However, molecular models do suggest that simultaneous rotation of the two rings could bring both N donors into a position for chelate formation (S is unlikely to be a donor). The lack of donor ability of this ligand may, in part, be associated with these steric constraints and the weaker donor capacity of benzothiazoles in general when compared with *e.g.* hydroimidazoles and imidazoles. The molecule does not display any unusual intermolecular contacts and bond lengths and angles are typical of benzothiazole derivatives.

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Structures of Two Acetyl(carbonyl)(cyclopentadienyl)iron Complexes with Phosphine Ligands. III. [(η^5-MeC$_5$H$_4$)Fe(CO)(COMe)(PPh$_2$Me)] (1) and [(η^5-C$_5$H$_5$)Fe(CO)(COMe)(PPhMe$_2$)] (2)*

BY HONG YE LIU, KLAAS ERIKS,† WARREN P. GIERING AND ALFRED PROCK

Metcalf Center for Science and Engineering, Department of Chemistry, Boston University, Boston, MA 02215, USA

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Abstract. (1) Acetyl(carbonyl)(η^5-methylcyclopentadienyl)(methyldiphenylphosphine)iron, [Fe(C$_2$H$_3$O)(CO)(C$_6$H$_7$)(C$_{13}$H$_{13}$P)], $M_r = 406.25$, triclinic, $P\bar{1}$, $a = 9.435$ (3), $b = 8.618$ (2), $c = 13.381$ (4) Å, $\alpha = 97.52$ (2), $\beta = 99.65$ (2), $\gamma = 108.67$ (2)°, $V = 996.2$ (5) Å^3, $Z = 2$, $D_m = 1.35$, $D_x = 1.355$ g cm^{-3}, $\mu = 8.68$ cm^{-1}, $F(000) = 424$, $R(F) = 0.065$, $wR = 0.052$ for 1791 reflections with $F_o > 3\sigma(F_o)$. (2) Acetyl(carbonyl)(η^5-cyclopentadienyl)(dimethylphenylphosphine)iron, [Fe(C$_2$H$_3$O)(CO)(C$_6$H$_5$)(C$_8$H$_{11}$P)], $M_r = 330.15$, triclinic, $P\bar{1}$, $a = 7.509$ (2), $b = 12.961$ (6), $c = 8.691$ (3) Å, $\alpha = 106.83$ (3), $\beta = 93.19$ (3), $\gamma = 97.93$ (3)°, $V = 797.8$ (5) Å^3, $Z = 2$, $D_m = 1.36$, $D_x = 1.374$ g cm^{-3}, $\mu = 10.66$ cm^{-1}, $F(000) = 344$, $R(F) = 0.034$, $wR = 0.038$ for 2069 reflections with $F_o > 3\sigma(F_o)$. λ(Mo $K\alpha$) = 0.71073 Å, $T = 293$ K for (1) and (2). The Fe—P bond distances are 2.185 (2) Å in (1) and 2.180 (1) Å in (2): the shortest Fe—P bond lengths observed thus far for σ-donor ligands in this type of complex. In all probability the absence of steric strain, rather than an electronic effect, is the explanation for these short distances. The torsion angles O(2)—C(2)—Fe—(CO) [C(2) = acetyl carbon] are 33° (*anti*) in (1) and 19° (*anti*) in (2). The additional methyl group in (1) does not cause ring slippage.

Introduction. In two earlier structural papers we reported the geometry of complexes of the type [(η^5-C$_5$H$_5$)Fe(CO)(COMe)L] (L = phosphine ligand) for L = PPh$_2$Et (Liu, Rahman, Koh, Eriks, Giering & Prock, 1989) and L = PPh$_3$ (Liu, Koh, Eriks, Geiring & Prock, 1990). We listed the Fe—P bond lengths for a series of phosphine ligands based on our results and those of others reported in the literature. According to our proposed model of Fe—P σ bonding, the Fe—P bond lengths should be nearly constant, close to 2.20 Å for pure σ-donor ligands in the absence of steric effects, and independent of the σ donicity of the ligand. We also reported that substituent groups on the cyclopentadienyl ring, or on the acetyl group, or on both seem to have very little influence on the Fe—P distance or on the distance from Fe to the five-membered ring. As a continuing effort to understand the requirements of Fe—P bond length, we report below the results of our structural studies of the complexes [(η^5-

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* Part II: Liu, Koh, Eriks, Giering & Prock (1990).

† Author to whom correspondence should be addressed.

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MeC$_5$H$_4$)Fe(CO)(COMe)(PPh$_2$Me)] (1) and [(η^5-C$_5$H$_5$)Fe(CO)(COMe)(PPhMe$_2$)] (2). It would seem that these complexes might be very similar to the L = PPh$_2$Et complex reported in our first paper, but when we found that even their space groups are different from the earlier one we proceeded with the determination of their structures.

Experimental. The complexes (1) and (2) were prepared by refluxing 0.50 g (2.4×10^{-3} mol) of [η^5-MeC$_5$H$_4$)Fe(CO)$_2$Me] and 0.50 g (2.6×10^{-3} mol) of [(η^5-C$_5$H$_5$)Fe(CO)$_2$Me] with excess of the corresponding ligands (twice the molar ratio), PPh$_2$Me (0.96 g) and PPhMe$_2$ (0.72 g), respectively, in nitrogen-purged dry acetonitrile, freshly distilled from phosphorus pentoxide (P$_2$O$_5$), for about 15 h. After completion of the reactions acetonitrile solvent was removed by rotary evaporation. Addition of 50 mL of petroleum ether to the residues in an ice bath gave orange solids. Bright orange crystals of each of the complexes were obtained from a methanol solution under a nitrogen atmosphere. Crystal densities were determined by flotation in a mixture of dimethylformamide (DMF) and carbon tetrachloride (CCl$_4$). Specimens used for the X-ray analysis were obtained by cutting the desired crystals from larger agglomerates.

An approximately rhombohedrally shaped crystal of (1), and a flat block of (2) with thickness equal to one fourth of the length of the sides, were used for data collection. Crystals were mounted on a glass fiber with epoxy cement and placed on a Nicolet $P2_1$ automated diffractometer equipped with a Mo tube and Nb filter. Unit-cell dimensions were determined from 15 reflections in a 2θ range of 17.3–33.3° for (1) and 16.7–34.5° for (2). Intensities were corrected for Lorentz–polarization effects in both (1) and (2). The value of μ of (1) is low (8.68 cm^{-1}) and an absorption scan (ψ scan on the reflection $\bar{2}10$ with χ value of 81.44°) was featureless, so that no absorption correction was applied. An absorption scan of (2) exhibited pronounced peaks, so in this case absorption corrections were applied. All details for data collection for both crystals are given in Table 1.

Table 1. *Data collection and refinement parameters*

	(1)	(2)
Crystal size (mm)	0.14 × 0.18 × 0.19	0.10 × 0.40 × 0.43
Scan method	$2\theta/\omega$	$2\theta/\omega$
Scan range (°)	2.0–2.3	2.0–2.3
Scan rate (° min^{-1})	3.91–29.30	3.91–2930
2θ range (°)	$3.14 < 2\theta < 50$	$3.32 < 2\theta < 47$
Reflections measured, h,k,l	$-10 \rightarrow 10, -9 \rightarrow 9, -14 \rightarrow 14$	$-8 \rightarrow 8, -14 \rightarrow 14, -9 \rightarrow 9$
$R_{\rm int}$	0.093	0.039
No. of reflections measured	5236	4768
No. of unique reflections with $F_o > 3\sigma(F_o)$	1791	2069
No. of parameters refined	235	181
Standard reflections	$\bar{4}0\bar{2}$, $\bar{2}\bar{1}5$, 124	$\bar{3}30$, $\bar{2}\bar{5}1$, $0\bar{1}5$, 003
Decay of standards	None	None
Max./min. transmission	0.8855/0.8554	0.9502/0.8223
$R(F)$	0.065	0.034
wR	0.052	0.038
Goodness of fit	1.406	1.645
$\Delta\rho_{\rm min}$ (e Å^{-3})	−0.75	−0.4
$\Delta\rho_{\rm max}$ (e Å^{-3})	1.05	0.41
Max. shift/e.s.d.	0.01	0.01

For the structure determination of (1), the Fe and P positions were determined from a Patterson map. All non-H atoms were located from subsequent least-squares refinements and difference electron density calculations. The refinements first contained isotropic and later anisotropic thermal parameters, resulting in values of the residuals of $R = 0.080$ and $wR = 0.066$, where $w = [\sigma(F_o)]^{-2}$, and $\sigma(F_o)$ was obtained from counting statistics. The function minimized was $\sum w|F_o^2 - F_c^2|$. At this stage H atoms were included in the calculations. Though most H atoms were visible in the difference map, only three, *viz* one on each of the three methyl groups, were taken from the $(F_o - F_c)$ map. All others were placed in calculated positions with C—H distances of 0.95 Å. In the final calculations the positions of the H atoms were not refined, and they were given an isotropic thermal parameter $U = 1.3$ times the U value of the atom to which they are attached. The final values of the residuals were $R = 0.065$ and $wR = 0.052$.

For the structure determination of (2), all calculational procedures were the same as those for (1) above, except that the starting Fe and P positions were located by direct methods, using the program *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The final values of the residuals for (2) were $R = 0.034$ and $wR = 0.038$. The final atomic coordinates of the non-H atoms for (1) and (2) are shown in Tables 2 and 3, respectively.* Atomic scattering factors, including f' and f'' for Fe, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were performed with the *UCLA Crystallographic Program Package* (Strouse, 1978) as modified at Argonne National Laboratory.

Discussion. Selected bond distances and angles for both complexes are listed in Tables 4 and 5. The *ORTEP* (Johnson, 1965) drawings of (1) and (2) are shown in Figs. 1 and 2, which also show the numbering of the atoms.

The first important results of the study are the Fe—P bond lengths of 2.185 (2) and 2.180 (1) Å in (1) and (2), respectively. Both of these are on the short side of the Fe—P range for σ bonding. The

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, least-squares planes and dihedral angles and diagrams showing the torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54627 (51 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0444]

Table 2. *Positional and thermal parameters* (Å^2 × 10^3) *of atoms in* (1)

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

	x	y	z	U_{eq}
Fe	−0.0103 (1)	0.0022 (1)	0.2437 (1)	38 (0)
P	0.2034 (2)	−0.0356 (3)	−0.3046 (2)	42 (1)
C(1)	0.0594 (9)	0.1028 (10)	0.1516 (6)	53 (4)
C(2)	0.0540 (8)	0.2076 (9)	0.3455 (6)	44 (3)
C(3)	0.0506 (11)	0.3693 (10)	0.3128 (7)	82 (4)
C(4)	−0.1898 (8)	−0.2060 (10)	0.1456 (6)	48 (4)
C(5)	−0.2453 (8)	−0.0746 (10)	0.1691 (6)	46 (4)
C(6)	−0.2275 (8)	−0.0397 (9)	0.2772 (6)	45 (3)
C(7)	−0.1601 (9)	−0.1463 (10)	0.3203 (6)	49 (4)
C(8)	−0.1376 (8)	−0.2495 (9)	0.2385 (7)	52 (4)
C(9)	−0.3203 (10)	−0.0004 (12)	0.0909 (7)	85 (5)
C(10)	0.2539 (9)	−0.1868 (9)	0.2230 (6)	44 (3)
C(11)	0.3751 (9)	−0.2372 (10)	0.2614 (7)	63 (4)
C(12)	0.4138 (10)	−0.3527 (12)	−0.1998 (9)	79 (5)
C(13)	0.3337 (12)	−0.4176 (12)	0.0996 (9)	81 (5)
C(14)	0.2153 (11)	−0.3671 (12)	0.0592 (7)	80 (5)
C(15)	0.1761 (9)	−0.2532 (10)	0.1214 (7)	60 (4)
C(16)	0.3821 (8)	0.1446 (9)	0.3334 (6)	44 (3)
C(17)	0.4512 (9)	0.1946 (10)	0.2533 (7)	57 (4)
C(18)	0.5860 (11)	0.3297 (12)	0.2726 (9)	77 (5)
C(19)	0.6525 (11)	0.4180 (11)	0.3732 (11)	79 (5)
C(20)	0.5852 (11)	0.3714 (12)	0.4534 (8)	75 (5)
C(21)	0.4487 (10)	0.2362 (11)	0.4330 (6)	60 (4)
C(22)	0.2047 (9)	−0.1112 (10)	0.4246 (6)	63 (4)
O(1)	0.1043 (7)	0.1701 (7)	0.0856 (4)	83 (3)
O(2)	0.0927 (6)	0.2175 (7)	0.4378 (4)	73 (3)

Table 3. *Positional and thermal parameters* (Å^2 × 10^3) *of atoms in* (2)

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

	x	y	z	U_{eq}
Fe	0.8939 (1)	0.2399 (0)	0.0465 (1)	35 (0)
P	0.8311 (1)	0.2277 (1)	0.2832 (1)	37 (0)
C(1)	1.0640 (5)	0.1620 (3)	0.0375 (4)	46 (1)
C(2)	1.0507 (4)	0.3778 (3)	0.1561 (4)	47 (1)
C(3)	1.2529 (5)	0.3896 (4)	0.1465 (6)	88 (2)
C(4)	0.6881 (5)	0.3129 (3)	−0.0325 (4)	61 (2)
C(5)	0.8273 (5)	0.3090 (3)	0.1365 (4)	58 (1)
C(6)	0.8435 (5)	0.1993 (3)	−0.2060 (4)	58 (1)
C(7)	0.7105 (5)	0.1335 (3)	−0.1495 (4)	58 (1)
C(8)	0.6172 (4)	0.2043 (4)	−0.0424 (4)	61 (2)
C(9)	1.0252 (4)	0.2259 (3)	0.4177 (4)	42 (1)
C(10)	1.1156 (5)	0.3215 (3)	0.5288 (4)	57 (1)
C(11)	1.2660 (6)	0.3179 (4)	0.6258 (5)	79 (2)
C(12)	1.3268 (6)	0.2206 (5)	0.6123 (6)	93 (2)
C(13)	1.2395 (6)	0.1263 (4)	0.5032 (6)	85 (2)
C(14)	1.0892 (5)	0.1278 (3)	0.4053 (4)	59 (1)
C(15)	0.7194 (5)	0.3345 (3)	0.4057 (4)	61 (1)
C(16)	0.6777 (5)	0.1045 (3)	0.2812 (5)	63 (2)
O(1)	1.1750 (4)	0.1068 (2)	0.0249 (3)	78 (1)
O(2)	0.9936 (4)	0.4601 (2)	0.2265 (3)	69 (1)

Table 4. *Bond lengths* (Å) *and selected angles* (°) *in* (1)

Fe—P	2.185 (2)	C(5)—C(6)	1.408 (10)
Fe—C(1)	1.700 (9)	C(5)—C(9)	1.494 (10)
Fe—C(2)	1.939 (8)	C(6)—C(7)	1.405 (10)
Fe—C(4)	2.121 (7)	C(7)—C(8)	1.406 (10)
Fe—C(5)	2.121 (7)	C(10)—C(11)	1.394 (10)
Fe—C(6)	2.099 (7)	C(10)—C(15)	1.380 (10)
Fe—C(7)	2.088 (7)	C(11)—C(12)	1.386 (11)
Fe—C(8)	2.101 (7)	C(12)—C(13)	1.370 (12)
P—C(10)	1.821 (8)	C(13)—C(14)	1.379 (12)
P—C(16)	1.830 (7)	C(14)—C(15)	1.382 (11)
P—C(22)	1.809 (7)	C(16)—C(17)	1.390 (10)
C(1)—O(1)	1.183 (8)	C(16)—C(21)	1.386 (10)
C(2)—O(2)	1.211 (7)	C(17)—C(18)	1.379 (11)
C(2)—C(3)	1.522 (10)	C(18)—C(19)	1.388 (12)
C(4)—C(8)	1.400 (10)	C(19)—C(20)	1.376 (12)
C(4)—C(5)	1.413 (10)	C(20)—C(21)	1.389 (11)

C(1)—Fe—C(2)	93.1 (4)	C(10)—P—C(22)	103.0 (4)
C(1)—Fe—P	94.1 (3)	C(16)—P—C(22)	103.6 (4)
C(2)—Fe—P	89.9 (2)	Fe—C(1)—O(1)	178.1 (7)
Fe—P—C(10)	116.6 (3)	O(2)—C(2)—C(3)	115.1 (7)
Fe—P—C(16)	117.9 (2)	Fe—C(2)—O(2)	124.2 (6)
Fe—P—C(22)	113.2 (3)	Fe—C(2)—C(3)	120.7 (6)
C(10)—P—C(16)	110.5 (3)		

Cyclopentadienyl ring: C—C—C
Range 107.2–108.7; av: 108.0

Phenyl rings: C—C—C
Range: 117.7–121.8; av: 120.0

Table 5. *Bond lengths* (Å) *and selected angles* (°) *in* (2)

Fe—P	2.180 (1)	C(2)—C(3)	1.514 (5)
Fe—C(1)	1.725 (4)	C(4)—C(8)	1.411 (5)
Fe—C(2)	1.948 (4)	C(4)—C(5)	1.416 (5)
Fe—C(4)	2.102 (3)	C(5)—C(6)	1.400 (5)
Fe—C(5)	2.108 (3)	C(6)—C(7)	1.423 (5)
Fe—C(6)	2.102 (3)	C(7)—C(8)	1.398 (5)
Fe—C(7)	2.134 (4)	C(9)—C(10)	1.389 (5)
Fe—C(8)	2.117 (3)	C(9)—C(14)	1.398 (5)
P—C(9)	1.824 (3)	C(10)—C(11)	1.386 (6)
P—C(15)	1.829 (3)	C(11)—C(12)	1.375 (6)
P—C(16)	1.831 (4)	C(12)—C(13)	1.365 (7)
C(1)—O(1)	1.161 (4)	C(13)—C(14)	1.381 (5)
C(2)—O(2)	1.214 (4)		

C(1)—Fe—C(2)	94.6 (2)	C(9)—P—C(16)	102.6 (2)
C(1)—Fe—P	92.9 (1)	C(15)—P—C(16)	101.7 (2)
C(2)—Fe—P	88.2 (1)	Fe—C(1)—O(1)	177.0 (3)
Fe—P—C(9)	115.0 (1)	O(2)—C(2)—C(3)	116.1 (3)
Fe—P—C(15)	116.5 (1)	Fe—C(2)—O(2)	123.0 (3)
Fe—P—C(16)	115.4 (1)	Fe—C(2)—C(3)	120.7 (3)
C(9)—P—C(15)	103.7 (2)		

Cyclopentadienyl ring C—C—C
Range: 107.2–109.0; av: 108.0

Phenyl ring C—C—C
Range: 119.1–120.5; av: 120.0

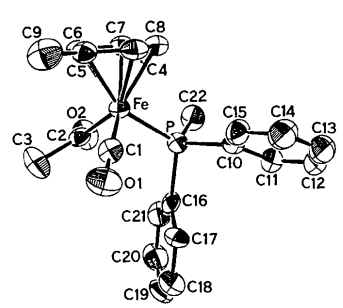

Fig. 1. *ORTEP* (Johnson, 1965) plot of [(η^5-MeC$_5$H$_4$)Fe(Co)(COMe)(PPh$_2$Me)]. Thermal ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

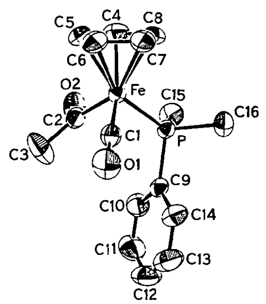

Fig. 2. *ORTEP* (Johnson, 1965) plot of [(η^5-C$_5$H$_5$)Fe(CO)(COMe)(PPhMe$_2$)]. Thermal ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

shortest Fe—P bond length that had previously been reported in the literature was 2.188 (2) Å for the ligand L = PPh$_2$[NHCH(Me)(Ph)], where it was assumed to be the result of the nitrogen hydrogen bonding (Korp & Bernal, 1981). The values of 2.185 (2) and 2.180 (1) Å are 0.01–0.02 Å shorter than the expected bond length of σ donors. In previous reports, the Fe—P bond lengths were 2.200 (2) Å for L = PPh$_2$Et (Liu, Rahman, Koh, Eriks, Giering & Prock, 1989) and 2.202 (2) and 2.195 (2) Å for L = PPh$_3$ (Liu, Koh, Eriks, Giering & Prock, 1990).

A rationalization for the shorter Fe—P bond distances in (1) and (2) may come from a comparison with the PPh$_2$Et complex, which is very similar to (1) in particular. Table 6 lists several properties of the three ligands and their complexes, and shows that the electronic properties of the three are very similar. χ_d (Rahman, Liu, Eriks, Prock & Giering, 1989) is a measure of the basicity of the ligand (the smaller the χ_d value, the stronger the base), and thus of its σ donicity, *i.e.* the ability of the ligand to donate σ electrons to the transition metal. The differences in χ_d values are small to begin with, and furthermore there is no correlation between the χ_d values and the Fe—P distances, so that the small differences in distance cannot be explained by the electronic properties of the ligands. A steric explanation using Tolman's cone angle θ does appear reasonable (Tolman, 1977). The Fe fragment offers only limited space for the phosphine ligand and steric strains can result as the ligand becomes larger, thus resulting in a longer Fe—P distance. Intramolecular H···H contacts indeed indicate that some steric strain is present in the PPh$_2$Et complex, which has a close H···H contact of 2.13 (10) Å between the phosphine ligand and the metal fragment. In the PPh$_2$Me and PPhMe$_2$ complexes the shortest analogous contacts are 2.36 (10) and 2.33 (10) Å, respectively, *i.e.* about two times the normal value of the van der Waals radius of an H atom. The value of 1.2 Å for the H-atom radius was also used in the calculation of the cone angles of phosphine ligands (Ferguson, Roberts, Alyea & Khan, 1978; Alyea, Dias, Ferguson & Restivo, 1977). A similar explanation based on steric strain has been given for the complex [Mo(η^2-COCH$_2$CMe$_3$)Br(PMe$_3$)$_4$], where the average Mo—P distance is 2.518 (6) Å for the (more crowded) equatorial sites, and 2.447 (4) Å for the (less crowded) axial sites (Carmona, Munoz & Rogers, 1988). Of course, in the latter complex the difference in M—P distances is much larger than in our complexes.

The Fe—C(1) and Fe—C(2) distances (Tables 4 and 5) are very similar to the corresponding ones in earlier structures, as are Fe—Cp(average) [2.106 (12) Å in (1) and 2.113 (10) Å in (2)] and Fe—Cp(center) [1.734 (7) Å in (1) and 1.739 (4) Å in (2)]. The Fe atom is located centrally above the five-membered ring in both structures. The additional methyl group in (1) does not cause any ring slippage.

Table 6. *Properties of* PPh$_2$Me, PPh$_2$Et *and* PPhMe$_2$ *and of their corresponding* [(η^5-C$_5$H$_5$)Fe(CO)(COMe)L] *complexes*

	PPh$_2$Me	PPh$_2$Et	PPhMe$_2$
pK_a of PR_3H$^+$	4.57	4.9	6.50
χ_d*	12.1	11.3	10.6
θ (°)†	136	140	122
ν(CO) (cm^{-1})*	1919.9	1919.1	1918.7
δC(CO)‡	220.83	221.50	220.88
^2J(PC) (Hz)‡	31.5 (5)	29.8 (5)	32.6 (5)
Fe—P (Å)	2.185	2.200	2.180

* Rahman, Liu, Eriks, Prock & Giering (1989).

† Tolman's cone angle; Tolman (1977).

‡ Results for [(η^5-C$_5$H$_5$)Fe(CO)(COMe)L] or [(η^5-MeC$_5$H$_4$)Fe(CO)(COMe)L] and for [(η^5-C$_5$H$_5$)Fe(CO)$_2L^+$], L = PPh$_2$Et or PPh$_2$Me, given by Liu (1989).

The torsion angle O(2)—C(2)—Fe—(CO) [C(2) = acetyl carbon] is 33° (*anti*) in (1) and 19° (*anti*) in (2), again, as in the previously reported structures, deviating from the theoretically predicted value of 0° (Davies, Seeman & Williams, 1986; Bodner, Patton, Smith, Georgiou, Tam, Wong, Strouse & Gladysz, 1987).

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