Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Acetyl(2,2′-bipyridine)chloro(1,1′-dichloroferrocen-2-yl)methylplatinum(IV)

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Received 4 June 2001
Accepted 14 June 2001
Online 22 June 2001

Reaction of acetyl chloride with [Pt(Cl_2Fc)(CH_3)(bipy)], where Cl_2Fc and bipy are 1,1′-dichloroferrocen-2-yl and 2,2′-bipyridine, respectively, results in the *trans* oxidative-addition of the acetyl chloride to the Pt^{II} precursor to give the Pt^{IV} title compound, *i.e.* [Pt(CH_3)(C_2H_3O)($C_{10}H_7Cl_2Fe$)Cl($C_{10}H_8N_2$)]. The three Pt—C bonds in the title compound are in a *fac*-configuration and range in length from 2.018 (12) to 2.079 (11) Å. The chloro substituents of the ferrocenyl unit are in an eclipsed conformation.

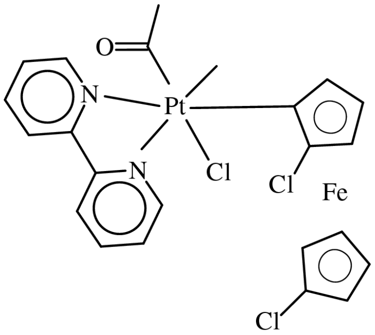
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.018 Å
R factor = 0.054
wR factor = 0.135
Data-to-parameter ratio = 15.8

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 a study of the reactivity in oxidative–addition reactions of the 16-electron complex [Pt(Cl_2Fc)(CH_3)(bipy)]. The structure determination of (I) (Fig. 1), shows that the three Pt—C bonds are in the *fac* configuration and that the acetyl chloride has undergone *trans* addition to the Pt^{II} precursor. The Pt—C bond lengths are 2.018 (12), 2.027 (10) and 2.079 (11) Å for acetyl, ferrocenyl and methyl groups, respectively. The conformation of the ferrocenyl group is that of an eclipsed geometry in which the chlorine substituents also eclipse one another: the two chlorines are twisted about the line joining the two ring centroids by only −1.8 (9)°. The Fe centre lies 1.650 (5) Å out of the C1–C5 plane and 1.645 (5) Å from the C6–C10 plane, with a centroid–Fe–centroid angle of 173.7 (3)°.



(I)

The chlorine substituents are displaced outwards from their respective ring planes, Cl1 by 0.09 (2) Å and Cl2 by 0.12 (2) Å. The two rings are inclined at an angle of 7.1 (9)°, with the longest inter-ring distances involving separations between corresponding atoms [C1···C6 3.406 (15) and C2···C7 3.416 (14) Å] where at least one of these carries a substituent. These parameters suggest a small but significant degree of repulsion between the two rings.

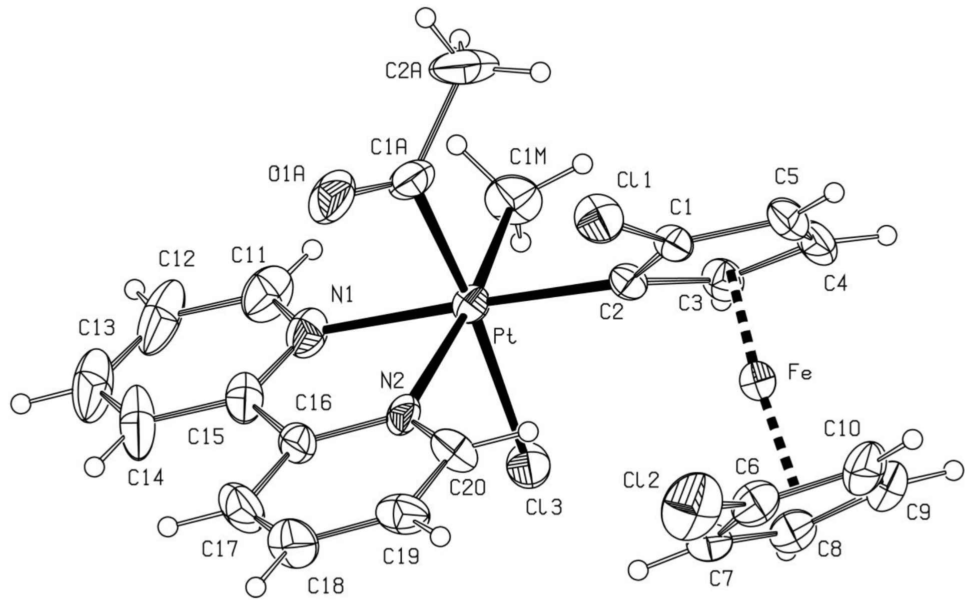


Figure 1
A view of the title compound showing atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Although individual molecules have chiral centres at Pt and at the Cl_2Fc ligand the single crystal selected was found to be racemic.

Experimental

[Pt(Cl_2Fc)(CH_3)(1,5-cyclooctadiene)] (2.52 g, 4.41 mmol) (Hollands *et al.*, 1985) was dissolved in benzene (40 ml) and a solution of bipy (1.51 g, 9.68 mmol) in benzene (10 ml) was added. The solution was heated under reflux for 6 h and then allowed to cool. Brown crystals of [Pt(Cl_2Fc)(CH_3)(bipy)] were isolated by filtration. Yield 2.65 g (97%). [Pt(Cl_2Fc)(CH_3)(bipy)] (0.18 g, 0.29 mmol) was dissolved in dichloromethane (40 ml) and acetyl chloride (0.07 ml, 1.02 mmol) was added. The solution was stirred for 1 h. Evaporation of the solvent, column chromatography of the residue on alumina and elution with benzene/chloroform (1:1) produced firstly unreacted material and secondly an orange oil. Crystallization of the oil from dichloromethane/hexane produced the pure title compound as orange–red crystals. Yield 0.12 g (58%).

Crystal data

[Pt(CH_3)(C_2H_3O)($C_{10}H_7Cl_2Fe$)-Cl($C_{10}H_8N_2$)]
$M_r = 698.71$
Monoclinic, $P2_1/a$
$a = 10.0991$ (11) Å
$b = 14.1486$ (15) Å
$c = 16.205$ (3) Å
$\beta = 100.850$ (10)°
$V = 2274.1$ (5) Å^3
$Z = 4$
$D_x = 2.041$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 80 reflections
$\theta = 15$–16°
$\mu = 7.15$ mm^{-1}
$T = 298$ (2) K
Plate, orange
0.33 × 0.33 × 0.10 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan North *et al.*, 1968
$T_{\min} = 0.034$, $T_{\max} = 0.124$
7096 measured reflections
4453 independent reflections
3164 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 26.0$°
$h = -14 \rightarrow 13$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity decay: 5.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.135$
$S = 1.04$
4453 reflections
282 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2 + 3.92P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.52$ e Å^{-3}
$\Delta\rho_{\min} = -1.54$ e Å^{-3}

Due to the relative weakness of the higher-angle data, only reflections with $2\theta \leq 52°$ were included in the refinement: the corresponding index limits are $-12 \leq h \leq 12, 0 \leq k \leq 17, 0 \leq l \leq 19$. Methyl H atoms were located from ΔF syntheses while others were placed geometrically. In refinement, methyl groups were constrained to be rigid rotating groups with C—H distances at 0.96 Å and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. Other H atoms were constrained to ride at 0.93 Å from their carrier atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *DIF*4 (Stoe & Cie, 1987); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); 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*.

We thank EPSRC (UK) for the provision of a diffractometer.

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m314 A. J. Blake *et al.* • [Pt(CH_3)(C_2H_3O)($C_{10}H_7Cl_2Fe$)Cl($C_{10}H_8N_2$)] *Acta Cryst.* (2001). E**57**, m313–m314