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Metal Complexes of *trans*-2,2,4,4-Tetrafluoro-1,3-di-*tert*-butyl-1,3-diphosphetane

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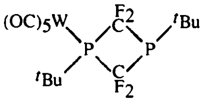
Abstract

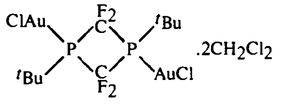
The title ligand, *L*, forms metal complexes $LW(CO)_5$ {pentacarbonyl(*trans*-1,3-di-*tert*-butyl-2,2,4,4-tetrafluoro-1,3-diphosphetane-*P*)tungsten, $[W(C_{10}H_{18}F_4P_2)(CO)_5]$, (1)} and $L(AuCl)_2.2CH_2Cl_2$ {μ-(*trans*-1,3-di-*tert*-butyl-2,2,4,4-tetrafluoro-1,3-diphosphetane-*P*:*P'*)-bis(chlorogold) dichloromethane solvate, $[Au_2Cl_2(C_{10}H_{18}F_4P_2)].2CH_2Cl_2$, (2)}. Complex (1) crystallizes with two independent but closely similar molecules; the P—W bond is short [2.482/2.488 (2) Å] and the P—C_{Bu} bonds are lengthened by *ca* 0.03 Å compared with those in the ligand disulfide, LS_2 [Fild, Jones, Ruhnau & Thöne (1994). *Z. Naturforsch. Teil B*, **49**, 1361–1367; the structure of *L* is not available for comparison]. The diphosphetane ring is folded by an angle of 21.4 (3)/22.0 (1)° about the C···C axis, in contrast with its exact planarity in both LS_2 and complex (2), which crystallizes with imposed *2/m* symmetry (*m* for the solvent); the Au—P bond is slightly shorter than the normal range at 2.212 (2) Å. Short contacts of 3.604 (2) Å are observed between the Au and solvent Cl atoms.

Comment

We reported recently the preparation of *trans*-2,2,4,4-tetrafluoro-1,3-di-*tert*-butyl-1,3-diphosphetane (hereafter *L*) (Fild, Jones, Ruhnau & Thöne, 1994). The structure of *L* was not determined (lack of suitable single crystals), but we were able to determine the structure of the disulfide, LS_2. Here, we extend our studies to the structure of two metal complexes of *L*, namely the tungsten, (I), and gold, (II), complexes.



(1)



(2)

Complex (1), $LW(CO)_5$ (Fig. 1), crystallizes with two independent molecules in the asymmetric unit. These molecules are related by a non-crystallographic glide plane. In contrast with the disulfide, LS_2, the central diphosphetane ring is not exactly planar; the fold angle about the C···C axis is 21.4 (3)/22.0 (1)°.

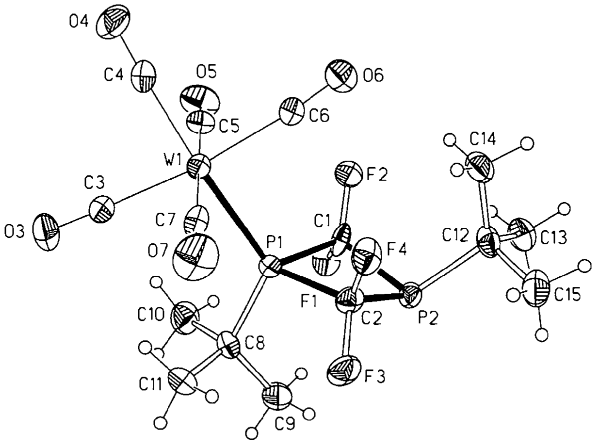


Fig. 1. One of the two independent molecules of (1) in the crystal. Ellipsoids correspond to 50% probability levels. H-atom radii are arbitrary.

Complex (2), $L(AuCl)_2.2CH_2Cl_2$ (Fig. 2), crystallizes with imposed *2/m* symmetry; atoms Au, Cl1, P, F, C2 and C3 lie in the mirror plane $(x,\frac{1}{2},z)$ (as do the solvent C and Cl atoms), and the C1 atom on the twofold axis $(\frac{1}{2},y,\frac{1}{2})$. The diphosphetane ring is thus exactly planar.

The corresponding diphenyl (rather than di-*tert*-butyl) ligand (Fild *et al.*, 1994) forms a bis[pentacarbonylchromium(0)] complex (Jones, 1996) in which the geometric consequences of complexation are easy to summarize: with respect to the free ligand, the P—CF_2 bonds are lengthened by *ca* 0.015 Å, whereas all other bond lengths and angles are scarcely altered. As indicated above, the structure of the current di-*tert*-butyl ligand, *L*, is unfortunately not available, only that of the corresponding disulfide, LS_2. Correspondingly, the results are less easy to interpret. Some effects of steric

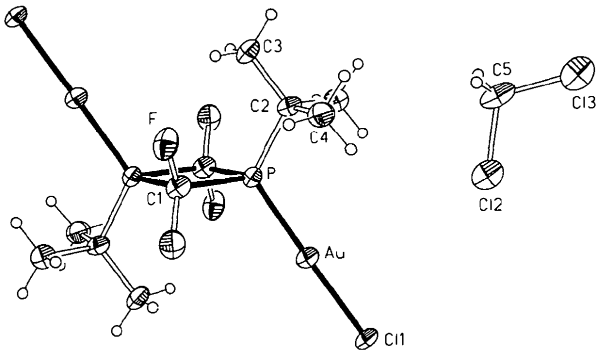


Fig. 2. Complex (2) in the crystal. Ellipsoids correspond to 50% probability levels. H-atom radii are arbitrary and only the asymmetric unit is numbered.

crowding might be expected for the tungsten complex, (1). Indeed, the formation of a mono- rather than bis-tungsten complex is probably a chemical consequence [*cf.* the less bulky AuCl group, of which two can be accommodated by *L* in complex (2)].

The major difference between LS_2 and complex (1) is the lengthening of the P—C_{Bu} bond from 1.847 (2) to 1.878/1.886 (6) Å. This cannot, however, be solely for steric reasons because the uncoordinated P—C_{Bu} bonds are also long [1.867/1.903 (6) Å]. In complex (2), the corresponding bond length is 1.846 (9) Å. Some crowding is nevertheless indicated by the wide C_{Bu}—P—W angle of 122.0/121.4 (2)° [*cf.* C_{Bu}—P—Au 117.3 (3)° in complex (2)]. Furthermore, the C_{Bu}—P—C_{ring} angles are compressed from 110.90/111.38 (8)° in LS_2 to 107.9/107.8/105.3/105.9 (3)° in complex (1), but are 111.0 (2)° in (2).

The P—CF_2 bond lengths [1.905/1.900 (2) Å] and P—C—P angle [96.32 (11)°] in LS_2 show no significant trends compared with those in the title complexes (see Tables 2 and 4), except that in (1), the bonds to the non-coordinating (three-coordinate) P atom are *ca* 0.02 Å shorter, as expected. The C—P—C angle of 83.94 (8)° is narrowed to 80.9/79.8 (3)° for the coordinated P atom and to 82.1/81.2 (3)° for the uncoordinated P atom (associated with the loss of planarity of the ring) in complex (1), but remains essentially unchanged in complex (2) [83.9 (4)°].

The P—W bond length of 2.482/2.488 (2) Å in (1) corresponds to the lower range of known P—W bonds; a search of the Cambridge Structural Database (Allen & Kennard, 1993) found 26 such bonds for carbon-substituted phosphines, with a range of 2.469–2.686 Å. The average, however, is only 2.526 Å. For $R_3PW(CO)_5$, the P—W bond length is 2.516 (2) Å for *R* = Me (Cotton, Darensbourg & Kolthammer, 1981), but much longer at 2.686 (4) Å for *R* = ^{t}Bu, presumably because of extreme steric effects (Pickardt, Rosch & Schumann, 1976).

The bond lengths at the Au atom in (2) may be considered normal, although Au—P is slightly short at 2.212 (2) Å. We have already noted the tendency of phosphines bearing electronegative substituents to form short Au—P bonds [*e.g.* 2.198 (2) Å in Cl_3PAuCl; Jones & Bembenek, 1992, and references therein].

In complex (2), short intermolecular Au···Cl2($\frac{3}{2}-x$, $\frac{1}{2}-y$, $1-z$) contacts of 3.604 (2) Å connect the complex and the solvent into bands parallel to the *y* axis (Fig. 3). No unusually short contacts are observed in complex (1).

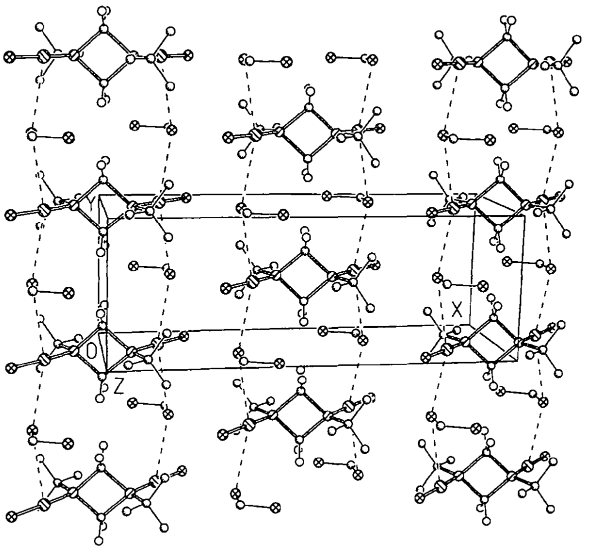


Fig. 3. Packing diagram of (2). H atoms are omitted and radii are arbitrary. Short Au···Cl contacts (see text) are indicated by dashed lines.

Experimental

Complex (1) was obtained from pre-irradiated $W(CO)_6$ and the ligand *L* in THF, purified chromatographically and recrystallized from petroleum ether (Ruhnau, 1992). Complex (2) was obtained from (tht)AuCl (tht = tetrahydrothiophene) and the ligand *L* in dichloromethane, and recrystallized from dichloromethane/diethyl ether (Ruhnau, 1992).

Compound (1)

Crystal data

$[W(C_{10}H_{18}F_4P_2)(CO)_5]$
M_r = 600.08
Monoclinic
$P2_1/n$
a = 16.505 (4) Å
b = 11.657 (3) Å
c = 21.918 (6) Å
β = 99.79 (2)°
V = 4155.6 (19) Å^3
Z = 8
D_x = 1.918 Mg m^{-3}
D_m not measured

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 50 reflections
θ = 10.0–11.5°
μ = 5.770 mm^{-1}
T = 178 (2) K
Prism
0.42 × 0.28 × 0.20 mm
Colourless

Data collection

Siemens *R*3 diffractometer
ω scans

R_{int} = 0.0270
θ_{max} = 25.05°

Absorption correction:
ψ scans (*XEMP*; Siemens, 1990)
$T_{\min} = 0.236$, $T_{\max} = 0.315$
8812 measured reflections
7283 independent reflections
5113 observed reflections
[$I > 2\sigma(I)$]

$h = -19 \rightarrow 19$
$k = -13 \rightarrow 2$
$l = -25 \rightarrow 26$
3 standard reflections
monitored every 147 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R(F)[F^2 > 2\sigma(F^2)] = 0.0306$
$wR(F^2) = 0.0786$
$S(F^2) = 0.993$
7283 reflections
499 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 2.105$ e Å^{-3}
$\Delta\rho_{\min} = -0.745$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (1)*

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

	x	y	z	$U_{\rm eq}$
W1	0.416289 (13)	0.10624 (2)	0.131771 (11)	0.02203 (8)
P1	0.40011 (9)	0.27190 (14)	0.19982 (7)	0.0218 (3)
P2	0.41462 (10)	0.51253 (14)	0.19337 (7)	0.0251 (4)
C1	0.4858 (3)	0.3852 (5)	0.2091 (3)	0.0240 (13)
C2	0.3388 (3)	0.3979 (5)	0.1621 (3)	0.0261 (13)
C3	0.3923 (4)	−0.0181 (5)	0.1910 (3)	0.0276 (14)
C4	0.4305 (4)	−0.0197 (5)	0.0713 (3)	0.0282 (14)
C5	0.5390 (4)	0.0960 (6)	0.1697 (3)	0.0324 (15)
C6	0.4397 (4)	0.2141 (5)	0.0632 (3)	0.0290 (15)
C7	0.2942 (4)	0.1186 (5)	0.0987 (3)	0.0293 (14)
C8	0.3667 (4)	0.2536 (5)	0.2770 (3)	0.0311 (14)
C9	0.3584 (4)	0.3645 (6)	0.3126 (3)	0.041 (2)
C10	0.4320 (4)	0.1786 (6)	0.3158 (3)	0.043 (2)
C11	0.2847 (4)	0.1901 (6)	0.2648 (3)	0.042 (2)
C12	0.4419 (4)	0.5983 (5)	0.1278 (3)	0.0301 (14)
C13	0.5126 (4)	0.6770 (6)	0.1571 (3)	0.040 (2)
C14	0.4664 (5)	0.5361 (6)	0.0728 (3)	0.042 (2)
C15	0.3644 (4)	0.6706 (6)	0.1060 (3)	0.041 (2)
F1	0.5307 (2)	0.3904 (3)	0.2689 (2)	0.0372 (9)
F2	0.5398 (2)	0.3653 (3)	0.1728 (2)	0.0338 (9)
F3	0.2630 (2)	0.4144 (3)	0.1784 (2)	0.0386 (9)
F4	0.3228 (2)	0.3851 (3)	0.09956 (15)	0.0347 (9)
O3	0.3762 (3)	−0.0911 (4)	0.2218 (2)	0.0423 (12)
O4	0.4380 (3)	−0.0880 (4)	0.0356 (2)	0.0422 (12)
O5	0.6061 (3)	0.0886 (5)	0.1919 (2)	0.0461 (13)
O6	0.4494 (3)	0.2659 (4)	0.0209 (2)	0.0412 (12)
O7	0.2253 (3)	0.1279 (4)	0.0812 (3)	0.0542 (15)
W1′	0.208138 (14)	0.88895 (2)	0.911358 (11)	0.02333 (8)
P1′	0.10969 (9)	0.72592 (14)	0.90394 (7)	0.0235 (3)
P2′	0.11691 (10)	0.48483 (15)	0.90004 (8)	0.0290 (4)
C1′	0.1206 (3)	0.6085 (5)	0.8468 (2)	0.0179 (11)
C2′	0.1395 (4)	0.6013 (5)	0.9589 (3)	0.0293 (13)
C3′	0.1218 (4)	1.0129 (6)	0.9104 (3)	0.0260 (14)
C4′	0.2930 (4)	1.0134 (6)	0.9189 (3)	0.034 (2)
C5′	0.1939 (4)	0.8985 (6)	0.8170 (3)	0.036 (2)
C6′	0.3039 (4)	0.7772 (6)	0.9137 (3)	0.0293 (15)
C7′	0.2208 (4)	0.8769 (6)	1.0048 (3)	0.0306 (15)
C8′	−0.0036 (3)	0.7503 (5)	0.9026 (3)	0.0278 (14)
C9′	−0.0558 (4)	0.6412 (6)	0.8993 (3)	0.037 (2)
C10′	−0.0339 (4)	0.8229 (6)	0.8449 (3)	0.038 (2)
C11′	−0.0105 (4)	0.8151 (6)	0.9612 (3)	0.035 (2)
C12′	0.2102 (3)	0.3875 (6)	0.9035 (3)	0.035 (2)
C13′	0.1900 (4)	0.3125 (6)	0.8458 (3)	0.041 (2)
C14′	0.2939 (4)	0.4472 (6)	0.9061 (4)	0.048 (2)
C15′	0.2090 (4)	0.3193 (6)	0.9624 (3)	0.039 (2)
F1′	0.0575 (2)	0.6040 (3)	0.7952 (2)	0.0386 (9)
F2′	0.1893 (2)	0.6229 (3)	0.8208 (2)	0.0370 (9)
F3′	0.0945 (2)	0.5920 (3)	1.0061 (2)	0.0390 (9)
F4′	0.2188 (2)	0.6126 (3)	0.9885 (2)	0.0373 (9)
O3′	0.0753 (3)	1.0853 (4)	0.9112 (2)	0.0415 (12)
O4′	0.3426 (3)	1.0832 (4)	0.9229 (3)	0.0510 (14)
O5′	0.1870 (3)	0.9068 (5)	0.7644 (2)	0.061 (2)
O6′	0.3611 (3)	0.7203 (4)	0.9171 (2)	0.0452 (13)
O7′	0.2284 (3)	0.8683 (4)	1.0575 (2)	0.0467 (13)

Table 2. *Selected geometric parameters (Å, °) for (1)*

W1—C3	2.030 (7)	W1′—C3′	2.027 (7)
W1—C4	2.018 (7)	W1′—C4′	2.004 (7)
W1—C6	2.045 (6)	W1′—C5′	2.044 (7)
W1—C5	2.058 (7)	W1′—C6′	2.042 (7)
W1—C7	2.027 (6)	W1′—C7′	2.029 (7)
W1—P1	2.482 (2)	W1′—P1′	2.488 (2)
P1—C1	1.920 (6)	P1′—C1′	1.885 (6)
P1—C2	1.892 (6)	P1′—C2′	1.897 (6)
P1—C8	1.878 (6)	P1′—C8′	1.886 (6)
P2—C1	1.889 (6)	P2′—C1′	1.862 (6)
P2—C2	1.879 (6)	P2′—C2′	1.867 (7)
P2—C12	1.867 (6)	P2′—C12′	1.903 (6)
C1—F1	1.393 (6)	C1′—F1′	1.402 (6)
C1—F2	1.312 (7)	C1′—F2′	1.363 (6)
C2—F3	1.372 (6)	C2′—F3′	1.376 (7)
C2—F4	1.359 (7)	C2′—F4′	1.365 (6)
C4—W1—P1	175.6 (2)	C4′—W1′—P1′	176.5 (2)
C2—P1—C1	80.9 (3)	C1′—P1′—C2′	79.8 (3)
C8—P1—C1	107.9 (3)	C1′—P1′—C8′	107.8 (3)
C8—P1—C2	105.3 (3)	C8′—P1′—C2′	105.9 (3)
C1—P1—W1	116.5 (2)	C1′—P1′—W1′	117.6 (2)
C2—P1—W1	116.5 (2)	C2′—P1′—W1′	116.6 (2)
C8—P1—W1	122.0 (2)	C8′—P1′—W1′	121.4 (2)
C2—P2—C1	82.1 (2)	C1′—P2′—C2′	81.2 (3)
C12—P2—C1	109.7 (3)	C1′—P2′—C12′	112.0 (3)
C12—P2—C2	109.4 (3)	C2′—P2′—C12′	110.0 (3)
P2—C1—P1	95.5 (3)	P2′—C1′—P1′	97.4 (3)
F1—C1—P1	113.2 (4)	F1′—C1′—P1′	114.9 (4)
F1—C1—P2	110.6 (4)	F1′—C1′—P2′	112.2 (4)
F2—C1—P1	112.2 (4)	F2′—C1′—P1′	111.6 (4)
F2—C1—P2	119.6 (4)	F2′—C1′—P2′	118.4 (4)
F2—C1—F1	105.8 (4)	F2′—C1′—F1′	103.0 (4)
P2—C2—P1	96.9 (3)	P2′—C2′—P1′	96.8 (3)
F3—C2—P1	116.0 (4)	F3′—C2′—P1′	115.1 (4)
F3—C2—P2	112.7 (4)	F3′—C2′—P2′	113.1 (4)
F4—C2—P1	110.7 (4)	F4′—C2′—P1′	110.6 (4)
F4—C2—P2	116.7 (4)	F4′—C2′—P2′	117.5 (4)
F4—C2—F3	104.4 (4)	F4′—C2′—F3′	104.2 (5)

Compound (2)

Crystal data

[Au_2Cl_2($C_{10}H_{18}F_4P_2$)].-2CH_2Cl_2
$M_r = 910.87$
Monoclinic
$C2/m$
$a = 19.990$ (6) Å
$b = 7.096$ (3) Å
$c = 8.606$ (3) Å
$\beta = 92.48$ (3)°
$V = 1219.6$ (8) Å^3
$Z = 2$
$D_x = 2.480$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10.0$–$11.5°$
$\mu = 12.831$ mm^{-1}
$T = 178$ (2) K
Prism
0.20 × 0.12 × 0.10 mm
Colourless

Data collection

Siemens *R*3 diffractometer
ω scans

$R_{\rm int} = 0.0427$
$\theta_{\max} = 25.02°$

Absorption correction:
ψ scans (*XEMP*; Siemens, 1990)
$T_{\min} = 0.155$, $T_{\max} = 0.277$
2322 measured reflections
1181 independent reflections
986 observed reflections
$[I > 2\sigma(I)]$

$h = -23 \rightarrow 23$
$k = -8 \rightarrow 8$
$l = 0 \rightarrow 10$
3 standard reflections
monitored every 147 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)[F^2 > 2\sigma(F^2)] = 0.0268$
$wR(F^2) = 0.0618$
$S(F^2) = 1.045$
1181 reflections
73 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0252P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 2.289$ e Å^{-3}
$\Delta\rho_{\min} = -1.218$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (2)*

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

	x	y	z	$U_{\rm eq}$
Au	0.64124 (2)	1/2	0.33587 (4)	0.02612 (14)
Cl1	0.71731 (12)	1/2	0.1453 (2)	0.0263 (5)
P	0.56968 (12)	1/2	0.5249 (3)	0.0213 (5)
F	0.4928 (2)	0.2067 (5)	0.6240 (4)	0.0357 (10)
C1	1/2	0.3227 (13)	1/2	0.027 (2)
C2	0.6049 (5)	1/2	0.7267 (10)	0.028 (2)
C3	0.5530 (5)	1/2	0.8509 (11)	0.037 (3)
C4	0.6491 (4)	0.3237 (11)	0.7393 (8)	0.035 (2)
C5	0.8315 (6)	1/2	0.8319 (11)	0.041 (3)
Cl2	0.8308 (2)	1/2	0.6264 (3)	0.0460 (7)
Cl3	0.9133 (2)	1/2	0.9152 (4)	0.0637 (9)

Table 4. *Selected geometric parameters (Å, °) for (2)*

Au—P	2.212 (2)	F—C1	1.360 (6)
Au—Cl1	2.284 (2)	C2—C3	1.522 (12)
P—C2	1.846 (9)	C2—C4	1.532 (9)
P—C1	1.883 (6)		
P—Au—Cl1	178.55 (10)	F—C1—P	114.7 (2)
C2—P—C1	111.0 (2)	P—C1—P$^{\rm i}$	96.1 (4)
C1—P—C1$^{\rm i}$	83.9 (4)	C3—C2—C4	111.0 (5)
C2—P—Au	117.3 (3)	C4$^{\rm iii}$—C2—C4	109.5 (8)
C1—P—Au	114.53 (12)	C3—C2—P	114.6 (7)
F$^{\rm ii}$—C1—F	105.6 (7)	C4—C2—P	105.2 (4)
F$^{\rm ii}$—C1—P	113.0 (2)		

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, y, 1-z$; (iii) $x, 1-y, z$.

H atoms were refined using a riding model or rigid methyl groups. Similarity restraints (278 in total) were employed for the U components of neighbouring light atoms of complex (1). The largest features of residual electron density (*ca* 2 e Å^{-3}) were located near the CF$_2$ groups (possibly indicating slight disorder) in (1) and in the solvent region in (2).

For both compounds, data collection: *P*3 (Nicolet, 1987); cell refinement: *P*3; data reduction: *XSCANS* (Siemens, 1994*a*); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *XP* (Siemens, 1994*b*); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BS1001). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrafluoroborate Salt of Tricarbonyl(2-methoxy-η^5-cyclohexadienyl)iron(II)

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Abstract

The structure of tricarbonyl[(1,2,3,4,5-η)-2-methoxy-2,4-cyclohexadien-1-yl]iron(1+) tetrafluoroborate, [Fe-