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(Benzylamine-*N*)(η^5-cyclopentadienyl)bis(triphenylphosphine-*P*)ruthenium(II) tetrafluoroborate

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.009 Å
R factor = 0.047
wR factor = 0.133
Data-to-parameter ratio = 13.5

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

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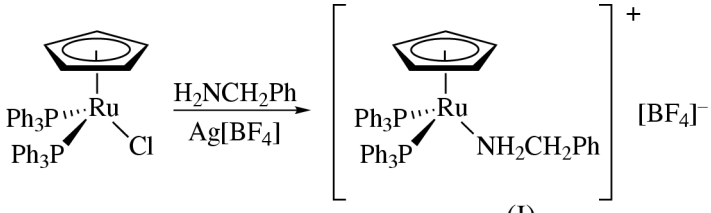
The reaction of $[RuCl(Cp)(PPh_3)_2]$ with $Ag[BF_4]$ in tetrahydrofuran, in the presence of benzylamine, leads to the mononuclear title compound $[Ru(Cp)(H_2NCH_2Ph)(PPh_3)_2]BF_4$, where Cp is cyclopentadienyl (C_5H_5). The Ru atom presents a pseudo-octahedral environment with the Cp ligand occupying three facial coordination sites, while the remaining coordination positions are occupied by the P atoms of the two triphenylphosphine ligands and by the N atom of the benzylamine ligand. The $[BF_4]^-$ counter-ion remains uncoordinated.

Comment

Hydroamination of alkenes and alkynes, which constitutes the formal addition of an N—H bond across a carbon–carbon multiple bond, represents an attractive route to numerous classes of organonitrogen molecules, such as alkylated amines, enamines or imines (Müller & Beller, 1998). Recent advances in catalytic aminations are based on early transition-metal and *f*-element complexes (Schaverien, 1994). However, catalytic additions of amines H—NR_2 to non-activated double or triple bonds mediated by late-transition-metal complexes are still rare (Schaverien, 1994). In this context, we have synthesized the Ru^{II} complex $[(Cp)Ru(H_2NCH_2Ph)(PPh_3)_2][BF_4]$, (I), to use it as a catalyst precursor for amination reactions. The synthesis and crystal structure reported herein is part of this study.



(I)

Experimental

$Ag[BF_4]$ (26.8 mg, 0.138 mmol) was added to a solution of benzylamine (15 µl, 0.138 mmol) and $[RuCl(Cp)(PPh_3)_2]$ (100 mg, 0.138 mmol) in tetrahydrofuran (20 ml) under a nitrogen atmosphere. The color changed immediately to yellow and an off-white solid precipitated. The mixture was stirred at room temperature for 30 min and filtered over celite. The solvent was removed under reduced pressure and the residue redissolved in dichloromethane and precipitated with hexanes. The yellow solid was washed with hexanes (2 × 10 ml) and dried under vacuum to afford 104 mg (85%) of the title compound.

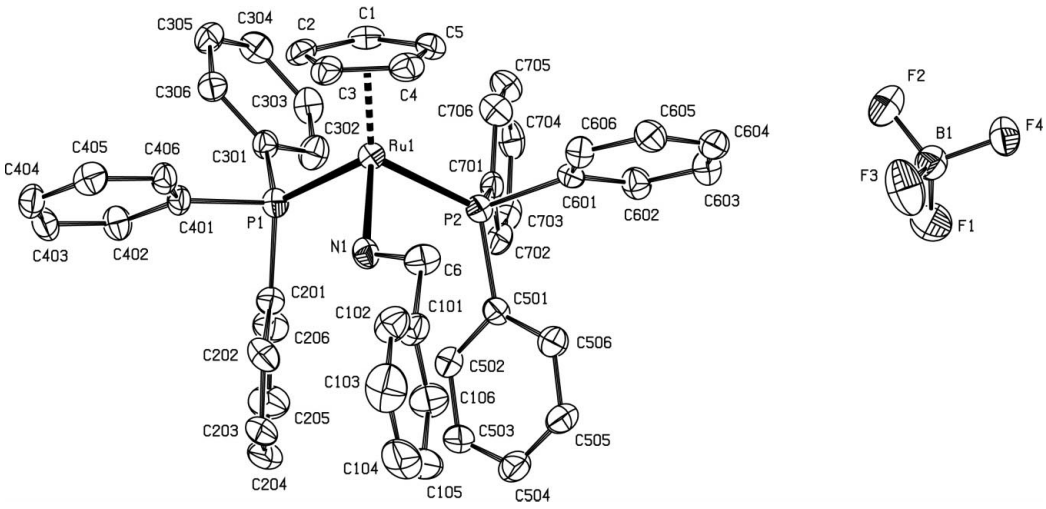


Figure 1
The structure of (I) with ellipsoids at the 50% probability level.

Crystal data

$[Ru(C_5H_5)(C_7H_9N)(C_{18}H_{15}P)_2]BF_4$
M_r = 884.66
Orthorhombic, *Pbca*
a = 13.4255 (3) Å
b = 21.1335 (6) Å
c = 28.6552 (8) Å
V = 8130.3 (4) Å^3
Z = 8
D_x = 1.445 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 22652 reflections
θ = 1.5–70.1°
μ = 4.31 mm^{-1}
T = 200 (2) K
Block, yellow
0.08 × 0.05 × 0.03 mm

Data collection

KappaCCD diffractometer
CCD scans
Absorption correction: empirical (*XABS*2; Parkin *et al.*, 1995)
T_{min} = 0.701, T_{max} = 0.898
54 625 measured reflections
7148 independent reflections
3839 reflections with $I > 2\sigma(I)$
R_{int} = 0.141
θ_{max} = 69.8°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 24$
$l = 0 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 0.90
7148 reflections
530 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

The NH_2 and the CH_2 H atoms were located by Fourier syntheses and their parameters were refined [N—H = 0.84 (4) and 1.00 (8) Å, and C—H = 0.96 (4) and 1.08 (5) Å]. All the other H-atom positions were calculated and refined riding on their parent atoms (C—H = 0.93 Å).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *EUCLID* (Spek, 1982); software used to prepare material for publication: *SHELXL*97.

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