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Tetracarbonylbis[(pentafluoroethyl)diphenylphosphine]chromium(0)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.035
wR factor = 0.087
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title complex, $[Cr(C_{14}H_{10}F_5P)_2(CO)_4]$, a slightly distorted octahedral geometry is present about the Cr atom which sits at a center of inversion, while the groups about the P atom are in a slightly distorted tetrahedral arrangement. The Cr—P—C angles are larger than the C—P—C angles of the ligand. The $—C_2F_5$ groups are in the E configuration across the center of inversion.

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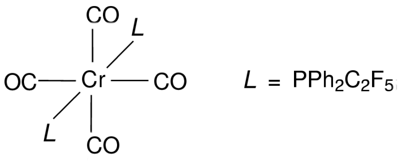
Comment

Phosphine compounds with fluoroalkyl substituents are being prepared in an effort to develop phosphine-ligand systems that approximate the electronic influence of phosphites. The structure of the title compound, (I), was undertaken to establish the connectivity of the first of these substances.



(I)

The Cr—P distance of 2.3368 (6) Å is slightly longer than the metal–phosphorus distance [2.252 (1) Å] in *trans*-$[(PhO)_3P]_2Cr(CO)_4$ (Preston *et al.*, 1972). This is probably due to increased steric strain, as the phenyl groups are closer to the P atom in the title compound than are the phenyl groups in the phosphite. However, the Cr—C bond distance in the title compound shows no similar elongation relative to the phosphite analog [average of 1.895 (4) Å in the title compound *versus* an average of 1.88 (1) Å].

A slight distortion from octahedral geometry is present immediately around the Cr which sits at a center of inversion. The two axial P-containing ligands have Cr—P vectors which are approximately perpendicular (*cf.* Table 1) to the essentially square planar arrangement [r.m.s. deviation of $Cr(CO)_4$ of 0.013 Å; see also Table 1] of the four carbonyls around the Cr atom. The phenyl and $–C_2F_5$ groups are also disposed in the E configuration about the inversion centre. As expected, the carbonyl groups are nearly linear (Table 1).

The ligand geometry indicates that the rings and C_2F_5 groups are staggered with respect to the four carbonyls (see torsion angles in Table 1). The P1—C15—C16—F4 grouping of atoms is in a planar zigzag conformation (r.m.s. deviation = 0.043 Å). This is probably a result of the only significant, albeit weak, hydrogen bonding/dipole–dipole bond interaction, involving H8 with F2, in which a six-membered ring (P1—C3—C8—H8···F2—C15) is formed in a near half-chair conformation. C15 is 0.858 Å from the mean plane of the P1—

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C3—C8—H8···F2 atoms (r.m.s. deviation = 0.042 Å)

The P1—C3 and P1—C9 bonds are significantly (28σ) shorter than the P1—C15 bond. The average P—C_{ring} bond length in the title compound is ~10σ longer than the average P—C_{ring} length [~1.786 (5) Å] in the four organophosphorus compounds reported by Gifkins & Jacobson (1980), Lapp & Jacobson (1980), and Baker & Baughman (1995). In these compounds, the P atom is doubly bonded to either an S or O atom, an –OMe or –OEt group, or a tri-halogen-substituted –OPh group. Thus, the elongation of the P—C_{ring} bonds is likely to be a result of the inductive effects of the –C_2F_5 group.

Experimental

The title compound was prepared by thermolysis of $Cr(CO)_6$ with two equivalents of the ligand in refluxing octane. The solution was filtered, and removal of the alkane solvent under vacuum was followed by isolation of the product from a petroleum ether slurry at 195 K. Crystals were grown by slow evaporation of a 1:1 solution in a petroleum ether/ether mixture at room temperature.

Crystal data

$[Cr(C_{14}H_{10}F_5P)_2(CO)_4]$
M_r = 772.44
Triclinic, $P\bar{1}$
a = 8.1720 (3) Å
b = 9.3069 (4) Å
c = 11.8450 (5) Å
α = 110.015 (4)°
β = 93.301 (4)°
γ = 102.208 (3)°
V = 819.00 (6) Å^3
Z = 1
D_x = 1.566 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 100 reflections
θ = 10.5–18.1°
μ = 0.54 mm^{-1}
T = 293 (2) K
Trapezoidal plate, pale yellow
0.31 × 0.25 × 0.19 mm

Data collection

Siemens Bruker *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: by integration (*XSCANS*; Bruker, 1996)
T_{min} = 0.873, T_{max} = 0.924
3507 measured reflections
2847 independent reflections
2366 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.0°
$h = -9 \rightarrow 1$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: average of 1.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.087
S = 1.08
2842 reflections
223 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.4223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—C2	1.894 (2)	P1—C3	1.833 (2)
Cr1—C1	1.896 (3)	P1—C9	1.835 (2)
Cr1—P1	2.3368 (6)	P1—C15	1.918 (3)
C1—Cr1—P1	93.83 (8)	C3—P1—C9	102.16 (11)
C1—Cr1—C2	88.55 (10)	C3—P1—C15	99.58 (11)
C2—Cr1—P1	91.80 (7)	C9—P1—C15	98.81 (11)
C3—P1—Cr1	113.08 (8)	O1—C1—Cr1	177.7 (2)
C9—P1—Cr1	121.34 (8)	O2—C2—Cr1	178.9 (2)
C15—P1—Cr1	118.45 (8)		
C3—P1—Cr1—C1	99.5 (1)	C15—P1—Cr1—C1	−16.4 (1)
C9—P1—Cr1—C1	−138.7 (1)		

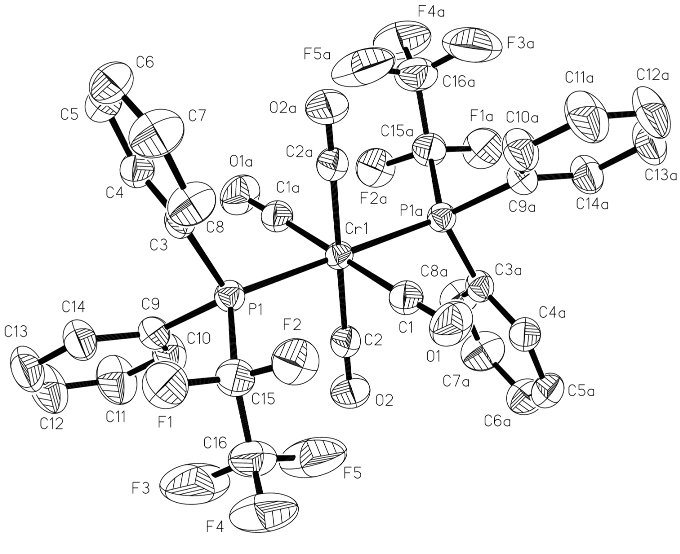

Figure 1
View of the title molecule, showing the labeling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···F2	0.96	2.38	3.111 (4)	133

The C—H bond lengths were constrained at 0.90 Å.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*); software used to prepare material for publication: *SHELXTL/PC* (Sheldrick, 1990*b*) and *SHELXL*93.

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