metal-organic papers

(η⁵-Pentamethylcyclopentadienyl)(η⁵-5,6,7-trihydro-4,8-dimethyl-s-indacenyl)ruthenium(II)

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Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.080
Data-to-parameter ratio = 19.2

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

In the title compound, [Ru($C_{10}H_{15}$)($C_{14}H_{15}$)], the coordinating rings make an angle of 1.0 (1)° with each other and are rotated away from the ideal eclipsed conformation by *ca* 13°.

Comment

The title compound, (1), is isostructural with its iron analogue (see preceding paper for introductory material; Jones *et al.*, 2002).

(1)
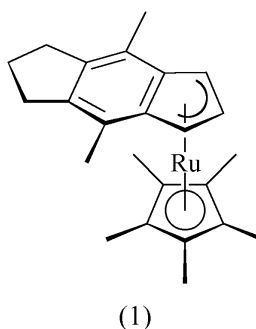
The molecule of compound (1) is shown in Fig. 1. The distances from the Ru atom to the centroids, *Cg*, of the coordinating ring planes, which are essentially parallel [interplanar angle 1.0 (1)°], are 1.840 Å to the C1–C8*A* ring and 1.788 Å to the C11–C15 ring, with an angle at Ru of 178.7°. The coordination of the indacenyl ring shows a very slight distortion towards η^3, with Ru—C3*A* and Ru—C8*A* being the longest distances. The rings are rotated appreciably away from the ideal eclipsed conformation, with torsion angles such as C12—*Cg*1—*Cg*2—C3 = −13°.

Experimental

The title compound was prepared in 80% yield by first metallating 1,5,6,7-tetrahydro-4,8-dimethyl-s-indacene with methyllithium in anhydrous tetrahydrofuran, and then quenching the anions thus formed with equivalent amounts of the complex [(C₅Me₅)Ru(CH₃CN)₃]PF₆. It was characterized by spectroscopic and analytical data (Hartig, 1991), and by the crystal structure described here. Single crystals were obtained by slow cooling of a warm saturated solution in hexane.

Crystal data

[Ru($C_{10}H_{15}$)($C_{14}H_{15}$)]	$Z = 2$
$M_r = 419.55$	$D_x = 1.423$ Mg m⁻³
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.651$ (2) Å	Cell parameters from 50
$b = 9.949$ (2) Å	reflections
$c = 11.575$ (3) Å	$\theta = 10–11.5°$
$\alpha = 82.82$ (2)°	$\mu = 0.80$ mm⁻¹
$\beta = 82.09$ (2)°	$T = 183$ (2) K
$\gamma = 88.88$ (2)°	Tablet, yellow
$V = 979.0$ (4) Å³	0.5 × 0.4 × 0.2 mm

Data collection

Nicolet *R*3 diffractometer	$\theta_{max} = 27.5°$
ω scans	$h = -11 \rightarrow 4$
Absorption correction: none	$k = -12 \rightarrow 12$
4763 measured reflections	$l = -14 \rightarrow 15$
4477 independent reflections	3 standard reflections
4120 reflections with $I > 2\sigma(I)$	every 147 reflections
$R_{int} = 0.011$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$+ 0.7774P]$
$wR(F^2) = 0.080$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.008$
4477 reflections	$\Delta\rho_{max} = 3.08$ e Å^{-3}
233 parameters	$\Delta\rho_{min} = -0.44$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å).

Ru—C12	2.162 (2)	Ru—C1	2.187 (2)
Ru—C13	2.164 (2)	Ru—C3	2.189 (2)
Ru—C15	2.167 (2)	Ru—C2	2.192 (2)
Ru—C11	2.167 (2)	Ru—C8*A*	2.234 (2)
Ru—C14	2.172 (2)	Ru—C3*A*	2.235 (2)

Methyl H atoms were identified in difference syntheses, idealized and then refined, using rigid methyl groups allowed to rotate but not tip. Other H atoms were included, using a riding model, with fixed C—H bond lengths (aromatic 0.95, methyl and methylene 0.99 Å); U_{iso}(H) values were fixed at $1.2U_{eq}$ of the parent atom. The methyl group at C20 converged slowly. The only significant difference peak (>0.5 e Å^{-3}) is one of 3.1 e Å^{-3}. It does not lie near the Ru atom (the nearest atom is H20B). Because its coordinates are related to those of the Ru atom by *ca* 1/2−*x*, 1/2−*y*, 1/2−*z*, we surmise that a small unidentified twinning component is probably responsible for this peak.

Data collection: *P*3 (Nicolet, 1987); cell refinement: *P*3; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.
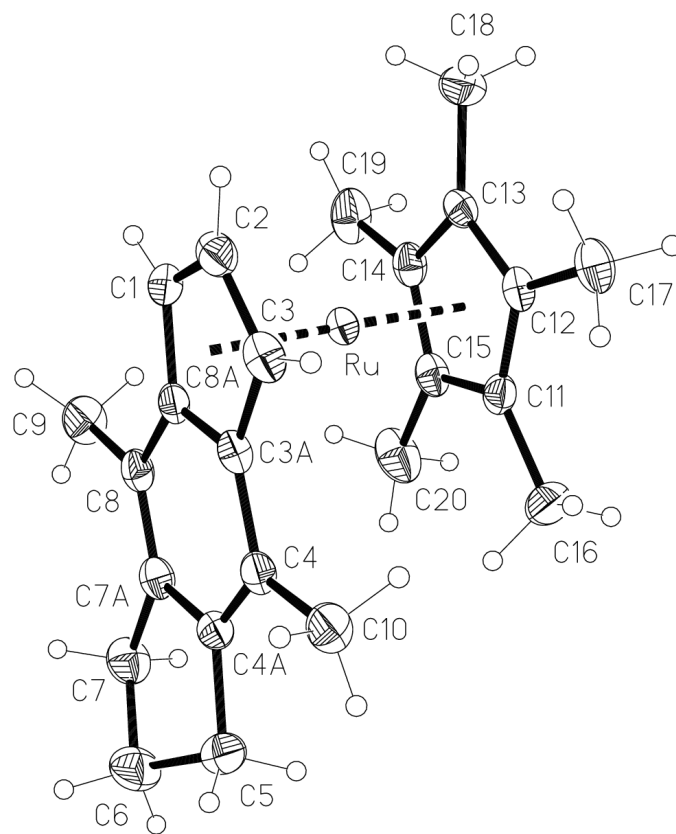
Figure 1
The molecule of compound (1) in the crystal. Ellipsoids represent 50% probability levels. H-atom radii are arbitrary.

References

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