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#### Key indicators

Single-crystal X-ray study

$T = 180$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.017$  Å

$R$  factor = 0.041

$wR$  factor = 0.101

Data-to-parameter ratio = 9.3

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

## A new polymorph of [(*S*)-(-)-2-amino-methyl-1-ethylpyrrolidine- $\kappa^2\text{N},\text{N}'$ ]- (1,5-cyclooctadiene)rhodium(I) trifluoromethanesulfonate

The structure of a new polymorph of the title compound,  $[\text{Rh}(\text{C}_8\text{H}_{12})(\text{C}_7\text{H}_{16}\text{N}_2)]\text{CF}_3\text{SO}_3$ , the first form of which we recently reported [Rouzaud *et al.* (2003). *Helv. Chim. Acta*, **86**, 1753–1759], has been determined at 180 K in space group  $C222_1$ . The differences between the two polymorphs are related to the way the individual ions close-pack within the unit cell.

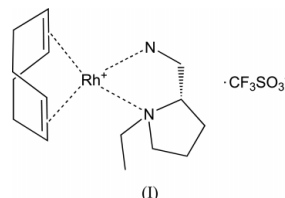
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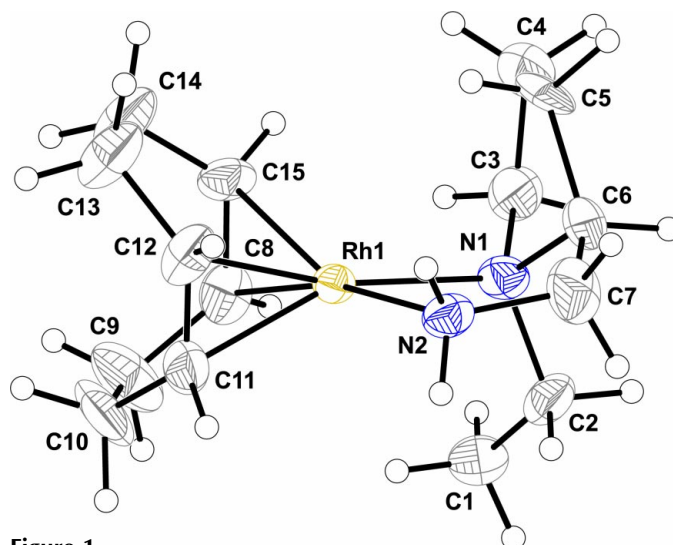
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#### Comment

As part of our work on the synthesis and structural characterization of novel chiral catalysts (Jones, Paz *et al.*, 2003*a,b,c,d*; Jones, Raja *et al.*, 2003; Rouzaud *et al.*, 2003), we recently reported the crystal structure of the first polymorph of the title compound, (I) (Rouzaud *et al.*, 2003), in the orthorhombic space group  $P2_12_12_1$ . While confirming the reproducibility of the synthetic method, we have now determined the structure of a second polymorph.

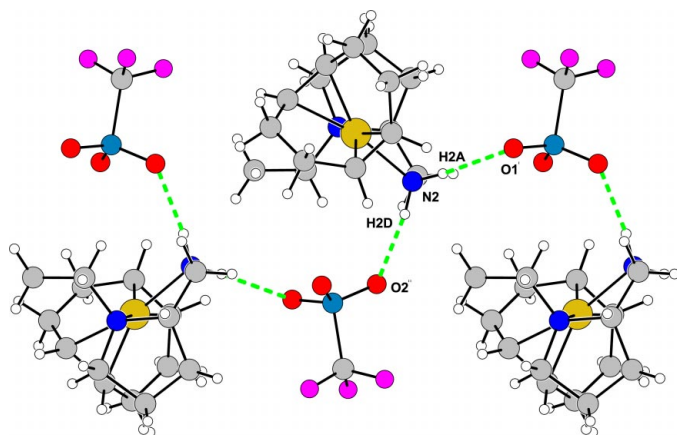


The  $[\text{C}_{15}\text{H}_{28}\text{N}_2\text{Rh}]^+$  complex cation is almost identical in the two polymorphs (Fig. 1), with the  $\text{Rh}^+$  metal centre present in

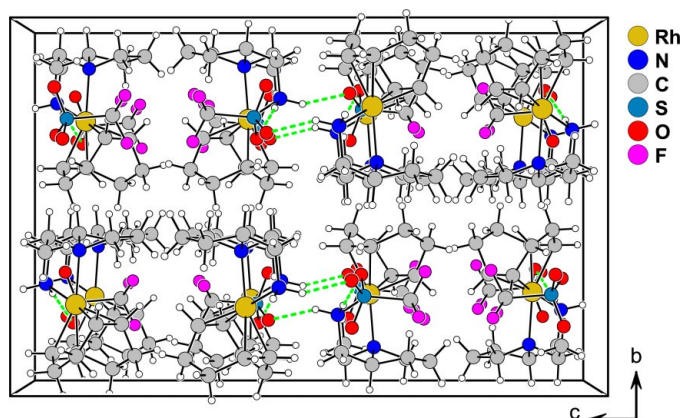


**Figure 1**

The structure of the  $[\text{C}_{15}\text{H}_{28}\text{N}_2\text{Rh}]^+$  complex cation in the title compound, with the atom labelling and displacement ellipsoids drawn at the 30% probability level; H atoms are shown as small spheres.


**Figure 2**

N–H···O hydrogen bonds between the trifluoromethanesulfonate anions and the complex cations (green dashed lines), leading to the formation of zigzag chains which run along the *a*-axis direction. For hydrogen-bonding geometry and symmetry codes, see Table 2.

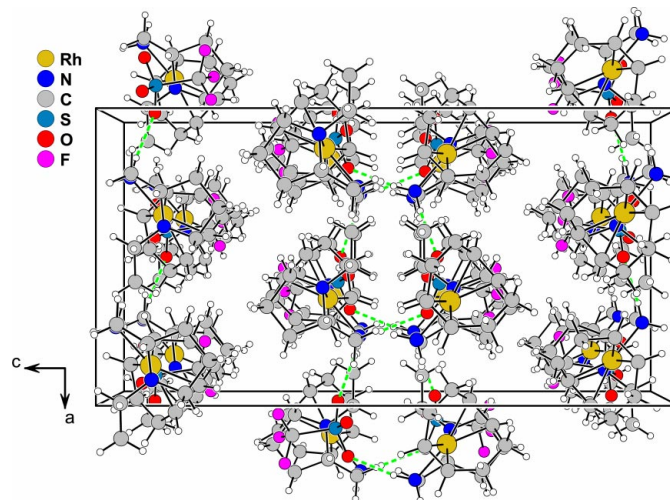

**Figure 3**

Perspective view of (I) along the *a*-axis direction. Hydrogen bonds between the trifluoromethanesulfonate anions and the complex cations are drawn as green dashed lines.

a slightly distorted square-planar geometry, coordinated by one 1,5-cyclooctadiene and one (*S*)-(-)-2-aminomethyl-1-ethylpyrrolidine chiral diamine, and showing similar bond lengths and angles (Table 1), which are consistent with those found in similar complexes. However, the molecular packing is completely different from that in the first polymorph. As previously observed, the trifluoromethanesulfonate anions establish physical bridges between neighbouring  $[\text{C}_{15}\text{H}_{28}\text{N}_2\text{Rh}]^+$  complex cations *via* N–H···O hydrogen bonds, leading to the formation of zigzag chains running along the *a*-axis direction (Fig. 2 and Table 2). Chains related by the twofold axes form layers in the *ab* plane, which alternate in an *ABAB*... fashion along the *c*-axis direction, with two layers per unit cell repeat (Figs. 3 and 4).

## Experimental

All chemicals were purchased from Aldrich and used without further purification. Solvents were dried and degassed using appropriate


**Figure 4**

Perspective view of (I) along the *b*-axis direction. Hydrogen bonds between the trifluoromethanesulfonate anions and the complex cations are drawn as green dashed lines.

methods. Standard Schlenk line techniques were also employed.  $[\text{RhCl}(\text{1,5-cyclooctadiene})_2]$  (100 mg) was dissolved in tetrahydrofuran (*ca* 10 ml), and  $\text{AgCF}_3\text{SO}_3$  (104 mg) was added. The resulting solution was stirred at ambient temperature for 1 h, after which it was filtered in order to remove  $\text{AgCl}$ . (*S*)-(-)-2-Aminomethyl-1-ethylpyrrolidine (52 mg) was added to the filtrate and the resulting solution was stirred for another hour, and then slightly concentrated *in vacuo*. The addition of *ca* 25 ml of hexane led to the precipitation of the title compound as a yellow powder, which was washed with hexane ( $2 \times ca$  20 ml) and diethyl ether ( $2 \times ca$  20 ml), and dried *in vacuo*. Crystals suitable for X-ray diffraction analysis were obtained by recrystallization from a solution in  $\text{CH}_2\text{Cl}_2/\text{Et}_2\text{O}$ .

### Crystal data

$[\text{Rh}(\text{C}_8\text{H}_{12})(\text{C}_7\text{H}_{16}\text{N}_2)]\text{CF}_3\text{SO}_3$   
 $M_r = 488.37$   
 Orthorhombic,  $C222_1$   
 $a = 11.7449$  (5) Å  
 $b = 14.5747$  (7) Å  
 $c = 22.9700$  (12) Å  
 $V = 3932.0$  (3) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.650$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 53723 reflections  
 $\theta = 1.0$ – $22.0^\circ$   
 $\mu = 1.02$  mm<sup>-1</sup>  
 $T = 180$  (2) K  
 Plate, yellow  
 $0.10 \times 0.10 \times 0.02$  mm

### Data collection

Nonius KappaCCD diffractometer  
 Thin-slice  $\omega$  and  $\varphi$  scans  
 Absorption correction: multi-scan  
 (SORTAV; Blessing, 1995)  
 $T_{\min} = 0.899$ ,  $T_{\max} = 0.980$   
 6274 measured reflections  
 2212 independent reflections

2004 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$   
 $\theta_{\text{max}} = 22.0^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -15 \rightarrow 15$   
 $l = -24 \rightarrow 24$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.101$   
 $S = 1.05$   
 2212 reflections  
 239 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 8.7148P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.52$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.38$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.0014 (2)  
 Absolute structure: Flack (1983),  
 878 Friedel pairs  
 Flack parameter =  $-0.06$  (8)

**Table 1**  
Selected geometric parameters (Å, °).

Rh1—C11	2.115 (9)	Rh1—C12	2.135 (10)
Rh1—N2	2.114 (6)	Rh1—C8	2.143 (9)
Rh1—C15	2.121 (9)	Rh1—N1	2.186 (7)
C11—Rh1—N2	94.4 (3)	C12—Rh1—C8	94.5 (4)
C11—Rh1—C15	93.0 (4)	C11—Rh1—N1	155.1 (3)
N2—Rh1—C15	155.8 (3)	N2—Rh1—N1	81.3 (3)
N2—Rh1—C12	90.8 (4)	C15—Rh1—N1	100.9 (3)
C15—Rh1—C12	81.4 (4)	C12—Rh1—N1	165.4 (3)
C11—Rh1—C8	82.8 (4)	C8—Rh1—N1	95.8 (4)
N2—Rh1—C8	166.8 (3)		

**Table 2**  
Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2A...O1 <sup>i</sup>	0.92	2.36	3.273 (16)	171
N2—H2D...O2 <sup>ii</sup>	0.92	2.06	2.908 (10)	153

Symmetry codes: (i)  $\frac{1}{2} + x, \frac{1}{2} + y, z$ ; (ii)  $x, 1 - y, 1 - z$ .

All H atoms were positioned geometrically, and allowed to ride on their parent atoms, with  $U_{\text{iso}}$  values fixed at 1.2 (for CH, methylene and NH<sub>2</sub>) or 1.5 (for the methyl group) times  $U_{\text{eq}}$  of the parent atom. As the trifluoromethanesulfonate anion was poorly resolved, bond-length restraints were applied in order to ensure a reasonable geometry. The highest peak in the final difference map is associated with the trifluoromethanesulfonate anion, 1.37 Å from O2, and the deepest hole is 0.92 Å from Rh1.

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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