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Bis(η^5-1,2,4-triisopropylcyclopentadienyl)-ruthenium

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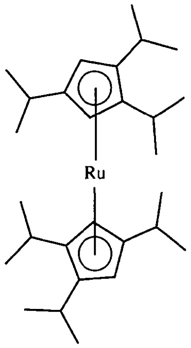
Abstract

The title compound, $[Ru\{(C_3H_7)_3C_5H_2\}_2]$, displays a rigorously parallel sandwich geometry, with the Ru atom residing on a crystallographically imposed inversion center. A resolvable disorder is present in one isopropyl substituent. The average Ru—C(ring) distance is 2.182 (9) Å, similar to that of other structurally characterized ruthenocenes.

Comment

We have been examining the effects of bulky isopropylated cyclopentadienyl rings on the structural, electrochemical and magnetic properties of first-row transition metal metallocenes (Burkey, Hays, Duderstadt & Hanusa, 1997). As an extension of this research to larger second-row transition metals, we have prepared the ruthenocene $[(C_3H_7)_3C_5H_2]_2Ru$, (1), and determined its crystal structure.



(1)

The structure of (1) contains a crystallographically imposed inversion center at the Ru atom. Consequently, the cyclopentadienyl rings are rigorously parallel, making (1) isostructural with $[(C_3H_7)_3C_5H_2]_2M$ (M = Fe, Co; Burkey *et al.*, 1997). The average Ru—C bond length is 2.182 (9) Å, with individual bond lengths ranging from 2.168 (4) to 2.191 (4) Å. Despite the steric bulk of the rings, the average Ru—C bond length in (1) is comparable with that in $(C_5H_5)_2Ru$ (average 2.191 Å; Seiler & Dunitz, 1980) and $(C_5Me_5)_2Ru$ [average 2.17 (1) Å; Albers *et al.*, 1986], but is less than that in the more sterically encumbered $[(C_6H_5)_4C_5H]_2Ru$ (average 2.202 Å; Hoobler *et al.*, 1991). The range of Ru—C bond distances in (1) (Δ = 0.023 Å) is intermediate between $(C_5H_5)_2Ru$ (range 2.181–2.188 Å, Δ = 0.007 Å) and $[(C_6H_5)_4C_5H]_2Ru$ (range 2.181–2.214 Å, Δ = 0.033 Å), an illustration of the progressively greater steric effect of the triisopropyl- and tetraphenylcyclopentadienyl rings. There is only a slight displacement of the methine C atoms of the isopropyl substituents in (1) from the cyclopentadienyl ring plane (average 0.11 Å); this amount is similar to that in $[(C_3H_7)_3C_5H_2]_2Fe$ (0.12 Å) and slightly less than in $[(C_6H_5)_4C_5H]_2Ru$ (0.14 Å).

A distinctive feature of (1) not found in analogous first-row metallocenes is the resolvable disorder in the isopropyl substituent on C(2). Assignment of occupancy factors 0.60 and 0.40 to C(8), C(9) and C(8*A*), C(9*A*), respectively, led to satisfactory refinement of the methyl C atoms. In the iron and cobalt structures, this isopropyl group displays larger displacement parameters than the other two, but the relatively close interring distances and long intermolecular contacts {*i.e.* 3.32 and 3.84 (1) Å, respectively, in $[(C_3H_7)_3C_5H_2]_2Fe$} probably prevents any observable disorder. In (1), the interplanar distance (3.63 Å) is comparable with the closest intermolecular contact [3.62 (1) Å between C(8) and C(11)(x, $-y$, z)]. The greater freedom for rotation combined with the close packing evidently encourages the C(2) isopropyl group in (1) to adopt more than one conformation.

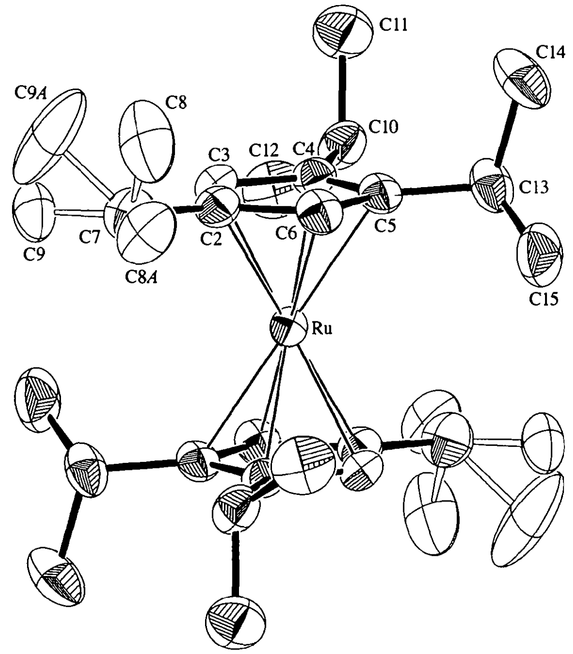


Fig. 1. *ORTEP*II (Johnson, 1976) view of the non-H atoms of (1), with displacement ellipsoids drawn at the 30% probability level.

Experimental

Using a route similar to that for $[(C_6H_5)_4C_5H]_2Ru$ (Hoobler *et al.*, 1991), (1) was prepared in 44% yield in dimethoxyethane (m.p. 421–423 K). ^1H NMR (300 MHz, C_6D_6): δ 4.28 (*s*, 4H, ring CH), 2.46 (septet, J = 6.8 Hz, 6H, $CHMe_2$), 1.24 (*d*, J = 6.8 Hz, 12H, CH_3), 1.19 (*d*, J = 6.7 Hz, 12H, CH_3), 1.15 (*d*, J = 6.7 Hz, 12H, CH_3). ^{13}C NMR (75.5 MHz, C_6D_6): δ 105.2 (ring $CCHMe_2$), 102.2 (ring $CCHMe_2$), 67.4 (ring CH), 28.1 ($CHMe_2$), 26.3 ($CHMe_2$), 25.8 (CH_3), 24.5 (CH_3), 24.3 (CH_3). Analysis for $C_{28}H_{46}Ru$: calculated for C 69.52, H 9.58, Ru 20.89%; found C 68.71, H 9.31, Ru 20.13%. X-ray quality crystals were grown by slow evaporation from toluene solution.

Crystal data

$[Ru(C_{14}H_{23})_2]$
M_r = 483.74
Triclinic
$P\bar{1}$
a = 9.156 (3) Å
b = 9.509 (2) Å
c = 8.701 (2) Å
α = 99.41 (2)°
β = 116.03 (2)°
γ = 73.95 (2)°
V = 653.4 (3) Å^3
Z = 1
D_x = 1.229 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 24 reflections
θ = 54.8–65.3°
μ = 5.009 mm^{-1}
T = 293 (2) K
Prism
0.30 × 0.30 × 0.25 mm
Pale yellow

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.180, T_{max} = 0.286
2095 measured reflections
1952 independent reflections

1944 reflections with $I > 3\sigma(I)$
R_{int} = 0.04
θ_{max} = 60.09°
$h = -3 \rightarrow 10$
$k = -9 \rightarrow 10$
$l = -9 \rightarrow 9$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F
R = 0.0350
wR = 0.0462
S = 2.046
1944 reflections
151 parameters
H atoms not refined
$w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{max}$ = 0.0297
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected bond lengths (Å)*

Ru—C(2)	2.189 (4)	Ru—C(5)	2.191 (4)
Ru—C(3)	2.168 (4)	Ru—C(6)	2.178 (4)
Ru—C(4)	2.184 (4)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1021). Services for accessing these data are described at the back of the journal.

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