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fac-(2,2′-Bipyridine)(1,1′-dichloroferrocen-2-yl)-trimethylplatinum(IV)

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Reaction of 2-lithio-1,1′-dichloroferrocene (LiCl$_2$Fc) with [PtI(CH$_3$)$_3$(bipy)], where bipy is 2,2′-bipyridine, results in the formation of *fac*-[Pt(CH$_3$)$_3$(Cl$_2$Fc)(bipy)], *i.e.* *fac*-[Pt(CH$_3$)$_3$(C$_{10}$H$_7$Cl$_2$Fe)(C$_{10}$H$_8$N$_2$)]. The three methyl groups of the octahedral metal complex are in the *fac* configuration, with Pt—C bond lengths of 2.072 (11), 2.082 (11) and 2.092 (11) Å, together with a Pt—C(Cl$_2$Fc) distance of 2.146 (11) Å. In one of the two independent molecules in the asymmetric unit, the C atoms of the cyclopentadienyl rings are in an eclipsed conformation, while in the other, they are staggered by 26.7°.

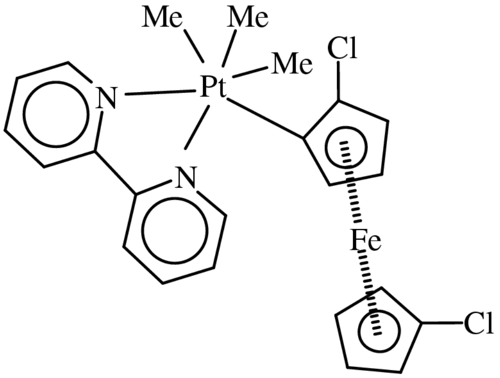
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.019 Å
Disorder in main residue
R factor = 0.061
wR factor = 0.168
Data-to-parameter ratio = 16.9

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

Comment

The title compound, (I), was prepared during an investigation into the stability and reactivity of Pt^{IV} compounds with four Pt—C σ-bonds. The structure of (I) (Fig. 1) shows that the three methyl groups are in the *fac* configuration and hence the ferrocenyl unit is *trans* to a methyl group. For one of the independent molecules, the Pt—C$_{methyl}$ distances are 2.072 (11), 2.082 (11) and 2.092 (11) Å, whilst the Pt—C(Cl$_2$Fc) distance of 2.146 (11) Å lies *trans* to the longest Pt—C$_{methyl}$ distance. The principal distortions from regular octahedral geometry at Pt arise from the small bite angle [N1—Pt—N2 77.1 (3)°] of bipy and the angle C3*M*—Pt—N2 of 99.4 (5)°.



(I)

In one of the independent molecules, the C atoms of the cyclopentadienyl rings are in an eclipsed conformation, with a twist of only 0.5°, while in the other, they are staggered by 26.7°. The Fe centre lies 1.634 (5) Å out of the C1–C5 plane and 1.610 (9) Å from the C6–C10 plane, with a centroid—Fe—centroid angle of 177.8°; the corresponding values for Fe′ in the second molecule are 1.657 (5) Å, 1.653 (9) Å and 173.1°.

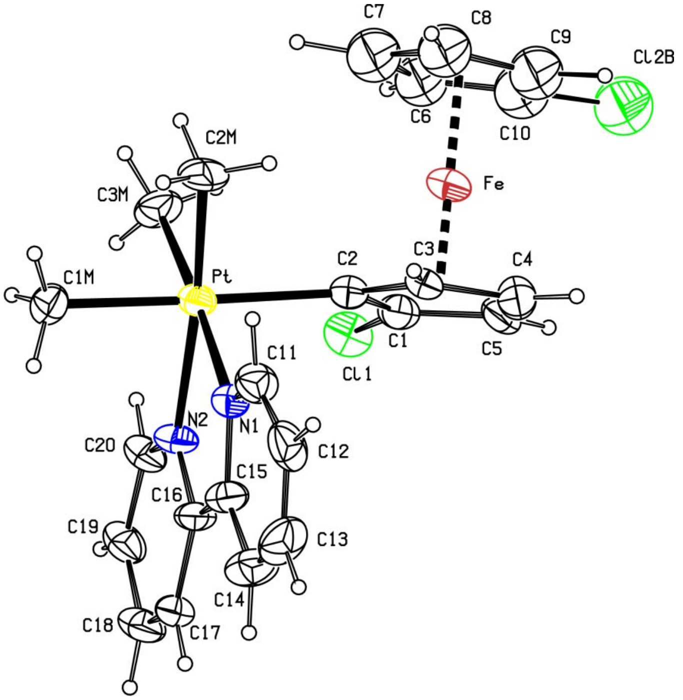


Figure 1

A view of one of the two independent molecules of the title compound in the asymmetric unit, showing the atom-numbering scheme. Only the major [0.602 (14)] component of the disorder of the chlorine substituent on the C_5H_4Cl ring is shown. Displacement ellipsoids are drawn at the 20% probability level.

In both molecules, the cyclopentadienyl rings are inclined at 4.8 (13)°. In one molecule, the disordered chlorines are twisted about the line connecting the ring centroids by 79.2 and 123.4°; in the other, the corresponding values are 82.9 and 116.2°. This is in contrast to the reported structures of other Cl_2Fc complexes (Hollands *et al.*, 1985; Blake *et al.*, 2001) where the chlorines are eclipsed.

Experimental

A solution of *n*-butyl lithium (1.10 mmol) in hexane was added to a stirred solution of 1,1′-dichloroferrocene (0.29 g, 1.13 mmol) (Kovar *et al.*, 1970) in tetrahydrofuran (25 ml). After 1 h, the solution was cooled to 195 K and $[PtIMe_3(bipy)]$ (0.50 g, 0.96 mmol) (Lile & Menzies, 1949) was added. The mixture was allowed to warm slowly to ambient temperature and was then heated under reflux for 3 h. The cooled solution was poured into benzene (50 ml), washed with water (3 x 100 ml), dried over $MgSO_4$ and filtered. Removal of the solvent under vacuum produced a dark-brown oil that was purified by dry-column chromatography on alumina. Hexane eluted 1,1′-dichloroferrocene (0.17 g) and then benzene eluted the title compound which was subsequently crystallized from CH_2Cl_2/hexane to produce orange–red crystals (yield 0.067 g, 11%)

Crystal data

$[Pt(CH_3)_3(C_{10}H_7Cl_2Fe)(C_{10}H_8N_2)]$
$M_r = 650.28$
Monoclinic, $P2_1/n$
$a = 20.137$ (4) Å
$b = 10.572$ (5) Å
$c = 21.810$ (6) Å
$\beta = 103.710$ (10)°
$V = 4511$ (3) Å^3
$Z = 8$

$D_x = 1.915$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17 reflections
$\theta = 9.5$–21.0°
$\mu = 7.09$ mm^{-1}
$T = 295$ (2) K
Plate, orange
0.28 × 0.28 × 0.05 mm

Data collection

Stoe Stadi-2 two-circle diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1983)
$T_{min} = 0.166$, $T_{max} = 0.670$
7797 measured reflections
7797 independent reflections
5558 reflections with $I > 2\sigma(I)$

$R_{int} = 0.009$
$\theta_{max} = 25.0°$
$h = -23 \rightarrow 23$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 25$
3 standard reflections
frequency: 360 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.168$
$S = 1.08$
7797 reflections
461 parameters

H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.098P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 2.43$ e Å^{-3}
$\Delta\rho_{min} = -1.13$ e Å^{-3}

The atoms of the disordered C_5H_4Cl rings were refined as part of a rigid group, with C—C bonds of 1.42 Å, C—C—C angles of 108° and with isotropic displacement parameters. Methyl H atoms were located in difference Fourier syntheses while others were placed geometrically. In the refinement, methyl groups were constrained to be rigid rotating groups with C—H distances at 0.96 Å and with $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were constrained to ride at 0.93 (bipy and C_5H_3Cl) or 1.8 Å (disordered C_5H_4Cl) from their carrier atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. The largest ΔF features occur near the Pt positions.

Data collection: local program; cell refinement: local program; data reduction: local program; program(s) used to solve structure: *SHELX*76 (Sheldrick, 1976); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL*97 and *PLATON*.

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Acta Cryst. (2001). E**57**, m318–m319 Alexander J. Blake *et al.* • $[Pt(CH_3)_3(C_{10}H_7Cl_2Fe)(C_{10}H_8N_2)]$ **m319**