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Bis(η^5-pentamethylcyclopentadienyl)[bis-η^5-*syn*-[2.2](4,7)indenophanyl]diruthenium(II)

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Key indicators

Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.004 Å
R factor = 0.028
wR factor = 0.071
Data-to-parameter ratio = 14.7

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

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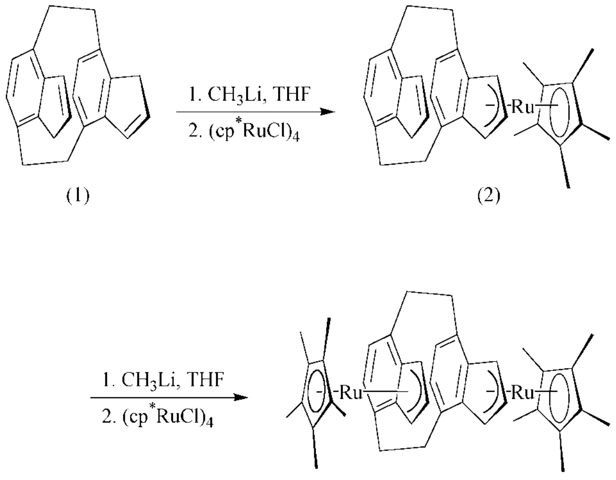
In the title compound, [$Ru_2(C_{10}H_{15})_2(C_{22}H_{18})$], the four five-membered rings are approximately eclipsed. The distances from these rings to the corresponding Ru atoms are 1.795 (1) and 1.798 (1) Å for the Cp*, and 1.835 (1) and 1.842 (1) Å for the indenyl five-membered rings.

Comment

Having prepared and studied various metal complexes of *anti*-[2.2]indenophanes (Hopf & Dannheim, 1988), we have now prepared ruthenium complexes of *syn*-[2.2]indenophane, (1). Whereas with the former ligands the metal atoms are oriented in a stepwise fashion, in (1) they were expected to be oriented above each other (eclipsed). Here we present the structure of the doubly capped ruthenium complex, (3).



(1) (2)

(3)

The molecule of (3) is shown in Fig. 1. In the following discussion, the rings are defined as ring 1 for atoms C19–C23, ring 2 for C3*A*/C4/C5/C6/C6*A*, ring 3 for C12*A*/C13/C14/C15/C14*A*, and ring 4 for C19′–C23′; the corresponding centroids are denoted Cent1, Cent2, *etc*.

The Ru atoms lie 1.795 (1), 1.835 (1), 1.842 (1) and 1.798 (1) Å from ring planes 1–4, respectively. The general form of the molecule is as expected, in that the four five-membered rings are essentially eclipsed, but there are some deviations from the idealized form (Fig. 2). The sequence Cent1—Ru1—Cent2···Cent3—Ru2—Cent4 is significantly bent at Cent3 (171°), and rings 2 and 3 of the central indenophane ligand are splayed away from each other [interplanar angle 10.92 (4)°]. The distance Cent2···Cent3 is 3.30 Å.

The six-membered rings display the usual flattened boat conformation, in which the bridgehead atoms C3, C7, C12 and C16 lie 0.151 (4), 0.142 (4), 0.149 (4) and 0.151 (4) Å, respec-

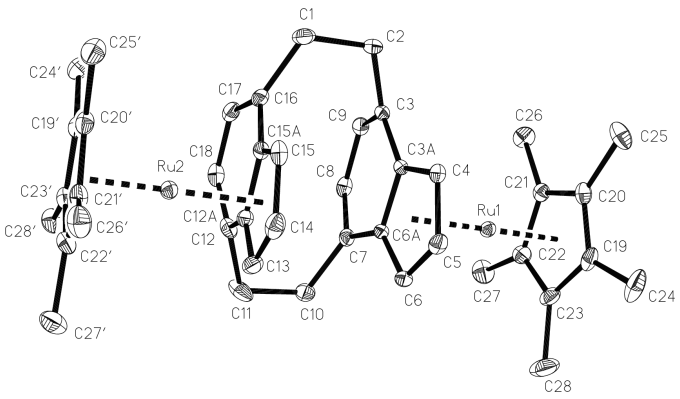


Figure 1
The molecule of compound (3) in the crystal. Displacement ellipsoids are shown at the 30% probability level. H atoms have been omitted for clarity.

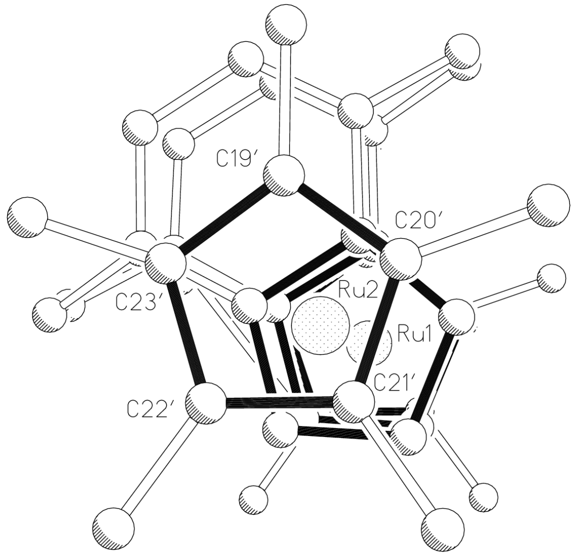


Figure 2
Alternative view of compound (3), perpendicular to the plane of ring C19–C23. Radii are arbitrary.

tively, out of the plane of the four other atoms; the interplanar angle between the four-atom planes is 1.90 (2)°, and the distance between the corresponding centroids is 3.08 Å.

Experimental

Hydrocarbon (1) was monometalated using methyl lithium in THF at 233 K. The mixture was stirred for 30 min at 273 K and the pentamethylcyclopentadienyl ruthenium chloride tetramer was then added. Reaction was complete after 1 h at room temperature. The solvent was removed *in vacuo* and the remaining solid recrystallized from hexane; the monoruthenium complex, (2), was isolated in 92% yield as yellow needles and characterized by spectroscopic and analytical data. Repetition of the reaction sequence furnished the bis-ruthenium complex, (3), as yellow prisms in 91% yield. Spectroscopic data are again in full accord with the structural assignment. For full details, see Hartig (1991).

Crystal data

$[Ru_2(C_{10}H_{15})_2(C_{22}H_{18})]$
$M_r = 754.94$
Monoclinic, $P2_1/c$
$a = 8.647$ (2) Å
$b = 13.604$ (4) Å
$c = 28.959$ (8) Å
$\beta = 92.41$ (2)°
$V = 3403.5$ (16) Å^3
$Z = 4$

$D_x = 1.473$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–$11.5°$
$\mu = 0.92$ mm^{-1}
$T = 178$ (2) K
Prism, yellow
$0.70 \times 0.40 \times 0.15$ mm

Data collection

Nicolet *R*3 diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.66$, $T_{\max} = 0.84$
11146 measured reflections
5984 independent reflections
5079 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.020$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 10$
$k = -16 \rightarrow 10$
$l = -34 \rightarrow 34$
3 standard reflections every 147 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.071$
$S = 1.05$
5984 reflections
407 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 1.6478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.61$ e Å^{-3}

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 C—H = 0.95 Å, and methyl and methylene C—H = 0.99 Å); $U_{\rm iso}$(H) values were fixed at $1.2U_{\rm eq}$ of the parent atom.

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.

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