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cis-Tetracarbonyl-1,4-h^2-1,2-bis(phenylphosphino-1,2-dimethyl)disilanemolybdenum(0)

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Abstract. $C_{20}H_{24}O_4Si_2P_2Mo$, monoclinic, $C2/c$, $a = 13{\cdot}375\,(4)$, $b = 11{\cdot}101\,(2)$, $c = 17{\cdot}250\,(9)$ Å, $\beta = 92{\cdot}94\,(2)°$, $M_r = 542{\cdot}5$, $Z = 4$, $D_x = 1{\cdot}41$ g cm^{-3}. The molecule possesses crystallographic C_2 symmetry with octahedral coordination at Mo; the phenyl substituents at the ring P atoms are therefore *trans* to one another. The five-membered chelate ring is non-planar, the Si–Si bond being twisted out of the PMoP′ plane by 28·7°. Bond lengths include Mo–P 2·550 (1), P–Si 2·266 (1) and Si–Si 2·378 (1) Å.

Introduction. Cell dimensions were determined by a least-squares fit to settings for 15 reflexions ($\pm hkl$) on a Syntex $P2_1$ four-circle diffractometer (Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å). The intensities from a prismatic crystal sealed in a Lindemann-glass capillary were collected on the diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Measurements were carried out in the θ–2θ mode ($3{\cdot}0 \le 2\theta \le 65{\cdot}0°$) at scan speeds varying linearly between 2·55° min^{-1} (150 c.p.s. and below) and 29·3° min^{-1} (3000 c.p.s. and above). Scan and background times were equal. Three standard reflexions showed no significant variations. Lorentz and polarization but no absorption [μ(Mo $K\alpha$) = 6·70 cm^{-1}] corrections were applied. 3173 reflexions were retained for use in the structure analysis, after application of the criterion $F \ge 3{\cdot}0\sigma(F)$ (based on counting statistics).

The structure was solved by Patterson and Fourier methods. Refinement with anisotropic temperature factors for all the non-hydrogen atoms and isotropic temperature factors for the H atoms was by full-matrix least squares, $\Sigma w\Delta^2$ being minimized. The terminal value of $R_G = [\Sigma w\Delta^2/\Sigma wF_o^2]^{1/2}$ was 0·057, with $R_w = \Sigma w^{1/2}\Delta/\Sigma w^{1/2}F_o = 0{\cdot}052$ and $R = 0{\cdot}059$.† The weights were given by $w = k/[\sigma^2(F_o) + gF_o^2]$, where k and g refined to 1·8757 and 0·000524 respectively. In the final cycle the largest observed shift/e.s.d. was 0·002. Complex neutral-atom scattering factors (Cromer & Waber, 1965; Cromer & Liberman, 1970) were employed. Positional parameters and, for H, isotropic thermal parameters are given in Tables 1 and 2. Bond lengths and angles are in Tables 3 and 4.

Table 1. *Positional parameters* (×10⁴) *for the nonhydrogen atoms*

	x	y	z
Mo	5000	6393 (1)	2500
P	3760 (1)	4761 (1)	2088 (1)
Si	4442 (1)	2905 (1)	1956 (1)
C(1)	5647 (3)	6369 (3)	1458 (2)
O(1)	6014 (2)	6360 (3)	880 (1)
C(2)	5932 (3)	7649 (3)	2913 (2)
O(2)	6445 (3)	8387 (3)	3167 (2)
C(3)	2884 (2)	4944 (3)	1244 (2)
C(4)	3122 (3)	5670 (4)	632 (2)
C(5)	2470 (4)	5778 (5)	9985 (2)
C(6)	1574 (4)	5194 (5)	9954 (3)
C(7)	1322 (4)	4489 (5)	555 (3)
C(8)	1975 (3)	4372 (4)	1210 (3)
C(9)	3483 (5)	1689 (4)	1974 (5)
C(10)	5116 (6)	2916 (6)	1037 (3)

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32683 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Positional parameters* ($\times 10^3$) *and isotropic temperature factors* ($\times 10^3$) *for the* H *atoms*

	x	y	z	U(Å^2)
H(21)	318 (3)	455 (3)	259 (2)	38 (10)
H(41)	367 (3)	610 (4)	66 (2)	47 (12)
H(51)	258 (5)	620 (6)	954 (4)	108 (22)
H(61)	118 (4)	527 (4)	947 (3)	74 (15)
H(71)	73 (4)	403 (5)	55 (3)	89 (18)
H(81)	179 (4)	396 (5)	163 (3)	76 (17)
H(91)	312 (5)	168 (5)	156 (4)	99 (23)
H(92)	314 (5)	171 (6)	242 (4)	112 (27)
H(93)	380 (4)	95 (5)	205 (3)	67 (14)
H(101)	470 (5)	296 (6)	64 (4)	97 (23)
H(102)	552 (5)	226 (6)	101 (4)	107 (24)
H(103)	557 (5)	361 (6)	100 (4)	114 (25)

Table 3. *Bond lengths*(Å)

P–Mo	2·550 (1)	C(1)–Mo	2·008 (4)
C(2)–Mo	1·994 (4)	Si–P	2·266 (1)
C(3)–P	1·864 (4)	C(9)–Si	1·863 (6)
C(10)–Si	1·836 (6)	Si–Si	2·378 (2)
O(1)–C(1)	1·119 (5)	O(2)–C(2)	1·152 (5)
C(4)–C(3)	1·369 (6)	C(8)–C(3)	1·373 (6)
C(5)–C(4)	1·411 (6)	C(6)–C(5)	1·363 (7)
C(7)–C(6)	1·346 (8)	C(8)–C(7)	1·423 (7)
H(21)–P	1·20 (4)	H(41)–C(4)	0·88 (4)
H(51)–C(5)	0·92 (7)	H(61)–C(6)	0·98 (5)
H(71)–C(7)	0·94 (6)	H(81)–C(8)	0·89 (5)
H(91)–C(9)	0·85 (7)	H(92)–C(9)	0·91 (7)
H(93)–C(9)	0·94 (5)	H(101)–C(10)	0·88 (6)
H(102)–C(10)	0·91 (7)	H(103)–C(10)	0·98 (7)

Table 4. *Bond angles* (°)

C(1)–Mo–P	91·7 (1)	C(2)–Mo–P	175·2 (1)
C(2)–Mo–C(1)	93·0 (2)	Si–P–Mo	114·4 (0)
C(3)–P–Mo	122·5 (1)	C(3)–P–Si	105·7 (1)
P–Mo–P	89·4 (0)	C(9)–Si–P	112·2 (2)
C(10)–Si–P	106·5 (2)	C(10)–Si–C(9)	111·5 (4)
O(1)–C(1)–Mo	179·5 (4)	O(2)–C(2)–Mo	177·8 (4)
C(2)–Mo–C(2)	91·3 (3)	C(4)–C(3)–P	121·0 (3)
C(8)–C(3)–P	121·9 (3)	C(8)–C(3)–C(4)	117·1 (4)
C(5)–C(4)–C(3)	120·8 (5)	C(6)–C(5)–C(4)	121·4 (5)
C(7)–C(6)–C(5)	118·6 (4)	C(8)–C(7)–C(6)	120·4 (5)
C(7)–C(8)–C(3)	121·6 (5)		

Discussion. This structural study of the title compound (I) (Johannsen & Stelzer, 1977) is part of a series on chelate complexes of Mo with bidentate phosphine ligands. The structures of the analogous *cis*-Mo(CO)$_4$-(PMe$_2$–XMe–XMe–Me$_2$P) [with X = P (II), or X = As (III)] compounds have been reported (Sheldrick, 1975*a*,*b*), as has that of *cis*-Mo(CO)$_4$(PMe$_2$–PBu^t–Me$_2$P) (Sheldrick, 1976).

The molecule of (I) (Fig. 1) possesses crystallographic C_2 symmetry with octahedral coordination at Mo. The five-membered chelate ring is non-planar, the Si–Si bond being twisted out of the PMoP′ plane by 28·7°, which is considerably greater than the values of 16·5° for the P–P bond in (II) and 16·0° for the As–As bond in (III). The Si atom is 0·427 Å below the weighted least-squares plane, P is 0·242 Å above. As in (II) and (III), no significant difference is observed between the Mo–C distances for the two carbonyl groups mutually *trans* to one another and the two *trans* to P. The Mo–P distance of 2·550 (1) Å is, however, significantly longer than those of 2·489 (6) and 2·453 (7) Å in (II) and (III). This suggests that the

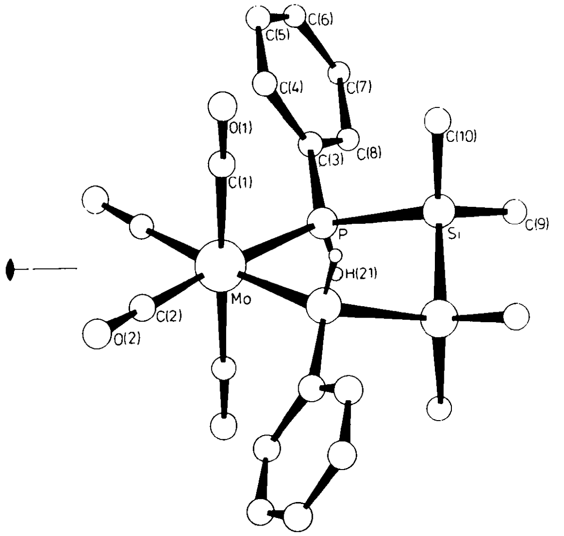

Fig. 1. A perspective view of molecule I with the atom numbering.

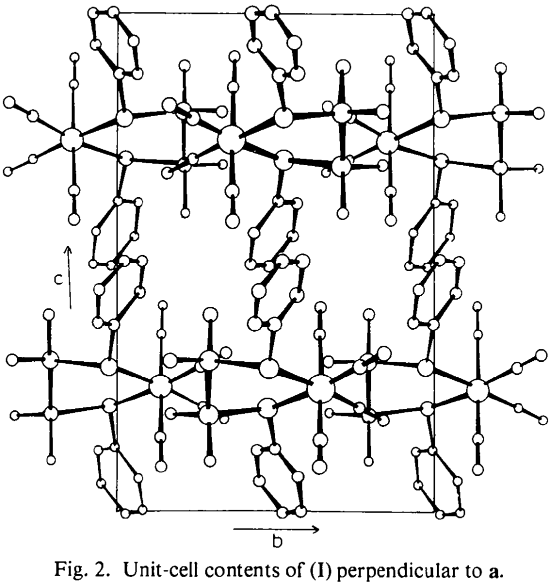

Fig. 2. Unit-cell contents of (I) perpendicular to a.

($d \rightarrow d$) π-bonding component in the Mo—P bond in (I) is less important than in these other derivatives. The P—Si length of 2·266 (1) Å is similar to that of 2·248 (3) Å in $P(SiH_3)_3$ (Beagley, Robiette & Sheldrick, 1968). The Si—Si distance of 2·378 (2) Å is normal.

Fig. 2 shows the unit-cell contents viewed perpendicular to **a**.

Calculations were carried out with *SHELX* 76 (G. M. Sheldrick, Cambridge) and local ancillary programs (WSS). We are indebted to Dr O. Stelzer for suggesting the problem and for the provision of crystals.

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Potassium Perchlorate

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Abstract. $KClO_4$, orthorhombic, *Pnma*, $a = 8{\cdot}866$ (2), $b = 5{\cdot}666$ (1), $c = 7{\cdot}254$ (1) Å, $Z = 4$, $V = 364{\cdot}4$ (1) Å^3, $M_r = 138{\cdot}56$. The final R is 0·031 for 533 independent reflexions. The K atom is twelvefold coordinated with K—O distances of 2·862 (3)—3·453 (3) Å. The Cl—O distances are 1·427 (4)—1·441 (3) Å.

Introduction. Crystals of $KClO_4$ were obtained from water. The structure has been solved from powder data by Gottfried & Schusterius (1933). Mani (1957) refined the structure with data obtained from two zero-layer Weissenberg films. In the present investigation intensities were collected on a four-circle Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The crystal was 0·012 × 0·025 × 0·018 mm. An $\omega/2\theta$ scan was used with scan intervals of 2·4–3·2° ($\Delta 2\theta$) and a scanning speed of 2·0–12·0° min^{-1}. The intensities were measured out to $2\theta = 70°$ and those 533 independent reflexions with $I/\sigma(I) > 3$ were used in the structure refinement. The background and integrated intensities were calculated with the Lehmann–Larsen profile-analysis method (local program; Lindqvist & Ljungström, 1977). The intensities were corrected for Lorentz, polarization and absorption effects (program *DATAPH*; Coppens, Leiserowitz & Rabinovich, 1965); μ(Mo $K\alpha$) = 10·03 cm^{-1} (*International Tables for X-ray Crystallography*, 1962). The final refinement included anisotropic temperature factors and an isotropic extinction parameter (*LINUS*; Coppens & Hamilton, 1970) and yielded an R of 0·031. The resulting positional parameters are given in Table 1.[*]

Table 1. *Final positional parameters*

Standard deviations are given in parentheses.

	x	*y*	*z*
K	0·1809 (1)	0·25	0·3374 (1)
Cl	0·0679 (1)	0·25	0·8105 (1)
O(1)	0·1885 (4)	0·25	0·9419 (4)
O(2)	0·4197 (2)	0·5438 (4)	0·1952 (3)
O(3)	0·4253 (4)	0·25	0·5981 (4)

Table 2. *Bond distances* (Å) *and angles* (°) *in* $KClO_4$

Cl—O(3)	1·427 (4)	2× O(1)—O(2)	2·343 (4)
Cl—O(1)	1·433 (4)	O(1)—O(2)	2·351 (6)
2× Cl—O(2)	1·441 (3)	2× O(2)—O(3)	2·346 (4)
2× K—O(2)	2·862 (3)	O(2)—O(2)	2·337 (5)
K—O(1)	2·870 (4)		
K—O(3)	2·877 (4)	O(1)—Cl—O(2)	109·2 (1)
2× K—O(2)	2·884 (3)	O(1)—Cl—O(3)	110·6 (2)
2× K—O(2)	2·983 (3)	O(2)—Cl—O(2)	108·3 (2)
2× K—O(1)	3·153 (2)	O(2)—Cl—O(3)	108·7 (2)
2× K—O(2)	3·453 (3)		

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[*] Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32684 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.