(Benzylamine-N)(η5-cyclopentadienyl)bis(triphenylphosphine-P)-ruthenium(II) tetrafluoroborate

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The reaction of [RuCl(Cp)(PPh₃)₂] with Ag[BF₄] in tetrahydrofuran, in the presence of benzylamine, leads to the mononuclear title compound [Ru(Cp)(H₂NCH₂Ph)(PPh₃)₂][BF₄], where Cp is cyclopentadienyl (C₅H₅). 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 [BF₄]⁻ 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—NR₂ 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 Ruᴵᴵ complex [(Cp)Ru(H₂NCH₂Ph)(PPh₃)₂][BF₄], (I), to use it as a catalyst precursor for amination reactions. The synthesis and crystal structure reported herein is part of this study.
Crystal data

$[\text{Ru} \left( \text{C}_5\text{H}_5 \right) \left( \text{C}_7\text{H}_9\text{N} \right) \left( \text{C}_{18}\text{H}_{15}\text{P} \right)_2] \text{BF}_4$

$M_r = 884.66$

Orthorhombic, Pbca

$a = 13.4255 \pm 3 \text{ Å}$

$b = 21.1335 \pm 6 \text{ Å}$

$c = 28.6552 \pm 8 \text{ Å}$

$V = 8130.3 \pm 4 \text{ Å}^3$

$Z = 8$

$D_x = 1.445 \text{ Mg m}^{-3}$

Cu Kα radiation

Cell parameters from 22652 reflections

$\theta = 1.5$–70.1°

$\mu = 4.31 \text{ mm}^{-1}$

$T = 200 (2) \text{ K}$

Block, yellow

$0.08 	imes 0.05 	imes 0.03 \text{ mm}$

Data collection

KappaCCD diffractometer

CCD scans

Absorption correction: empirical ($XABS2$; Parkin et al., 1995)

$T_{min} = 0.701, T_{max} = 0.898$

54 625 measured reflections

7148 independent reflections

Data collection:

COLLECT (Nonius, 1998); cell refinement:

HKLSCALEPACK (Otwinowski & Minor, 1997); data reduction:

HLDENZO (Otwinowski & Minor, 1997) and SCALEPACK; program(s) used to solve structure:

DIRDIF (Beurskens et al., 1992); program(s) used to refine structure:

SHELXL97 (Sheldrick, 1997); molecular graphics:

EUCLID (Spek, 1982); software used to prepare material for publication:

SHELXL97.

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References


Figure 1

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

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: SHELXL97 (Sheldrick, 1997); molecular graphics: EUCLID (Spek, 1982); software used to prepare material for publication: SHELXL97.