Pd_2Cl_2(PPh_2O)_2(PPh_2OH)_2$*	2.404 (8) 2.408 (8)	This work
$Pd_2I_2(PPh_2O)_2(PPh_2OH)_2$	2.375 (5)	Colbron, Craig & Sembiring (1990)
$Pd_2(SCN)_2(PPh_2O)_2(PPh_2OH)_2$	2.421 (7)	Naik, Palenik, Jacobson & Carty (1974)
$Pd_2Cl_2[P(OEt)_2O]_2[P(OEt)_2OH]_2$	2.386 (11)	Nifant'ev, Kukhareva, Antipin, Struchkov & Klabunovsky (1983)
	2.40	Berry, Beveridge, Bushnell & Dixon (1985)
$PdS_2PMe_2[P(OEt)_2O][P(OEt)_2OH]$	2.414 (20)	Cornick, Gould, Jones & Stephenson (1977)
$PdCl(Ph_2PH)(PPh_2O)(PPh_2OH)$	2.418 (5)	Gebauer, Frenzen & Dehnicke (1992)

* Two independent molecules.

Cell refinement and data collection were performed using local versions of a four-circle diffractometer control system (Grant & Gabe, 1974). The structure was solved by direct methods using data reduction and phase determination programs in the *NRCVAX* package (Gabe, Le Page, Charland, Lee & White, 1989). The refinement was performed with rigid phenyl groups by full-matrix least squares using the same program package. The eight phenyl rings were refined as rigid groups with C—C = 1.395 and C—H = 1.08 Å and angles at phenyl C atoms of 120°. An isotropic *U* value was refined for each phenyl C atom; a fixed isotropic temperature factor 0.8 Å² greater than the temperature factor of the bonded C atom was assigned to each phenyl H atom. The positions of H1 and H2 were obtained from a difference map and their positions refined with isotropic temperature factors. Anisotropic temperature factors were used for all Cl, O, P and Pd atoms. The drawings were obtained using the *ORTEP* (Johnson, 1965) program within the *NRCVAX* package.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71771 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1069]

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Acta Cryst. (1994). C**50**, 700–702

μ_3-(*O*,*O*′-Diethyl dithiophosphato-2κ*S*,3:4κ^2*S*′)-di-μ_3-sulfido-1:2:3κ^3*S*;1:2:4κ^3*S*-μ-sulfido-1:3κ^2*S*-sulfido-1κ*S*-tris(triphenylphosphine)-2κ*P*;3κ*P*;4κ*P*-molybdenumtrisilver

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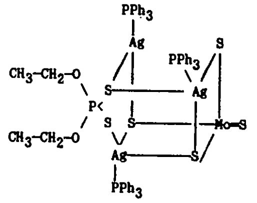
Abstract

The structure of the title compound, $[MoAg_3S_4(C_4H_{10}O_2PS_2)(C_{18}H_{15}P)_3]$, contains an incomplete cubane-like cluster core, $[MoAg_3(C_4H_{10}O_2PS_2)S_3]^{2+}$, in which a diethyl dithiophosphate group acts as a triply bridging ligand to coordinate three Ag atoms with bond lengths of 2.626 (4) [Ag(1)—S(6)], 2.690 (4) [Ag(2)—S(5)] and 2.698 (4) Å [Ag(3)—S(5)].

Comment

Several complexes of the *M*—Ag—S series (*M* = Mo, W) have been found in recent years (Gheller *et al.*, 1984; Müller & Menge, 1972; Müller, Bögge, Königer-Ahlborn & Hellman, 1979) but research into the incomplete cubane-like structures in such a series is just beginning (Nianyong, Yifan & Xintao, 1990). The title compound (I) $[MoAg_3(C_4H_{10}O_2PS_2)(C_{18}H_{15}P)_3S_4]$ is an incomplete cubane-like cluster in which one Ag—S distance is too long for effective bonding [Ag(3)···S(3) 2.971 Å]. The structure of the title compound is similar to that of other cubane-like clusters $[MoAg_3S_3Cl](X)(Ph_3P)_3$ (X = O or S) (Nianyong, Jianhui, Shaowu & Xintao, 1992; Jianhui, Nianyong, Shaowu & Xintao, 1992) except that the Cl^- anion is replaced by a bidentate diethyl dithiophosphate ligand.



(I)

The mean interatomic distances are Mo—Ag 3.046 (2), Ag—(μ_3-S) 2.577 (4), Mo—(μ-S) 2.233 (4) and Mo=S 2.131 (4) Å. The Mo atom is tetrahedrally coordinated by four S atoms and the Ph_3P ligands complete the tetrahedral geometry at each Ag atom.

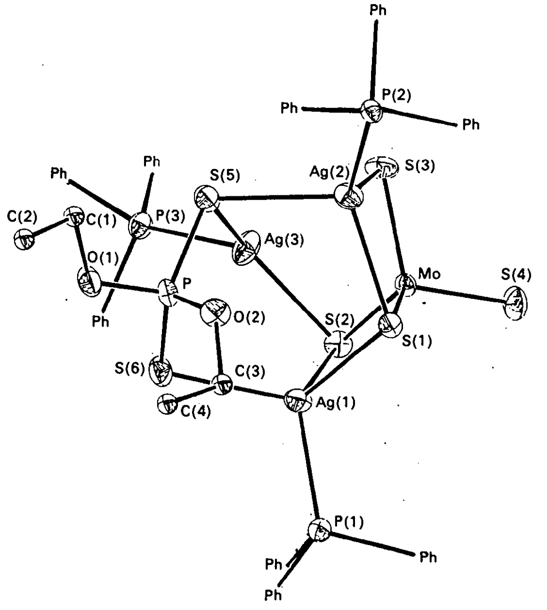


Fig. 1. View of the title compound. Displacement ellipsoids are shown at the 30% probability level.