

trans-Tricarbonylbis[diphenyl(benzoylmethyl)phosphine]iron(0)Cui Da-Jun,* Zeng Xianshun,
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Key indicators

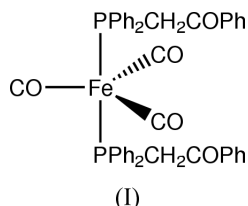
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.029
 wR factor = 0.083
Data-to-parameter ratio = 13.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the crystal structure of the title compound, *trans*-(Ph₂PCH₂COPh)₂Fe(CO)₃, the molecule occupies a special position: the Fe atom and one of the carbonyl groups lie on the twofold axis. The Fe atom has an almost undistorted trigonal-bipyramidal coordination environment. The *trans*-phosphine ligands are in axial positions, the PFeP unit being almost linear [P—Fe—P 178.86 (3)°; Fe—P 2.2113 (4) Å] and orthogonal to the Fe(CO)₃ plane [P—Fe—C 88.85 (6)°, 90.568 (14)°, and 90.61 (6)°].

Comment

For many years we used *trans*-(Ph₂Ppy)₂Fe(CO)₃ (py = pyridine) as a neutral organometallic tridentate ligand and reacted it with various metal derivatives to yield a series of homo- and heterobinuclear complexes (Zhang & Cheng, 1996). Owing to the important role of semilabile ligands containing a combination of soft and hard donor atoms (Jeffrey & Rauchfuss, 1979), we used Ph₂PCH₂COPh (Bouaoud *et al.*, 1986) as a ligand to synthesize *trans*-(Ph₂PCH₂COPh)₂Fe(CO)₃. In the literature method, *trans*-(P)₂Fe(CO)₃ (P = phosphine ligand) is synthesized under basic conditions and some phosphine ligands containing functional groups such as COOH, *etc.* can therefore not be used.

Recently, we described a new synthetic route leading to *trans*-(Ph₂PCH₂COPh)₂Fe(CO)₃ (Cui *et al.*, 2003), and now we report its crystal structure. According to the new synthesis, KOH is reacted with Fe(CO)₅ to give KH(CO)₄, and then H₂SO₄ and phosphine are added. The KH(CO)₄ formed initially is then reacted with H₂SO₄ to give H₂Fe(CO)₄, which is activated by the excess acid and further reacted with the phosphine ligand, thus producing the target complex, *trans*-(Ph₂PCH₂COPh)₂Fe(CO)₃, (I), in high yield.



The structural study has shown that the molecule of the title compound (Fig. 1) occupies a special position: the Fe atom and one of the carbonyl groups lie on a twofold axis. The Fe atom has a trigonal-bipyramidal coordination environment, with two phosphine ligands in axial positions and three carbonyl groups lying in the equatorial plane. The PFeP unit has the expected parameters, with Fe—P distances of 2.2113 (4) Å

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and a P—Fe—P angle of 178.86 (3)°. Its geometry is comparable to those observed in *trans*-(Ph₂Ppyridine)₂Fe(CO)₃ (Fe—P 2.202 Å, P—Fe—P 174.2°; Li *et al.*, 1997) and *trans*-(Ph₂Ppy)₂Fe(CO)₃ (2.206 Å, 177.1°; Zhang *et al.*, 1996). The directions of the Fe—P bonds are almost exactly orthogonal to the equatorial FeC₃ plane, all P—Fe—C angles being close to 90° [P1—Fe1—C21 88.85 (6), P1—Fe1—C22 90.568 (14), P1—Fe1—C21ⁱ 90.61 (6)°; symmetry code: (i) 1 - x, y, $\frac{1}{2}$ - z]. The C—Fe—C angles are not much different from the ideal value of 120° [C21—Fe1—C22 118.71 (6)°].

Experimental

The synthesis was carried out under an Ar atmosphere. After a mixture of KOH (0.26 g, 4.6 mmol) and Fe(CO)₅ (0.3 ml, 2.3 mmol) in CH₃OH (20 ml) was stirred for 30 min, H₂SO₄ (0.46 g, 4.6 mmol) in 10 ml of CH₃OH and Ph₂PCH₂COPh (1.39 g, 4.6 mmol) in 5 ml of CH₂Cl₂ were added. After stirring for 2 h, the resulting yellow solid was collected by filtration and dried *in vacuo*. Yield 1.43 g (83%).

Crystal data

[Fe(C ₂₀ H ₁₇ OP)(CO) ₃]	$D_x = 1.369 \text{ Mg m}^{-3}$
$M_r = 748.49$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 553 reflections
$a = 20.4621 (14) \text{ \AA}$	$\theta = 2.6\text{--}22.3^\circ$
$b = 8.6575 (6) \text{ \AA}$	$\mu = 0.55 \text{ mm}^{-1}$
$c = 20.9787 (14) \text{ \AA}$	$T = 298 (2) \text{ K}$
$\beta = 102.342 (1)^\circ$	Plate, yellow
$V = 3630.5 (4) \text{ \AA}^3$	$0.40 \times 0.35 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker CCD area-detector diffractometer	3203 independent reflections
φ and ω scans	2656 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.010$
$T_{\text{min}} = 0.874$, $T_{\text{max}} = 0.936$	$\theta_{\text{max}} = 25.0^\circ$
7325 measured reflections	$h = -20 \rightarrow 24$
	$k = -10 \rightarrow 10$
	$l = -23 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 1.7565P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.083$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
3203 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
232 parameters	
H-atom parameters constrained	

All H atoms were placed in geometrically calculated positions and included in the refinement in the riding motion approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ of the carrier C atom.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998) and SHELXTL (Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick,

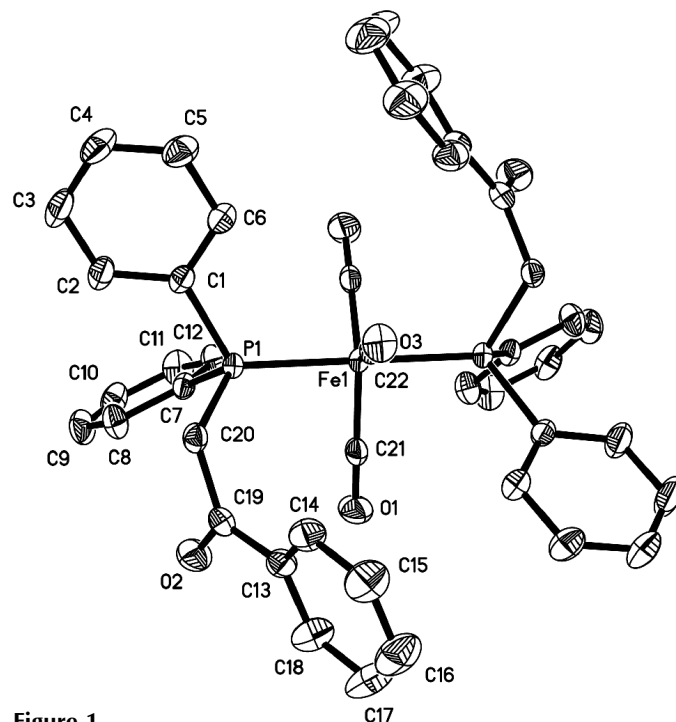


Figure 1 Molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level; the H atoms are omitted.

1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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