

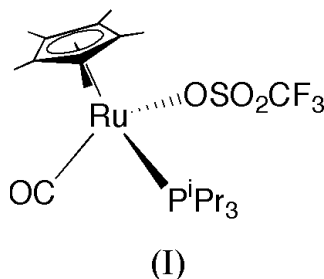
Carbonyl(η^5 -pentamethylcyclopentadienyl)-
(triflate-O)(triisopropylphosphine-P)ruthenium(II)Jill Fuss,^a Valerie C. Pierre^{b*} and
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Key indicators

Single-crystal X-ray study
 $T = 128$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.038
 wR factor = 0.089
Data-to-parameter ratio = 14.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title complex, $[\text{RuCp}^*(\text{OSO}_2\text{CF}_3)\{\text{P}(\text{iPr})_3\}(\text{CO})]$ or $[\text{Ru}(\text{CF}_3\text{O}_3\text{S})(\text{C}_{10}\text{H}_{15})(\text{C}_9\text{H}_{21}\text{P})(\text{CO})]$, consists of an Ru atom coordinated by an inner sphere trifluoromethanesulfonate, a carbonyl, and a triisopropylphosphine in a 'three-legged stool' fashion.Received 9 July 2002
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Comment

Monocyclopentadienyl complexes of group VIII transition metals are of interest because of their participation in a variety of important stoichiometric and catalytic processes. Especially of interest are those group VIII complexes with highly labile substituents, such as trifluoromethanesulfonate groups, since such groups can easily generate a vacant coordination site during chemical reactions.



In the compound studied, the Ru atom is coordinated in a 'three-legged piano-stool' fashion by one O atom of the trifluoromethanesulfonate, a carbonyl, a triisopropylphosphine, and a pentamethylcyclopentadienyl (Cp^*). An examination of the Cambridge Structural Database (CSD; Allen & Kennard, 1993) reveals that even though there exist numerous cationic species containing Cp or Cp^* , a $\text{P}(\text{iPr})_3$ and a carbonyl ligand, there are only four neutral species. The distance between the Ru atom and the coordinated O atom of the trifluoromethanesulfonate [2.195 (3) Å] is longer than that of most Ru–O distances in similar Ru–trifluoromethanesulfonate compounds [2.125 (5)–2.177 (4) Å; Gemel *et al.*, 1997; Svetlanova-Larsen *et al.*, 1996; Burns & Hubbard, 1994; Ontko *et al.*, 1998]; only one of six compounds had a longer distance [2.277 (2) Å; Mauthner *et al.*, 1997]. The Ru–P bond distance [2.395 (1) Å] is comparable with that found in similar Cp^* –Ru–carbonyl–triisopropylphosphine complexes (typically 2.39 Å; Campion *et al.*, 1988; Bernad *et al.*, 2000). The Ru– C_{Cp^*} bond distances [2.203 (4)–2.287 (4) Å] are within the expected range for half-sandwich Ru^{II} compounds (Johnson *et al.*, 1995).

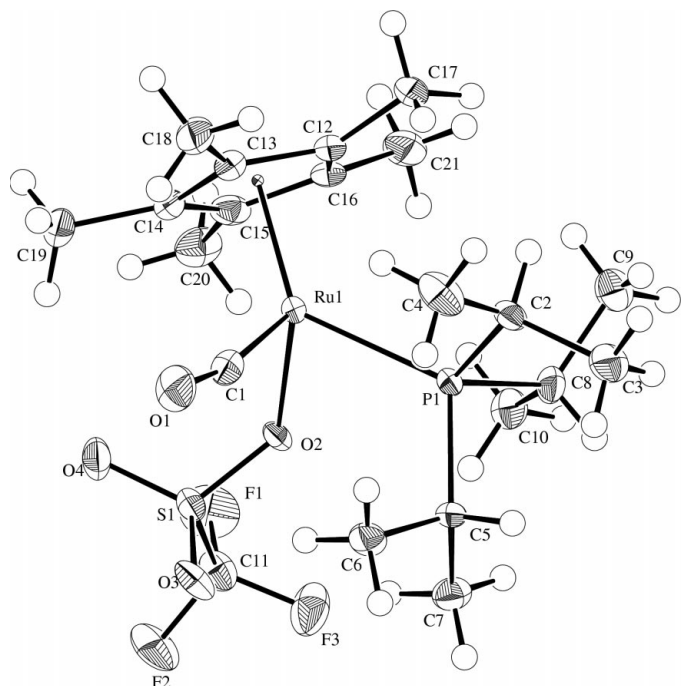


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids.

Experimental

The title compound was synthesized from $\text{RuCp}^*(\text{P}^i\text{Pr})_3(\text{O}_2\text{CF}_3)$, stirred under an atmosphere of CO. Crystals were grown by diffusion of pentane into a deuterobenzene solution.

Crystal data

$[\text{Ru}(\text{CF}_3\text{O}_3\text{S})(\text{C}_{10}\text{H}_{15})(\text{C}_9\text{H}_{21}\text{P})(\text{CO})]$
 $M_r = 573.60$
 Monoclinic, $P2_1/c$
 $a = 16.056$ (1) Å
 $b = 9.1258$ (5) Å
 $c = 17.005$ (1) Å
 $\beta = 98.796$ (1)°
 $V = 2462.3$ (3) Å³
 $Z = 4$

$D_x = 1.547$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4749 reflections
 $\theta = 1.7$ – 24.7 °
 $\mu = 0.83$ mm⁻¹
 $T = 128$ (2) K
 Tablet, orange
 $0.27 \times 0.13 \times 0.08$ mm

Data collection

SMART CCD diffractometer
 ω scans
 Absorption correction: empirical (Blessing, 1995)
 $T_{\min} = 0.81$, $T_{\max} = 0.91$
 10 547 measured reflections
 4036 independent reflections

3238 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$
 $\theta_{\text{max}} = 24.7$ °
 $h = -6 \rightarrow 18$
 $k = -10 \rightarrow 10$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.089$
 $S = 1.08$
 4035 reflections

280 parameters
 H-atom parameters constrained
 $(\Delta/\sigma)_{\text{max}} = -0.042$
 $\Delta\rho_{\text{max}} = 0.70$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.71$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ru1—C1	1.862 (4)	Ru1—C14	2.227 (4)
Ru1—O2	2.195 (3)	Ru1—C15	2.261 (4)
Ru1—C13	2.198 (4)	Ru1—C16	2.284 (4)
Ru1—C12	2.218 (4)	Ru1—P1	2.3952 (11)
C1—Ru1—O2	96.76 (14)	O2—Ru1—P1	87.12 (7)
C1—Ru1—P1	91.82 (13)		

H atoms were refined as riding, with C—H distances of 0.98 or 1.00 Å.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1995); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1992); software used to prepare material for publication: *SHELXL97*.

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