3 is the bending of the tertiary phosphine ligands away from the chloride site and towards the hydride site. This distortion from linearity of the P—Pt—P axis might be expected to correlate with the steric bulk of the PR_3 ligands. Inspection of Table 3 shows that the $\angle$P—Pt—P is closest to 180° with the smallest ligand (PMe_3). The Pt—Cl(1) distance in (2) is similar to that found in (1) (Table 3), indicating little difference in *cis*-influence for the PPh_3 and P(*p*-tolyl)$_3$ ligands.

The geometry of the P(*p*-tolyl)$_3$ ligands in (2) is unexceptional and the bond lengths and angles associated with this ligand are comparable with those found in other P(*p*-tolyl)$_3$ complexes of platinum-group metals (Kaduk & Ibers, 1977).

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References

Bender, R., Braunstein, P., Jud, J. M. & Dusausory, Y. (1984). *Inorg. Chem.* **23**, 4489–4502.

Collamati, I., Furlani, A. & Attioli, G. (1970). *J. Chem. Soc. A*, pp. 1694–1702.

Eaborn, C., Pidcock, A. & Steele, B. R. (1976). *J. Chem. Soc. Dalton Trans.* pp. 767–776.

Eisenberg, R. & Ibers, J. A. (1965). *Inorg. Chem.* **4**, 773–778.

Frenz, B. A. (1978). *The Enraf-Nonius CAD-4 SDP-A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.

Hartley, F. R. (1973). *The Chemistry of Platinum and Palladium.* Barking: Applied Science Publishers.

International Tables for X-ray Crystallography (1974). Vol IV; Table 2.2, pp. 71–102; Table 2.3.1, pp. 149–150. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Kaduk, J. A. & Ibers, J. A. (1977). *Inorg. Chem.* **16**, 3278–3282.

Packett, D. L., Jensen, G. M., Cowan, R. L., Strouse, C. E. & Trogler, W. C. (1985). *Inorg. Chem.* **24**, 3578–3583.

Tolman, C. A. (1977). *Chem. Rev.* **77**, 313–348.

Acta Cryst. (1990). C**46**, 51–54

Structures of Two Isomorphous Fe—Phosphine Complexes. II. (η-C_5H_5)Fe(CO)(COMe)(PPh_3) (1) and (η-MeC_5H_4)Fe(CO)(COMe)(PPh_3) (2)

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Abstract. The complexes are monoclinic, $P2_1/c$, λ(Mo $K\alpha$) = 0·71073 Å, T = 293 K. (1) Acetyl(carbonyl)(η^5-cyclopentadienyl)(triphenylphosphine)iron, $[Fe(C_2H_3O)(C_5H_5)(CO)\{P(C_6H_5)_3\}]$, M_r = 454·29, a = 7·903 (3), b = 15·884 (6), c = 17·763 (6) Å, β = 96·21 (4)°, V = 2216·7 (1·7) Å^3, Z = 4, D_x = 1·361, D_m = 1·38 g cm^{-3}, μ = 7·7 cm^{-1}, F(000) = 944, $R(F)$ = 0·061, wR = 0·048 for 1992 reflections with $F_o > 3\sigma(F_o)$. (2) Acetyl(carbonyl)(η^5-methylcyclopentadienyl)(triphenylphosphine)iron, $[Fe(C_2H_3O)(C_6H_7)(CO)\{P(C_6H_5)_3\}]$, M_r = 468·32, a = 7·873 (2), b = 16·018 (6), c = 18·048 (7) Å, β = 96·15 (3)°, V = 2262·9 (1·3) Å^3, Z = 4, D_x = 1·374, D_m = 1·36 g cm^{-3}, μ = 7·6 cm^{-1}, F(000) = 976, $R(F)$ = 0·065, wR = 0·047 for 1769 reflections with $F_0 > 3\sigma(F_o)$. The Fe—P bond length is 2·202 (2) Å for (1) and 2·195 (2) Å for (2), and the torsion angles O(2)—C(2)—Fe—(CO) [C(2) = acetyl carbon] are 18 (*anti*) and 22° (*anti*), respectively. In both structures the Fe atom is located centrally above the five-membered ring; Fe—ring-center 1·752 (5) and 1·746 (8) Å for (1) and (2) respectively.

Introduction. Complexes of the type (η-C_5H_5)-Fe(CO)(COMe)L and (η-MeC_5H_4)Fe(CO)(COMe)L where L is a phosphine ligand are very useful in our studies of quantitative analysis of ligand effects (QALE) (Rahman, Liu, Eriks, Prock & Giering, 1989). The QALE model allows one to separate σ and π effects in the Fe—L bond and, more generally, it postulates that electronic and steric ligand effects are separable. Furthermore, the model leads to the conclusion that the Fe—P bond distance is a good diagnostic of σ-bonded or π-bonded phosphine ligands. The abrupt onset of steric effects (steric threshold) for σ-donor ligands suggests that

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the length of the M—P bond is essentially constant and independent of the σ donicity of the ligand. Accordingly, we have begun a systematic study of this bond length by determining the crystal structures of a series of complexes with varying phosphine ligands. The two structures reported here contain the ligand PPh_3, which is a pure σ donor and thus should give rise to a relatively long Fe—P bond length.

Experimental. The complexes were prepared by refluxing (η-C_5H_5)Fe$(CO)_2$Me and (η-MeC_5H_4)-Fe$(C)_2$Me, respectively, and excess ligand, PPh_3, in acetonitrile solution under nitrogen (Green & Westlake, 1971). Dark-orange crystals of (η-C_5H_5)-Fe(CO)(COMe)PPh_3 [hereafter denoted (1)] and (η-MeC_5H_4)Fe(CO)(COMe)PPh_3 [hereafter denoted (2)] were grown by slow crystallization from an acetonitrile solution. The density of each of the crystals was measured by flotation in mixtures of cyclohexane and carbon tetrachloride. Suitable crystals were mounted at the end of a glass fiber with epoxy cement and were centered on a Nicolet $P2_1$ automated diffractometer equipped with an Mo tube and Nb filter. Lattice parameters were calculated from 15 reflections in a 2θ range of 7·1–20·9° for (1) and 6·0–23·2° for (2). All information about data collection for both crystals is listed in Table 1. The two crystals were found to be isomorphous. Intensities were corrected for Lorentz and polarization effects. No absorption correction was applied, since the value of μ is only 7·7 and 7·6 cm^{-1} for (1) and (2), respectively.

For the determination of the structure of (1), Fe and P positions were located from a Patterson map, after which successive least-squares and Fourier calculations yielded parameters for all the non-H atoms. Initial refinements used isotropic thermal parameters, which were later changed to anisotropic thermal motion. At this stage most of the H atoms could be located from a difference Fourier map. The remaining H atoms were placed in calculated positions at 1·00 Å from carbon. In the final least-squares calculations, the H atoms were included but not refined, and they were assigned U values of 1·3 times the U of the carbon to which they are attached. The final values of $R = \sum||F_o| - |F_c||/\sum|F_o|$ and $wR = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$ were 0·061 and 0·048, where $w = [\sigma(F_o)]^{-2}$ and $\sigma(F_o)$ is obtained from counting statistics.

For the determination of the structure of (2) the positions of (1) were used as starting parameters, since the two are isomorphous. The additional methyl C atom was found from a difference Fourier map. H-atom positions and thermal parameters were treated as in (1) above, and final refinement calculations were also identical to those of (1). The final values of the residuals were $R = 0·065$ and $wR = 0·047$. Final atomic coordinates for the non-H atoms in both complexes are given in Table 2.*

Table 1. *Data-collection and refinement parameters*

	(1)	(2)
Crystal size (mm)	0·29 × 0·28 × 0·34	0·12 × 0·21 × 0·28
Scan method	$\omega/2\theta$	$\omega/2\theta$
Scan range (°)	2·0–2·3	2·0–2·3
Scan rate (° min^{-1})	3·91–29·30	3·91–29·30
2θ (°)	$4 < 2\theta < 45·0$	$4 < 2\theta < 45·0$
Reflections measured; h, k, l	$0\rightarrow8$, $-17\rightarrow17$, $-19\rightarrow19$	$0\rightarrow8$, $-17\rightarrow17$, $-19\rightarrow19$
R_{int}	0·075	0·081
No. of reflections measured	6552	6694
No. of unique reflections with $F > 3\sigma$	1992	1769
No. of parameters refined	271	280
Standard reflections	$\bar{2}\bar{1}4$, $\bar{2}5\bar{3}$, 104	$3\bar{2}\bar{2}$, $1\bar{1}0$, 100
Decay of standards	None	None
Max./min. transmission	0·806/0·754	0·913/0·841
$R(F)$	0·061	0·065
wR	0·048	0·047
Goodness-of-fit	1·570	1·265
$\Delta\rho$(min.) (e Å^{-3})	−0·70	−0·80
$\Delta\rho$(max.) (e Å^{-3})	+0·80	+0·80
Max. shift/e.s.d.	0·01	0·02

All computations were performed with the *UCLA Crystallographic Program Package* (Strouse, 1979) as modified at Argonne National Laboratory. Atomic scattering factors, including anomalous scattering, were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. Selected bond lengths and angles for both complexes are shown in Table 3. Fig. 1 shows the molecule for compound (2) and gives the numbering of the atoms. The figure for (1) is identical to the one shown except for the absence of the methyl group on the five-membered ring. Orientations of the cyclopentadienyl and phenyl rings are identical in both cases.

The important Fe—P bond distance is 2·202 (2) Å in (1) and 2·195 (2) Å in (2). The phosphine ligand PPh_3 is a pure σ donor (Rahman *et al.*, 1989), and the QALE model predicts that for this ligand the Fe—P bond length should be long, around 2·20 Å, as was already found in the structure with a PPh_2Et ligand reported previously (Liu, Rahman, Koh, Eriks, Giering & Prock, 1989). In that paper a number of structures from the literature are also listed, all of which tend to support the prediction of the QALE analysis, *viz* that pure σ-donor ligands should exhibit long Fe—P bond distances, whereas σ-donor/π-acceptor ligands should have shorter distances, around 2·10 Å.

In the two isomorphous complexes reported here, the O atoms in O(2)—C(2)—Fe—(CO) [C(2) = acetyl

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* Lists of structure factors, anisotropic thermal parameters, hydrogen coordinates and dihedral angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52153 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Positional and equivalent isotropic thermal parameters*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}(Å^2 × 10^3)
(η-C_5H_5)Fe(CO)(COMe)PPh_3				
Fe	0·1521 (1)	0·3096 (1)	0·3837 (1)	51 (< 1)
P	0·0201 (2)	0·2068 (1)	0·3181 (1)	46 (1)
C(1)	−0·0326 (9)	0·3465 (4)	0·4134 (4)	64 (3)
C(2)	0·1941 (8)	0·2367 (5)	0·4697 (4)	63 (3)
C(3)	0·1148 (11)	0·2498 (6)	0·5412 (5)	107 (4)
C(4)	0·3969 (8)	0·3119 (5)	0·3464 (6)	77 (3)
C(5)	0·2844 (9)	0·3528 (5)	0·2926 (4)	66 (3)
C(6)	0·2159 (9)	0·4213 (5)	0·3260 (5)	71 (3)
C(7)	0·2850 (11)	0·4240 (5)	0·4035 (6)	82 (4)
C(8)	0·3984 (10)	0·3562 (6)	0·4145 (5)	85 (4)
C(9)	−0·0916 (7)	0·1304 (4)	0·3709 (3)	44 (2)
C(10)	−0·2256 (8)	0·1570 (4)	0·4094 (4)	59 (3)
C(11)	−0·3163 (8)	0·1010 (5)	0·4495 (4)	67 (3)
C(12)	−0·2731 (10)	0·0169 (5)	0·4530 (4)	78 (4)
C(13)	−0·1400 (10)	−0·0105 (5)	0·4151 (5)	88 (4)
C(14)	−0·0508 (8)	0·0457 (4)	0·3741 (4)	71 (3)
C(15)	−0·1501 (7)	0·2402 (4)	0·2459 (3)	48 (2)
C(16)	−0·2809 (8)	0·1863 (4)	0·2188 (4)	66 (3)
C(17)	−0·4053 (9)	0·2113 (5)	0·1636 (4)	81 (3)
C(18)	−0·4024 (8)	0·2901 (5)	0·1332 (4)	73 (3)
C(19)	−0·2741 (9)	0·3452 (4)	0·1588 (4)	75 (3)
C(20)	−0·1490 (8)	0·3200 (4)	0·2158 (4)	61 (3)
C(21)	0·1516 (8)	0·1385 (4)	0·2659 (4)	48 (3)
C(22)	0·3008 (8)	0·1045 (4)	0·3038 (4)	64 (3)
C(23)	0·3944 (8)	0·0466 (5)	0·2676 (5)	74 (3)
C(24)	0·3454 (11)	0·0218 (5)	0·1947 (6)	83 (4)
C(25)	0·2005 (11)	0·0551 (5)	0·1562 (4)	81 (4)
C(26)	0·1043 (8)	0·1141 (4)	0·1922 (4)	61 (3)
O(1)	−0·1557 (7)	0·3751 (3)	0·4345 (3)	99 (3)
O(2)	0·2944 (7)	0·1770 (3)	0·4720 (3)	95 (2)
(η-MeC_5H_4)Fe(CO)(COMe)PPh_3				
Fe	0·1586 (1)	0·3038 (1)	0·3825 (1)	36 (< 1)
P	0·0254 (2)	0·1987 (1)	0·3237 (1)	33 (1)
C(1)	−0·0260 (12)	0·3428 (5)	0·4084 (5)	47 (4)
C(2)	0·2001 (10)	0·2392 (5)	0·4722 (5)	41 (3)
C(3)	0·1275 (12)	0·2626 (6)	0·5436 (5)	79 (5)
C(4)	0·4032 (10)	0·3001 (6)	0·3478 (5)	54 (4)
C(5)	0·2943 (11)	0·3362 (6)	0·2916 (5)	49 (4)
C(6)	0·2248 (10)	0·4081 (5)	0·3186 (5)	44 (4)
C(7)	0·2954 (10)	0·4179 (5)	0·3937 (5)	42 (4)
C(7*a*)	0·2646 (13)	0·4897 (6)	0·4446 (6)	80 (5)
C(8)	0·4089 (11)	0·3503 (6)	0·4112 (5)	50 (4)
C(9)	−0·0883 (10)	0·1244 (5)	0·3782 (4)	36 (3)
C(10)	−0·2144 (10)	0·1548 (5)	0·4194 (5)	44 (3)
C(11)	−0·3082 (10)	0·1006 (6)	0·4592 (5)	53 (4)
C(12)	−0·2726 (12)	0·0167 (6)	0·4603 (5)	60 (4)
C(13)	−0·1481 (13)	−0·0133 (6)	0·4209 (6)	64 (4)
C(14)	−0·0567 (10)	0·0396 (5)	0·3794 (5)	49 (4)
C(15)	−0·1443 (10)	0·2284 (5)	0·2512 (4)	36 (3)
C(16)	−0·2794 (10)	0·1754 (5)	0·2273 (4)	42 (3)
C(17)	−0·4030 (10)	0·1985 (7)	0·1717 (5)	59 (4)
C(18)	−0·3957 (12)	0·2744 (6)	0·1377 (5)	59 (4)
C(19)	−0·2638 (12)	0·3280 (6)	0·1602 (5)	60 (4)
C(20)	−0·1416 (10)	0·3046 (6)	0·2162 (4)	49 (3)
C(21)	0·1594 (10)	0·1274 (5)	0·2747 (5)	36 (3)
C(22)	0·3048 (10)	0·0939 (5)	0·3154 (4)	38 (3)
C(23)	0·3996 (11)	0·0346 (6)	0·2820 (6)	55 (4)
C(24)	0·3530 (12)	0·0087 (5)	0·2112 (6)	59 (4)
C(25)	0·2130 (13)	0·0442 (6)	0·1703 (5)	57 (4)
C(26)	0·1162 (10)	0·1039 (5)	0·2021 (5)	40 (3)
O(1)	−0·1530 (9)	0·3731 (4)	0·4255 (4)	75 (3)
O(2)	0·2966 (8)	0·1796 (4)	0·4797 (3)	70 (3)

Table 3. *Selected bond distances* (Å) *and angles* (°) *in* (η-C_5H_5)Fe(CO)(COMe)(PPh_3) *and* (η-MeC_5H_4)Fe(CO)(COMe)(PPh_3)

	(1)	(2)		(1)	(2)
Fe—P	2·202 (2)	2·195 (2)	C(7)—C(7*a*)	—	1·508 (11)
Fe—C(1)	1·708 (8)	1·694 (10)	C(9)—C(10)	1·387 (8)	1·390 (10)
Fe—C(2)	1·917 (8)	1·921 (9)	C(9)—C(14)	1·383 (8)	1·381 (10)
Fe—C(4)	2·112 (7)	2·088 (8)	C(10)—C(11)	1·386 (8)	1·388 (10)
Fe—C(5)	2·132 (7)	2·116 (8)	C(11)—C(12)	1·379 (9)	1·373 (11)
Fe—C(6)	2·137 (7)	2·126 (8)	C(12)—C(13)	1·379 (9)	1·358 (11)
Fe—C(7)	2·110 (7)	2·120 (8)	C(13)—C(14)	1·391 (9)	1·382 (11)
Fe—C(8)	2·099 (7)	2·118 (8)	C(15)—C(16)	1·388 (8)	1·393 (10)
P—C(9)	1·820 (6)	1·838 (8)	C(15)—C(20)	1·376 (8)	1·375 (10)
P—C(15)	1·834 (6)	1·830 (8)	C(16)—C(17)	1·370 (8)	1·372 (10)
P—C(21)	1·823 (6)	1·845 (8)	C(17)—C(18)	1·363 (9)	1·366 (11)
C(1)—O(1)	1·171 (7)	1·181 (9)	C(18)—C(19)	1·379 (9)	1·375 (11)
C(2)—O(2)	1·234 (7)	1·219 (8)	C(19)—C(20)	1·394 (8)	1·372 (10)
C(2)—C(3)	1·490 (10)	1·511 (11)	C(21)—C(22)	1·402 (8)	1·399 (10)
C(4)—C(5)	1·393 (9)	1·383 (11)	C(21)—C(26)	1·377 (8)	1·369 (10)
C(4)—C(8)	1·398 (10)	1·394 (11)	C(22)—C(23)	1·381 (9)	1·386 (10)
C(5)—C(6)	1·378 (9)	1·386 (10)	C(23)—C(24)	1·370 (10)	1·355 (11)
C(6)—C(7)	1·426 (10)	1·418 (10)	C(24)—C(25)	1·374 (10)	1·382 (11)
C(7)—C(8)	1·401 (10)	1·418 (10)	C(25)—C(26)	1·404 (9)	1·384 (10)
C(1)—Fe—C(2)	92·4 (3)	92·2 (4)	C(9)—P—C(21)	101·6 (3)	101·1 (4)
C(1)—Fe—P	92·6 (2)	92·1 (3)	C(15)—P—C(21)	103·4 (3)	103·2 (4)
C(2)—Fe—P	90·3 (2)	91·4 (3)	Fe—C(1)—O(1)	177·0 (6)	177·3 (8)
Fe—P—C(9)	116·8 (2)	118·2 (3)	O(2)—C(2)—C(3)	114·0 (7)	112·9 (8)
Fe—P—C(15)	115·1 (2)	114·9 (3)	Fe—C(2)—O(2)	123·2 (6)	124·3 (7)
Fe—P—C(21)	116·6 (2)	116·0 (3)	Fe—C(2)—C(3)	122·8 (6)	122·7 (6)
C(9)—P—C(15)	101·2 (3)	101·0 (4)			

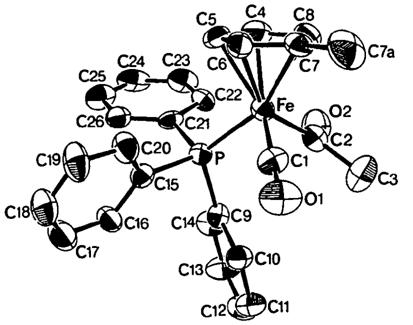

Fig. 1. *ORTEP* plot (Johnson, 1965) of (η-MeC_5H_4)Fe(CO)(COMe)PPH_3. Thermal ellipsoids are drawn at the 50% probability level and H atoms have been omitted for the purpose of clarity.

carbon] are *anti*, and the torsion angles are 18 and 22° in (1) and (2), respectively. This is noteworthy because in the PPh_2Et complex reported earlier this torsion angle was found to be 43°, and in that paper the comment was already made that this value differs considerably from the values of either 0 (*anti*) or 0° (*syn*) that are found theoretically from extended Huckel and *ab initio* SCF MO calculations (Davies, Seeman & Williams, 1986). Furthermore, Davies *et al.* (1986) reported in a footnote in their paper that the structure of the exact same complex (1) was determined by them (no structural information was given in the paper) and that this structure, as well as others, confirms the *anti* orientation of the two O atoms. It is implied in the statements in that paper that the torsion angle is 0° and thus agrees with their theoretical results. Our results presented here, as well as in the earlier complex with the PPh_2Et ligand, appear to be at variance with the structures reported by Davies *et al.* (1986).

Clearly the presence of a methyl group on the cyclopentadienyl ligand does not affect the structure. In both structures the Fe atom is located centrally above the five-membered ring, the average values of the Fe—C distances are 2·118 (11) Å in (1) and 2·114 (10) Å in (2), and the individual Fe—C distances are within 4–5 standard deviations of each other. The distance from Fe to the ring-center is 1·752 (5) Å in (1) and 1·746 (8) Å in (2).

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References

DAVIES, S. G., SEEMAN, J. I. & WILLIAMS, I. H. (1986). *Tetrahedron Lett.* **27**, 619–622.

GREEN, M. & WESTLAKE, D. J. (1971). *J. Chem. Soc. A*, pp. 367–371.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

LIU, H. Y., RAHMAN, MD. M., KOH, L. L., ERIKS, K., GIERING, W. P. & PROCK, A. (1989). *Acta Cryst.* C**45**, 1683–1686.

RAHMAN, MD. M., LIU, H. Y., ERIKS, K., PROCK, A. & GIERING, W. P. (1989). *Organometallics*, **8**, 1–7.

STROUSE, C. (1978). *UCLA Crystallographic Program Package*. Univ. of California, Los Angeles, USA.

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Structure of the Michael Addition Product of Allylamine and a Vinylidene Phosphine Complex of Tungsten

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Abstract. {*N*-[2,2-Bis(diphenylphosphino)ethyl]allylamine-*P*,*P*}tricarbonyl(triphenylphosphine)tungsten(0) allylamine solvate (1:0·5), $[W(CO)_3(C_{18}H_{15}P)(C_{29}H_{29}NP_2)].\frac{1}{2}C_3H_7N$, $M_r = 1040{\cdot}77$ (includes solvate), triclinic, $P\bar{1}$, $a = 11{\cdot}847$ (1), $b = 12{\cdot}335$ (1), $c = 17{\cdot}402$ (2) Å, $\alpha = 87{\cdot}70$ (1), $\beta = 86{\cdot}86$ (1), $\gamma = 73{\cdot}28$ (1)°, $U = 2431{\cdot}0$ (5) Å^3, $Z = 2$, $D_x = 1{\cdot}42$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 23{\cdot}87$ cm^{-1}, $F(000) = 1052$, room temperature, $R = 0{\cdot}0322$, 5930 unique reflections with $I > 2{\cdot}0\sigma(I)$. Coordination of the vinylidenebis(diphenylphosphine) (vdpp) ligand to a W atom in the complex *mer*-$[(OC)_3W(vdpp)(PPh_3)]$ activates the C=C bond which readily undergoes Michael addition with allylamine ($C_3H_5NH_2$) to give *mer*-$[(OC)_3W\{(Ph_2P)_2CHCH_2N(H)C_3H_5\}(PPh_3)]$.

Introduction. The C=C double bond of the functionalized diphosphine vdpp is not susceptible to nucleophilic attack by compounds such as amines, hydrazines, carbon nucleophiles, *etc.* (Colquhoun & McFarlane, 1982). However, upon complexation with transition metals such as Cr, W, Mo or Pt the activity of the vinylidene group is increased and it readily undergoes Michael-type additions (Cooper, Hassan, Shaw & Thornton-Pett, 1985). Here we report the structure of the product of the reaction between $[(OC)_3W(vdpp)\ (PPh_3)]$ and allylamine.

Experimental. Crystals of the title compound were obtained directly by dissolving $[(OC)_3W(vdpp)(PPh_3)]$ in allylamine. The crystals were found to be efflorescent and so one of suitable size (*ca* 0·7 × 0·5 × 0·3 mm) was covered in epoxy resin prior to data collection. All measurements were made on a Nicolet *P*3/*F* diffractometer; 25 reflections centred ($35 \leq 2\theta \leq 40{\cdot}0°$), graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scans, ω scan widths 2·0° + α-doublet separation, scan speeds 2·0–29·3° min^{-1} subject to a fast pre-scan. Complete data set (h0→13, k −14→14, l −19→19; $4{\cdot}0 \leq 2\theta \leq 45°$) measured over 111 hours with no significant decay of a standard reflection (measured every 50 reflections); 6203 unique data, 5930 with $I > 2{\cdot}0\sigma(I)$ considered observed and used in structure solution and refinement. Structure solution by Patterson synthesis (for W atom) and difference Fourier methods; empirical absorption correction (Walker & Stuart, 1983); full-matrix least-squares refinement on F with anisotropic thermal parameters for all non-H atoms with the exception of the terminal —CH_2CHCH_2 group and also a $C_3H_5NH_2$ solvent molecule (which was refined with an occupancy factor of $\frac{1}{2}$) which were refined with isotropic thermal parameters. All H atoms were included in calculated positions and were refined with isotropic temperature factors except for those on the terminal —CH_2CHCH_2 group and the solvent molecule which were not included. Scattering and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Refinement converged to $R = 0{\cdot}0322$, $wR = 0{\cdot}0344$, $w^{-1} = \sigma^2(F_o) + 0{\cdot}0002(F_o)^2$, number of parameters = 486;

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