(Cotton & LaPrade, 1968; Behrens & Weiss, 1975; Becker & Stille, 1978; Sonada, Bailey & Maitlis, 1979). The C(2)—C(3) and C(4)—C(5) bond lengths are significantly shorter than the remainder of the C—C distances in the ligand, suggesting the double-bond localization of a diene. The difference between the averages of longer and shorter bond lengths is 0·058 Å for $[(\eta^3\text{-C}_6\text{H}_5\text{CH}_2)\text{Rh}\{(\{(\text{CH}_3)_3\text{C}\}_2\text{P})_2\text{C}_2\text{H}_4\}]$; on this basis, the magnitude of the 'allyl-diene' distortion is comparable to that observed in $[\{\eta^3\text{-(CH}_3)_5\text{C}_6\text{CH}_2\}\text{-Rh(P}\{\text{OCH(CH}_3)_2\}_3)_2]$ (0·055 Å), $[(\eta^3\text{-C}_6\text{H}_5\text{CH}_2)\text{Co-}\{\text{P(OCH}_3)_3\}_3]$ (0·057 Å), and $[\{\eta^3\text{-C}_6\text{H}_5\text{CH(C}_2\text{H}_5)\}\text{-Rh(1,5-cyclooctadiene)}]$ (0·065 Å) (Stuhler & Pickardt, 1981). The η^3 coordination of the benzyl ligand in solution and the fluxionality of the complex have been established by $^{31}\text{P}\{^1\text{H}\}$, $^{13}\text{C}\{^1\text{H}\}$, and ^{1}H NMR spectroscopy (Del Paggio, 1986; Del Paggio, Andersen & Muetterties, 1987).

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Monoclinic and Triclinic Forms of [1,2-Bis(diphenylphosphino)propane]-(η^6-methyldiphenylphosphine)(methyldiphenylphosphine)molybdenum(0) Benzene Solvate

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Abstract. $[\text{Mo(C}_{13}\text{H}_{13}\text{P)}_2(\text{C}_{27}\text{H}_{26}\text{P}_2)]_2.\text{C}_6\text{H}_6$, M_r = 1895·8, triclinic, $P\bar{1}$, a = 13·808 (2), b = 15·860 (3), c = 23·315 (5) Å, α = 103·18 (2), β = 102·45 (1), γ = 96·79 (1)°, U = 4779 (4) Å^3, Z = 2, D_x = 1·317 Mg m^{-3}, μ(Mo $K\alpha$) = 0·43 mm^{-1}, F(000) = 1972, T = 298 K, R = 0·081 for 6515 observed $[I > 3\sigma(I)]$ reflections. $[\text{Mo(C}_{13}\text{H}_{13}\text{P)}_2(\text{C}_{27}\text{H}_{26}\text{P}_2)].\text{C}_6\text{H}_6$, M_r = 987·0, monoclinic, $P2_1/a$, a = 19·165 (9), b = 13·154 (10), c = 20·195 (9) Å, β = 105·09 (4)°, U = 4916 (9) Å^3, Z = 4, D_x = 1·333 Mg m^{-3}, μ(Mo $K\alpha$) = 0·43 mm^{-1}, F(000) = 2056, T = 298 K, R = 0·093 for 2268 observed $[I > 3\sigma(I)]$ reflections. The triclinic form contains two independent molecules in the asymmetric unit. One molecule has the configurations S(*exo*-P), S(prophos) and the other S(*exo*-P), R(prophos) for the phosphine ligands. Thus all possible combinations of the possible configurations of the phosphine ligands (*SS*, *RR*, *RS* and *SR*) are present in the centrosymmetric unit cell. There is one molecule of benzene solvent in the asymmetric unit. In the asymmetric unit of the monoclinic form the molecule has the configurations S(*exo*-P), S(prophos) and the unit cell contains only *SS* and *RR* molecules. There is one molecule of benzene solvent per Mo in this form.

Introduction. Formation of η^6-arylphosphine molybdenum complexes is thought to occur *via* an intramolecular σ to π rearrangement of an octahedral disolvated tetraphosphine molybdenum intermediate (Frizzell, Luck, Morris & Peng, 1985). In the series of compounds $[\text{Mo}(\eta^6\text{-PhPMePh)(P—P)(PMePh}_2)]$ (P—P = chelating diphosphine) the dangling P atom of the

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Table 1. *Experimental details*

System	Triclinic	Monoclinic
Chemical formula	$(C_{53}H_{52}MoP_4)_2.C_6H_6$	$C_{53}H_{52}MoP_4.C_6H_6$
Formula weight	1895·8	987·0
Crystal color	Dark red	Dark red
Crystal size (mm)	0·088 × 0·100 × 0·150	0·094 × 0·131 × 0·138
No. of reflections in cell determination/θ range (°)	25/($8·4 < \theta < 15·3$)	22/($7·7 < \theta < 14·6$)
ω-scan range (°)	$0·50 + 0·35\tan\theta$	$0·60 + 0·35\tan\theta$
Max. scan time (s)[*]	45	40
Max. 2θ (°)/quadrants	45/h, $\pm k$, $\pm l$[†]	40/h, k, $\pm l$
No. of standard reflections/ interval (s)	3/9500	3/9500
No. of data collected	12667	5079
No. of unique data	10814[‡]	3970[§]
No. of data with $I > 3\sigma(I)$	6515	2268
Final R (wR)	0·081 (0·085)[¶]	0·093 (0·120)[**]
Max. Δ/σ in final cycle	0·12	0·08
Weights	$[\sigma^2(F) + 0·00187F^2]^{-1}$	$4F^2[\sigma^2(I) + (0·13F^2)^2]^{-1}$
Max. peak in final difference Fourier map (e Å^{-3})	1·0	1·4

[*] Prescan at 10° min^{-1}. Prescan intensity accepted if $I/\sigma(I) \geq 25$, otherwise scan speeds chosen to give this ratio within max. scan time specified. Backgrounds by extending scan by 25% on either side of peak were measured for half the time taken to collect peak.

[†] h, k, $\pm l$ for $h = 0$.

[‡] Data with $F_{obs} = 0·0$ rejected.

[§] 887 systematically absent or zero F_{obs} data rejected and 140 symmetry equivalent data merged [$R_{merge}(I) = 0·078$].

[¶] Blocked least squares (three blocks), Mo + P anisotropic.

[**] Full-matrix least squares, Mo + P anisotropic, $S = 1·50$ e.

η^6-bonded ligand is a centre of chirality which is known to be stable to inversion at 298 K. Chirality is induced at this centre when a chiral diphosphine forms part of the tetraphosphine intermediate. Thus ^{31}P NMR studies indicate that one of the possible diastereoisomers of Mo(η^6-PhPMePh)(P—P)(PMePh$_2$) with either *R* or *S* configuration at the dangling P atom forms in 18% enantiomeric excess (e.e.) over the alternative diastereomer when P—P is (*R*)-prophos and 33% e.e. when P—P is (*S*,*S*)-chiraphos as a result of such a rearrangement (Sella, 1986). The chiral diphosphines induce chirality by presenting a chiral array of phenyl rings held rigid by the 'locked' conformation of the chelate ring (Auburn, Mackenzie & Bosnich, 1985). In synthesizing some of these compounds using racemic prophos two types of dark-red crystalline material were obtained and the stereochemistries of the phosphorus ligands in both crystalline forms have been characterized by X-ray crystallography.

Experimental. Crystals of [Mo(η^6-PMePh$_2$)(propos)-(PMePh$_2$)] synthesized using racemic prophos (Sella, 1986) were grown by slow diffusion of methanol into a benzene solution of the compound. The crystals (sealed in 0·2–0·3 mm Lindemann capillaries under N_2) were found to be of two types, monoclinic and triclinic. Structures of both types were determined, although the data collected for the monoclinic form was not as extensive (max. $2\theta = 40°$) as that for the triclinic form. Enraf–Nonius CAD-4 diffractometer; Mo $K\alpha$ radiation ($\lambda = 0·71069$ Å); graphite monochromator; ω–2θ scans; Lorentz and polarization corrections applied to all data collected; absorption corrections not necessary. Experimental details are summarized in Table 1. Both structures were solved by the use of the Patterson function to locate the Mo atoms and standard least-squares and Fourier methods to locate the remaining non-H atoms. H-atom positions (except those in the solvent) were located in ΔF maps before their positions were optimized for the monoclinic structure. H atoms (representing 11% of the total electron density in the unit cell) were not included in the refinements for the triclinic form. Similarly refinement of the C atoms in both forms with anisotropic thermal parameters would more than double the numbers of variables in the least-squares refinement and was not attempted. Least-squares refinements minimizing $\sum w\Delta F^2$ converged to the indicated agreement indices which are somewhat higher than normal for the above reasons.

Calculations were on PDP 11/23 and Gould 9705 computers using programs in the *SHELX* (Sheldrick, 1976) and Enraf–Nonius *SDP* (Frenz, 1981) packages. Scattering factors stored in the programs were taken from *International Tables for X-ray Crystallography* (1974).

The final atomic positional parameters and selected bond lengths and bond angles in these and related structures are given in Tables 2 and 3 respectively.[*]

Discussion. The asymmetric unit of the triclinic form contains two independent molecules. The tabulated coordinates show molecule (1) to have the configurations *S*(*exo*-P), *S*(prophos) while molecule (2) has the configurations *S*(*exo*-P), *R*(prophos) (Fig. 1). Thus all possible combinations of possible configurations of the phosphine ligands (*SS*, *RR*, *RS* and *SR*) are present in the centrosymmetric unit cell. However, in the asymmetric unit of the monoclinic form the molecule (Fig. 2) has the configurations *S*(*exo*-P), *S*(prophos) and the unit cell contains only *SS* and *RR* molecules.

The geometry of the Mo atoms in both forms is best described as a distorted three-legged piano stool. In all three independent molecules the chelating prophos ligand considerably reduces the P(1)—Mo—P(2) angle to 79·1 (1)° and little variation is seen between the three structures. On the other hand the angles made by P(1) and P(2) with P(3) vary between 90·5 and 97·4°. These distortions are the result of steric interactions of the σ-bonded phosphines with P(4), the P—Mo—P bond angles between the prophos and PPh$_2$Me ligand in the *meso* isomers (*SR*, *RS*) being larger than those in the *SS* or *RR* isomers.

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[*] Lists of structure-factor amplitudes, H-atom parameters, complete bond lengths/bond angles in both structures, least-squares mean planes and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44293 (73 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Final atomic positional* ($\times 10^4$) *and thermal* ($\times 10^3$) *parameters*

Asterisks indicate atoms refined anisotropically and given in terms of their equivalent isotropic temperature factor, $U_{eq} = \frac{1}{3}$ trace $\mathbf{U}$.

	x	y	z	U or U_{eq}(Å^2)
(*a*) Triclinic				
Mo(1)	2244 (1)	2286 (1)	601 (1)	32·0 (4)*
P(1)	3376 (3)	3461 (2)	1408 (2)	37·6 (13)*
P(2)	1409 (3)	2297 (2)	1415 (2)	38·8 (14)*
C(1)	3005 (11)	3555 (9)	2143 (7)	51 (4)
C(2)	1858 (10)	3313 (9)	2052 (6)	45 (4)
C(3)	1606 (13)	3252 (11)	2672 (8)	75 (5)
C(111)	3504 (11)	4594 (9)	1325 (6)	49 (4)
C(112)	3078 (13)	5252 (12)	1693 (8)	77 (5)
C(113)	3179 (16)	6119 (14)	1579 (10)	102 (7)
C(114)	3669 (17)	6292 (14)	1201 (10)	107 (7)
C(115)	4125 (15)	5685 (14)	838 (9)	100 (7)
C(116)	4018 (12)	4789 (11)	889 (7)	65 (5)
C(121)	4720 (9)	3433 (8)	1719 (6)	37 (3)
C(122)	5481 (11)	4192 (9)	1920 (7)	51 (4)
C(123)	6478 (12)	4128 (10)	2197 (7)	62 (4)
C(124)	6720 (11)	3326 (10)	2278 (7)	57 (4)
C(125)	5980 (12)	2594 (10)	2092 (7)	60 (4)
C(126)	4944 (11)	2632 (10)	1823 (7)	55 (4)
C(211)	1576 (11)	1429 (9)	1836 (7)	52 (4)
C(212)	2589 (13)	1357 (11)	2073 (8)	77 (5)
C(213)	2776 (14)	719 (12)	2438 (8)	77 (5)
C(214)	1970 (17)	215 (14)	2501 (10)	104 (7)
C(215)	977 (19)	254 (15)	2264 (11)	121 (8)
C(216)	794 (14)	907 (12)	1937 (8)	85 (6)
C(221)	28 (10)	2193 (9)	1257 (6)	44 (4)
C(222)	−454 (11)	2842 (9)	1534 (6)	51 (4)
C(223)	−1530 (12)	2720 (10)	1364 (7)	61 (4)
C(224)	−2119 (13)	1974 (12)	964 (8)	77 (5)
C(225)	−1658 (14)	1297 (12)	710 (8)	86 (6)
C(226)	−562 (12)	1422 (11)	852 (7)	65 (5)
P(3)	1297 (3)	3372 (3)	224 (2)	39·5 (14)*
P(4)	4132 (3)	2236 (3)	−399 (2)	60 (2)*
C(31)	731 (11)	4132 (9)	730 (7)	51 (4)
C(41)	5246 (13)	2600 (11)	254 (8)	77 (5)
C(311)	1865 (10)	4137 (9)	−152 (6)	48 (4)
C(312)	1644 (12)	4980 (11)	−108 (7)	70 (5)
C(313)	2124 (12)	5546 (11)	−421 (8)	68 (5)
C(314)	2725 (12)	5248 (11)	−779 (8)	69 (5)
C(315)	2953 (13)	4417 (11)	−803 (8)	74 (5)
C(316)	2553 (11)	3866 (10)	−475 (7)	60 (4)
C(321)	121 (11)	2855 (9)	−382 (7)	52 (4)
C(322)	−709 (13)	2537 (11)	−223 (7)	67 (5)
C(323)	−1600 (15)	2041 (13)	−662 (10)	94 (6)
C(324)	−1558 (16)	1931 (14)	−1267 (10)	103 (7)
C(325)	−698 (17)	2176 (14)	−1460 (10)	108 (7)
C(326)	143 (14)	2700 (12)	−999 (9)	85 (6)
C(411)	3178 (12)	1749 (10)	−86 (7)	63 (4)
C(412)	2118 (12)	1634 (10)	−392 (7)	59 (4)
C(413)	1361 (12)	1140 (10)	−225 (7)	60 (4)
C(414)	1646 (11)	828 (10)	305 (7)	55 (4)
C(415)	2671 (11)	925 (10)	630 (7)	56 (4)
C(416)	3435 (11)	1421 (10)	446 (7)	56 (4)
C(421)	4500 (12)	1247 (10)	−823 (7)	57 (4)
C(422)	5369 (17)	1346 (15)	−1023 (10)	113 (7)
C(423)	5559 (15)	588 (14)	−1421 (9)	91 (6)
C(424)	4969 (17)	−182 (15)	−1617 (10)	105 (7)
C(425)	4078 (21)	−271 (18)	−1430 (12)	141 (9)
C(426)	3896 (18)	431 (17)	−975 (11)	125 (8)
Mo(2)	8470 (1)	2148 (1)	5421 (1)	30·0 (4)*
P(5)	6999 (3)	1119 (2)	5390 (2)	31·4 (12)*
P(6)	7372 (3)	2137 (2)	4446 (2)	33·6 (13)*
C(4)	5959 (10)	991 (8)	4697 (6)	40 (3)
C(5)	6433 (11)	1077 (9)	4158 (7)	53 (4)
C(6)	5581 (11)	1008 (9)	3586 (7)	55 (4)
C(511)	7065 (10)	−67 (9)	5307 (6)	45 (4)
C(512)	6722 (11)	−534 (10)	5683 (7)	55 (4)
C(513)	6752 (11)	−1466 (10)	5587 (7)	57 (4)
C(514)	7114 (12)	−1865 (11)	5111 (7)	66 (5)
C(515)	7456 (11)	−1420 (10)	4741 (7)	58 (4)
C(516)	7449 (10)	−503 (9)	4848 (6)	47 (4)
C(521)	6358 (9)	1325 (8)	6010 (6)	36 (3)
C(522)	5344 (10)	1415 (9)	5917 (6)	43 (4)
C(523)	4954 (11)	1621 (10)	6425 (7)	56 (4)
C(524)	5513 (12)	1732 (10)	7014 (7)	63 (4)
C(525)	6535 (12)	1614 (10)	7096 (7)	60 (4)
C(526)	6969 (10)	1433 (8)	6597 (6)	41 (3)
C(611)	6490 (10)	2916 (8)	4288 (6)	42 (4)
C(612)	6311 (12)	3160 (11)	3751 (7)	66 (5)
C(613)	5538 (14)	3677 (12)	3625 (8)	83 (6)
C(614)	5038 (14)	3943 (12)	4059 (9)	88 (6)
C(615)	5203 (12)	3655 (11)	4595 (8)	68 (5)
C(616)	5948 (10)	3153 (9)	4724 (6)	49 (4)
C(621)	7987 (10)	2103 (9)	3818 (6)	46 (4)
C(622)	8573 (11)	2872 (10)	3805 (7)	59 (4)
C(623)	9144 (13)	2847 (11)	3345 (8)	74 (5)
C(624)	9089 (15)	2059 (13)	2905 (9)	91 (6)
C(625)	8473 (14)	1288 (12)	2932 (8)	86 (6)
C(626)	7938 (11)	1299 (9)	3381 (7)	52 (4)
P(7)	8023 (3)	3490 (2)	6015 (2)	34·1 (13)*
P(8)	9655 (3)	1118 (3)	6661 (2)	53 (2)*
C(71)	6909 (10)	3543 (9)	6329 (6)	47 (4)
C(81)	9438 (15)	−97 (13)	6298 (9)	101 (7)
C(711)	8970 (11)	4037 (9)	6752 (7)	50 (4)
C(712)	9761 (12)	4699 (10)	6774 (7)	59 (4)
C(713)	10532 (13)	5049 (11)	7333 (8)	76 (5)
C(714)	10427 (14)	4756 (12)	7837 (9)	85 (6)
C(715)	9664 (16)	4100 (14)	7804 (9)	97 (6)
C(716)	8889 (14)	3760 (12)	7236 (9)	80 (5)
C(721)	8038 (10)	4454 (8)	5673 (6)	41 (3)
C(722)	8594 (11)	4495 (10)	5262 (7)	56 (4)
C(723)	8688 (12)	5205 (11)	5004 (7)	67 (5)
C(724)	8199 (12)	5899 (10)	5203 (7)	63 (4)
C(725)	7609 (12)	5863 (11)	5606 (7)	68 (5)
C(726)	7520 (11)	5136 (10)	5848 (7)	54 (4)
C(811)	9670 (10)	1588 (9)	6018 (6)	43 (4)
C(812)	9433 (10)	1055 (9)	5390 (6)	46 (4)
C(813)	9552 (11)	1488 (10)	4928 (7)	54 (4)
C(814)	9839 (12)	2421 (11)	5061 (8)	68 (5)
C(815)	10077 (11)	2922 (10)	5670 (7)	55 (4)
C(816)	9958 (11)	2509 (9)	6132 (7)	51 (4)
C(821)	11029 (11)	1226 (9)	7008 (7)	52 (4)
C(822)	11687 (12)	1007 (11)	6663 (7)	67 (5)
C(823)	12688 (14)	1021 (12)	6950 (9)	79 (5)
C(824)	13040 (19)	1231 (16)	7531 (12)	125 (8)
C(825)	12438 (25)	1601 (21)	7894 (14)	175 (12)
C(826)	11325 (18)	1547 (15)	7635 (11)	116 (8)
C(1S)	3907 (22)	3461 (17)	7370 (12)	139 (9)
C(2S)	4558 (18)	3761 (15)	7058 (12)	115 (8)
C(3S)	4412 (26)	4051 (21)	6581 (16)	176 (12)
C(4S)	3367 (30)	4064 (23)	6242 (16)	197 (14)
C(5S)	2720 (20)	3725 (17)	6502 (13)	134 (9)
C(6S)	2818 (24)	3452 (20)	7037 (15)	172 (12)
(*b*) Monoclinic				
Mo	1340·5 (8)	2390 (1)	2486·1 (8)	28·6 (4)*
P(1)	110 (3)	2982 (4)	2351 (2)	33 (1)*
P(2)	908 (3)	1159 (4)	3177 (2)	38 (1)*
P(3)	929 (3)	1313 (4)	1442 (3)	38 (1)*
P(4)	1836 (4)	4674 (5)	1516 (3)	63 (2)*
C(1)	−346 (10)	2237 (15)	2895 (9)	42 (5)
C(2)	−126 (10)	1142 (15)	2991 (9)	38 (5)
C(3)	−469 (11)	643 (17)	3517 (11)	58 (6)
C(31)	175 (10)	404 (16)	1357 (10)	48 (6)
C(41)	1223 (14)	5564 (22)	1779 (13)	92 (9)
C(111)	−525 (10)	2973 (16)	1494 (9)	39 (5)
C(112)	1133 (10)	2337 (16)	1305 (10)	49 (6)
C(113)	−1583 (11)	2316 (18)	653 (10)	58 (6)
C(114)	1452 (11)	3053 (18)	200 (10)	56 (6)
C(115)	−885 (11)	3675 (17)	354 (10)	57 (6)
C(116)	−437 (9)	3665 (15)	1001 (9)	35 (5)
C(121)	−95 (10)	4270 (15)	2665 (9)	39 (5)
C(122)	759 (10)	4760 (19)	2370 (10)	48 (6)
C(123)	932 (12)	5654 (19)	2663 (11)	65 (8)
C(124)	−443 (11)	6015 (18)	3244 (10)	58 (6)
C(125)	200 (11)	5584 (17)	3525 (10)	52 (6)
C(126)	332 (10)	4647 (16)	3232 (9)	43 (5)
C(211)	1144 (10)	1322 (16)	4127 (9)	44 (5)
C(212)	1100 (10)	2265 (16)	4393 (10)	48 (6)
C(213)	1202 (12)	2381 (19)	5110 (12)	75 (8)
C(214)	1311 (12)	1608 (19)	5525 (12)	71 (8)
C(215)	1322 (13)	609 (20)	5277 (12)	79 (8)
C(216)	1233 (11)	490 (18)	4572 (10)	57 (6)
C(221)	1148 (10)	−182 (16)	3138 (9)	43 (5)
C(222)	714 (11)	−1003 (18)	2962 (10)	60 (6)
C(223)	922 (11)	−1963 (18)	2932 (10)	58 (6)
C(224)	1655 (12)	−2140 (19)	3136 (12)	71 (8)
C(225)	2153 (11)	−1381 (18)	3314 (11)	61 (6)
C(226)	1935 (11)	365 (18)	3340 (11)	60 (6)
C(311)	1612 (10)	416 (16)	1277 (9)	39 (5)
C(312)	1683 (10)	−549 (16)	1583 (10)	47 (6)
C(313)	2182 (11)	−1255 (18)	1471 (11)	60 (6)
C(314)	2636 (10)	1017 (17)	1060 (10)	51 (6)
C(315)	2573 (10)	45 (16)	777 (10)	49 (6)
C(316)	2086 (11)	659 (18)	908 (10)	60 (6)

Table 2 (*cont.*)

	x	*y*	*z*	*U* or U_{eq}(Å²)		*x*	*y*	*z*	*U* or U_{eq}(Å²)
C(321)	655 (9)	1926 (15)	587 (9)	34 (5)	C(421)	2664 (11)	5396 (17)	1720 (10)	52 (6)
C(322)	1037 (10)	2759 (17)	463 (10)	54 (6)	C(422)	2975 (11)	5592 (18)	1176 (11)	63 (6)
C(323)	845 (11)	3271 (17)	−164 (10)	51 (6)	C(423)	3607 (13)	6161 (20)	1278 (12)	77 (8)
C(324)	268 (10)	2943 (16)	−684 (10)	48 (6)	C(424)	3899 (13)	6634 (20)	1861 (12)	80 (8)
C(325)	−105 (10)	2138 (16)	−567 (10)	49 (6)	C(425)	3607 (13)	6481 (20)	2406 (12)	81 (8)
C(326)	65 (11)	1584 (17)	69 (11)	51 (6)	C(426)	2980 (12)	5867 (19)	2352 (11)	68 (8)
C(411)	2031 (11)	3726 (17)	2201 (10)	51 (6)	C(1S)	1443 (13)	7258 (21)	4885 (13)	86 (9)
C(412)	2301 (9)	2773 (15)	2079 (9)	37 (5)	C(2S)	874 (15)	7211 (24)	5138 (15)	109 (10)
C(413)	2543 (11)	2017 (17)	2616 (10)	54 (6)	C(3S)	720 (15)	6299 (23)	5434 (14)	101 (10)
C(414)	2405 (11)	2278 (17)	3255 (11)	58 (6)	C(4S)	1157 (13)	5520 (20)	5447 (12)	82 (9)
C(415)	2142 (11)	3193 (17)	3385 (10)	56 (6)	C(5S)	1706 (13)	5555 (21)	5125 (12)	81 (8)
C(416)	1903 (10)	3959 (17)	2853 (10)	49 (6)	C(6S)	1835 (14)	6445 (21)	4835 (13)	86 (9)

Table 3. *Selected bond distances* (Å) *and bond angles* (°) *for* [Mo(η^6-PhPMePh)(prophos)($PMePh_2$)] [*triclinic* (1) *and monoclinic* (2) *forms*] *and for the related compounds* [Mo(η^6-$PhPMe_2$)(PMe_2Ph)$_3$] (3), [Mo(η^6-PhPMePh)($PMePh_2$)$_2$(CN^{t}Bu)] (4) *and* [Mo(η^6-$PhPPh_2$)(PPh_3)(CN^{n}Bu)]$_2$ (5)

	(1)[a]					(5)[d]	
	Mo(1)	Mo(2)	(2)[a]	(3)[b]	(4)[c]	Mo(1)	Mo(2)
Mo—P(1)	2·430 (3)	2·425 (4)	2·431 (5)	2·434 (4)	2·420 (3)	2·463 (3)	2·458 (3)
Mo—P(2)	2·422 (5)	2·441 (4)	2·422 (5)	2·451 (6)	2·416 (3)	2·472 (3)	2·474 (3)
Mo—P(3)	2·485 (5)	2·486 (4)	2·494 (5)	2·423 (5)	—	—	—
Mo—C(1)	2·32 (2)	2·32 (1)	2·35 (1)	2·30 (1)	2·32 (1)	2·30 (1)	2·36 (1)
Mo—C(2)	2·27 (2)	2·31 (2)	2·26 (2)	2·29 (2)	2·27 (1)	2·27 (1)	2·27 (1)
Mo—C(3)	2·32 (1)	2·29 (2)	2·30 (2)	2·30 (2)	2·30 (1)	2·32 (1)	2·25 (1)
Mo—C(4)	2·25 (1)	2·26 (2)	2·23 (2)	2·27 (2)	2·27 (1)	2·28 (1)	2·26 (1)
Mo—C(5)	2·32 (2)	2·29 (1)	2·31 (2)	2·27 (2)	2·26 (1)	2·29 (1)	2·31 (1)
Mo—C(6)	2·30 (2)	2·26 (1)	2·36 (2)	2·25 (2)	2·24 (1)	2·23 (1)	2·33 (1)
⟨Mo—C⟩	2·30	2·29	2·30	2·28	2·28	2·28	2·30
P(1)MoP(2)	79·5 (1)	79·1 (1)	79·1 (2)	91·6 (2)	94·0 (1)	98·0 (1)	103·1 (1)
P(1)MoP(3)	90·5 (1)	97·4 (1)	90·2 (2)	93·0 (2)	—	—	—
P(2)MoP(3)	92·9 (1)	95·8 (1)	91·5 (2)	89·2 (2)	—	—	—

References: (*a*) This work; (*b*) Mason, Thomas & Heath (1975); (*c*) Luck, Morris & Sawyer (1984*a*); (*d*) Luck, Morris & Sawyer (1984*b*).

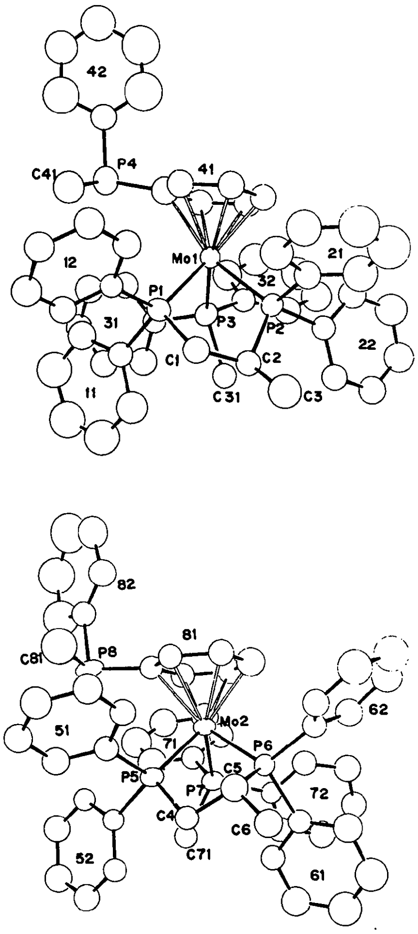

Fig. 1. *ORTEP* (Johnson, 1965) view of both molecules in the triclinic modification. Thermal ellipsoids are drawn at the 50% probability level.

The Mo—P distances show some variation: Mo—P(1) and Mo—P(2) average 2·43 Å whereas Mo—P(3) is significantly different at 2·49 Å. This is the result of steric interactions, the cone angle of prophos being smaller than that for two $PMePh_2$ ligands. However these values are comparable with Mo—P distances in related compounds, *e.g.* 2·44 Å in [Mo(η^6-$PhPMe_2$)(PMe_2Ph)$_3$] (Table 3).

The mean Mo—C bond length in both structures (2·30 Å) is not significantly different from the Mo—C distances observed in the other phosphine complexes in Table 3 nor from the average Mo—C distance in [Mo{η^6-1,3,5-$(CH_3)_3C_6H_3$}(dmpe)]$_2$$N_2$ [2·28 (2) Å; Forder & Prout (1974)]. All these Mo—C distances are significantly shorter than examples where the η^6 ring is *trans* to carbonyl ligands. Typical Mo—C distances in the latter compounds are 2·35–2·38 Å (Alyea, Ferguson & Somogyvari, 1983; Hossain & van der Helm, 1978; Iverson, Hunter, Blount, Damewood & Mislow, 1981; Koshland, Myers & Chesick, 1977).

The atom P(4) is found to bend out of the mean plane through the six C atoms of the η^6-arene ring by 0·122 (4) and 0·227 (5) Å in the two molecules of the triclinic modification and by 0·141 Å for the monoclinic structure. Distortions of similar magnitude have been observed in other molybdenum η^6-arylphosphine compounds (Luck, Morris & Sawyer, 1984*a*,*b*). These distortions reduce the steric interactions between the three σ-bonded and the η^6-bonded phosphine ligands. As in the other structures in Table 3, there is no evidence for any interactions between P(4) and the

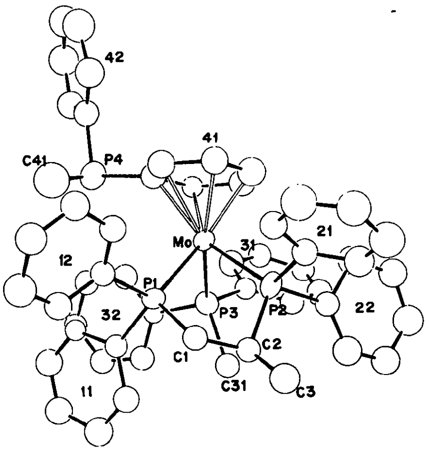

Fig. 2. *ORTEP* view of the molecule in the monoclinic modification.

molybdenum centre. Amongst the phenyl rings of the other P ligands the P atoms deviate from the mean plane of the rings by as much as 0·316 (5) Å.

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Structure of *mer*-Tricarbonyl(triethylphosphine)[vinylidenebis(diphenylphosphine)]tungsten(0)

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Abstract. $[W(CO)_3\{(C_2H_5)_3P\}CH_2C\{(C_6H_5)_2P\}_2]$, $M_r = 782{\cdot}49$, monoclinic, $P2_1/c$, $a = 12{\cdot}813\ (2)$, $b = 12{\cdot}339\ (2)$, $c = 21{\cdot}980\ (3)$ Å, $\beta = 103{\cdot}39\ (1)°$, $U = 3380{\cdot}5\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}536$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 3{\cdot}417$ mm^{-1}, $F(000) = 1560$, room temperature, $R = 0{\cdot}0345$, 3762 unique reflections with $I > 2{\cdot}0\sigma(I)$. The title compound is an octahedrally coordinated mononuclear tungsten(0) complex with three carbonyls arranged in a *mer* configuration, a triethylphosphine and a chelating vinylidenebis(diphenylphosphine). Important bond lengths are W—P(1) 2·500 (4), W—P(2) 2·438 (4), W—P(3) 2·458 (4), ⟨W—C⟩ 1·99 (1), P(1)—C(131) 1·827 (9), P(2)—C(131) 1·835 (9) and C(131)—C(132) 1·32 (1) Å.

Introduction. There are an abundance of complexes containing bis(diphenylphosphino)methane as a monodentate, chelating or bridging ligand (Puddephatt, 1983). Complexes of functionalized variations of this ligand such as vinylidenebis(diphenylphosphine) (vdpp) (Colquhoun & McFarlane, 1982), however, are less common. Complexes of vdpp with transition metals (such as Cr, Mo, W or Pt) are of interest because complex formation increases the activity of the vinylidene group, which readily undergoes Michael-type additions, even at very low temperatures (Cooper, Hassan, Shaw & Thornton-Pett, 1985). In view of this, it is surprising to find that few such complexes have been structurally characterized: examples are confined to a bridged digold complex (Schmidbaur, Herr, Muller & Riede, 1985), a bridged osmium triangle and a mononuclear iron species (Clucas, Dawson, Dolby, Harding, Pearson & Smith, 1986). We present here the structure of a mononuclear tungsten complex.