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Key indicators

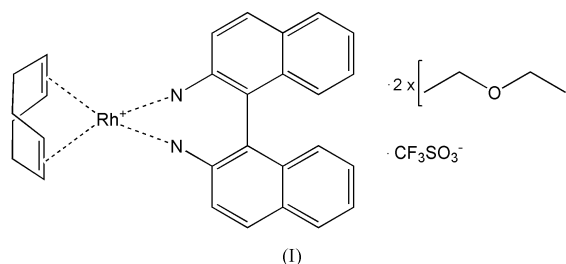
Single-crystal X-ray study
T = 180 K
Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
R factor = 0.038
wR factor = 0.083
Data-to-parameter ratio = 14.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.[(*R*)-(+)-1,1-Binaphthyl-2,2'-diamine]-(1,5-cyclooctadiene)rhodium(I) trifluoromethanesulfonate diethyl ether disolvate

The title compound, $[\text{Rh}(\text{C}_8\text{H}_{12})(\text{C}_{20}\text{H}_{16}\text{N}_2)](\text{CF}_3\text{SO}_3) \cdot 2\text{C}_4\text{H}_{10}\text{O}$, was readily synthesized from the reaction between $[\text{RhCl}(1,5\text{-cyclo-octadiene})]_2$ and (*R*)-(+)-1,1-binaphthyl-2,2'-diamine in tetrahydrofuran. The compound crystallizes in the non-centrosymmetric space group $P2_12_12_1$ and contains one crystallographically unique Rh^+ metal centre in a pseudo-square-planar geometry.

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Comment

Research in the field of asymmetric hydrogenation catalysis has developed significantly in recent years. De Rege *et al.* (2000) have shown that it is possible to immobilize chiral molecules in mesoporous silica *via* non-covalent interactions. Consequently, we are interested in the synthesis and direct application of chiral catalysts for asymmetric hydrogenation catalysis, which takes place within mesoporous materials (Raynor *et al.*, 2000). In particular, we are investigating the use of chiral amines to induce chirality in the final product. For this purpose, we have synthesized the title compound in an analogous fashion to a previous literature precedent (Pertici *et al.*, 1996).



The reaction between $[\text{RhCl}(1,5\text{-cyclo-octadiene})]_2$ and AgCF_3SO_3 in tetrahydrofuran (THF) leads to the formation of the $[\text{Rh}(\text{THF})_2(1,5\text{-cyclo-octadiene})]^+$ cationic species. The addition of (*R*)-(+)-1,1-binaphthyl-2,2'-diamine displaces the THF ligands, affording the title compound (I) in near quantitative yield. The structure contains the complex cation $[\text{Rh}[(\text{R})-(+)-1,1\text{-binaphthyl-2,2'-diamine}](1,5\text{-cyclo-octadiene})]^+$ (Fig. 1), with the Rh^+ metal centre in a pseudo-square-planar geometry. The $\text{Rh}-\text{N}$ and $\text{Rh}-\pi$ bond distances (Table 1) are comparable to those found in similar complexes (Beller *et al.*, 1998).

Experimental

All chemicals were purchased from Aldrich and used without further purification. Solvents were dried and degassed using appropriate methods. Standard Schlenk line techniques were employed. $[\text{RhCl}(1,5\text{-cyclo-octadiene})]_2$ (50 mg) was dissolved in tetrahydrofuran (THF), followed by the addition of AgCF_3SO_3 (55 mg). The

resulting solution was stirred at ambient temperature for one hour, after which it was filtered in order to remove AgCl. The filtrate was added to a solution of (*R*)-(+)-1,1-binaphthyl-2,2'-diamine (60 mg) in THF and stirred for another hour. Yellow crystals of the title compound were precipitated by the addition of hexane (*ca.* 30 ml). The mixture was then filtered, the yellow powder was washed with hexane (3 x *ca.* 20ml) and diethylether (3 x *ca.* 20 ml), and then dried *in vacuo*. Crystals suitable for X-ray diffraction analysis were obtained by recrystallization from a CH₂Cl₂/Et₂O solution. Elemental composition, calculated: C 54.04%, H 4.34%, N 4.34%. Found: C 54.22%, H 4.39%, N 4.20%. M^+ = 495 g mol⁻¹. ¹H NMR (CD₃OD): 1.90 (*br, m*, 4H, CH₂ cod); 2.40 (*br, m*, 4H, CH₂ cod); 4.04 (*br, m*, 2H, CH); 4.42 (*br, m*, 2H, CH); 6.95–8.10 (10H, naph). ¹³C NMR: 30.99; 32.08 (CH₂); 81.16 (*d*, J_{C-Rh} = 12.8 Hz); 82.30 (*d*, J_{C-Rh} = 12.5 Hz); 121.23, 126.02, 128.17, 129.61, 131.71, 134.77, 139.98 (Ph).

Crystal data

[Rh(C ₈ H ₁₂)(C ₂₀ H ₁₆ N ₂)](CF ₃ SO ₃) ⁻ 2C ₄ H ₁₀ O	$D_x = 1.402 \text{ Mg m}^{-3}$
$M_r = 792.74$	Mo $K\alpha$ radiation
Orthorhombic, $P2_12_12_1$	Cell parameters from 14723 reflections
$a = 10.7729 (2) \text{ \AA}$	$\theta = 1.0\text{--}25.0^\circ$
$b = 18.0442 (4) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$c = 19.3180 (5) \text{ \AA}$	$T = 180 (2) \text{ K}$
$V = 3755.19 (15) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.18 \times 0.12 \times 0.07 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer	5644 reflections with $I > 2\sigma(I)$
Thin-slice ω and φ scans	$R_{\text{int}} = 0.049$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$\theta_{\text{max}} = 25.0^\circ$
$T_{\text{min}} = 0.915$, $T_{\text{max}} = 0.955$	$h = -12 \rightarrow 12$
21982 measured reflections	$k = -20 \rightarrow 21$
6615 independent reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 2.0203P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.083$	$(\Delta/\sigma)_{\text{max}} = 0.029$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
6615 reflections	$\Delta\rho_{\text{min}} = -0.57 \text{ e \AA}^{-3}$
447 parameters	Absolute structure: Flack (1983);
H-atom parameters constrained	2872 Friedel pairs
	Flack parameter = $-0.03 (3)$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Rh1—C22	2.117 (4)	Rh1—C21	2.141 (4)
Rh1—C26	2.124 (5)	Rh1—N1	2.143 (3)
Rh1—C25	2.136 (4)	Rh1—N2	2.153 (3)
C22—Rh1—C26	97.38 (18)	C25—Rh1—N1	94.03 (15)
C22—Rh1—C25	82.52 (15)	C21—Rh1—N1	163.46 (18)
C26—Rh1—C25	37.08 (18)	C22—Rh1—N2	88.16 (15)
C22—Rh1—C21	37.70 (18)	C26—Rh1—N2	162.31 (18)
C26—Rh1—C21	82.04 (17)	C25—Rh1—N2	160.47 (17)
C25—Rh1—C21	90.57 (19)	C21—Rh1—N2	92.48 (16)
C22—Rh1—N1	158.79 (17)	N1—Rh1—N2	88.47 (11)
C26—Rh1—N1	92.12 (15)		

H atoms were placed in calculated positions and allowed to ride during subsequent refinement, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$

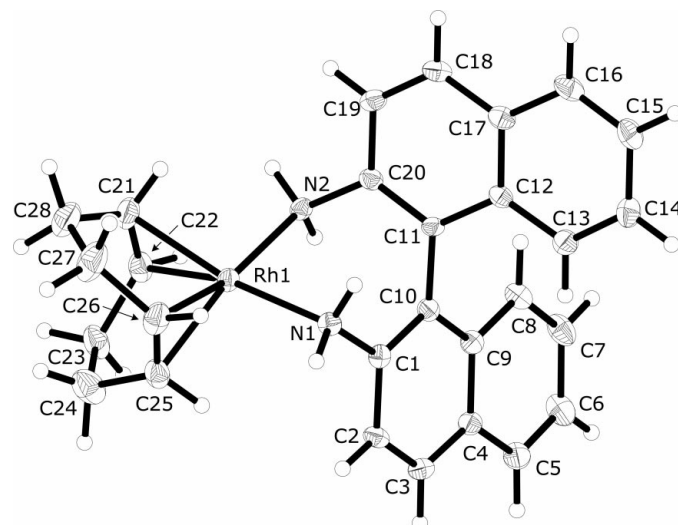


Figure 1

A view of the complex cation, showing the labelling scheme for non-H atoms. Ellipsoids are drawn at the 30% probability level.

for methyl groups and $x = 1.2$ for other H atoms.

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: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Bruker, 2001); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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