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Jörg Saßmannshausen *et al.*

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Tribenzyl $\{\eta^5$ -[2-(*p*-tolyl)prop-2-yl]-cyclopentadienyl}zirconium

Jörg Saßmannshausen, Jörg Rust and Richard Goddard*

Max-Planck-Institut für Kohlenforschung, Kaiser-Wilhelm-Platz 1, D-45470 Mülheim an der Ruhr, Germany

Correspondence e-mail: goddard@mpi-muelheim.mpg.de

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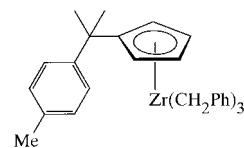
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The title compound, $[\text{Zr}(\text{C}_7\text{H}_7)_3(\text{C}_{15}\text{H}_{17})]$, (I), crystallizes from light petroleum with two independent molecules in the asymmetric unit. Whereas in the parent molecule, $\text{Zr}(\eta^5\text{-C}_5\text{H}_5)(\text{CH}_2\text{Ph})_3$, all three Zr—CH₂Ph angles are equal, in (I), they differ significantly. In spite of their different environments, both independent molecules in (I) exhibit a small, an expected, and a large Zr—CH₂Ph angle. The angles are similar to those of the closely related tribenzyl $\{\eta^5$ -(benzyl-dimethylsilyl)cyclopentadienyl}zirconium complex. The smallest Zr—CH₂Ph angle and the consequently relatively short Zr···C_{ipso} distance are indicative of η^2 -bonding of the benzyl group.

Comment

The title compound, (I), crystallizes from light petroleum with two independent molecules in the asymmetric unit. Whereas in the parent molecule, $\text{Zr}(\eta^5\text{-C}_5\text{H}_5)(\text{CH}_2\text{Ph})_3$ (Scholz *et al.*, 1993), all three Zr—CH₂Ph angles are equal to 94.5 (5)°, in (I), they differ significantly. In spite of their different environments, both independent molecules in (I) exhibit a small [86.4 (2) and 85.5 (2)°], an expected [103.9 (2) and 103.1 (2)°], and a large [119.9 (2) and 122.4 (2)°] Zr—CH₂Ph angle. The angles are similar to those of the closely related $[\eta^5$ -(benzyl-dimethylsilyl)cyclopentadienyl]tribenzylzirconium complex where comparable angles are 86.5 (10), 102.0 (13) and 122.4 (11)° (Ciruelo *et al.*, 1997). The smallest Zr—CH₂Ph angle and the consequently relatively short Zr—C_{ipso} bond [2.643 (2) and 2.632 (2) Å] are indicative of η^2 -bonding of the benzyl group. The methylene H atoms in the benzylic groups were refined isotropically in order to ascertain whether any of them were agostic (Brookhart & Green, 1983). None of the Zr—C—H angles is significantly less than 90°, indicating that the Zr···C_{ipso} interaction is favoured over an agostic interaction.



(I)

Experimental

The title compound was prepared according to the method described by Saßmannshausen (2000). Crystals suitable for X-ray diffraction were grown by slow cooling a light petroleum solution of the compound to 233 K.

Crystal data

$[\text{Zr}(\text{C}_7\text{H}_7)_3(\text{C}_{15}\text{H}_{17})]$
 $M_r = 561.88$
 Monoclinic, $P2_1/c$
 $a = 25.0892$ (18) Å
 $b = 11.0572$ (8) Å
 $c = 22.0033$ (15) Å
 $\beta = 111.395$ (2)°
 $V = 5683.4$ (7) Å³
 $Z = 8$

$D_x = 1.313$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 14107 reflections
 $\theta = 1.86$ – 28.00 °
 $\mu = 0.409$ mm⁻¹
 $T = 100$ K
 Prism, yellow
 0.53 × 0.42 × 0.11 mm

Data collection

Siemens SMART diffractometer
 CCD ω scans
 Absorption correction: ψ scan
 (XPREP; Siemens, 1995)
 $T_{\min} = 0.844$, $T_{\max} = 0.956$
 49 130 measured reflections
 13 434 independent reflections

9028 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.128$
 $\theta_{\max} = 28$ °
 $h = -21 \rightarrow 33$
 $k = -14 \rightarrow 14$
 $l = -28 \rightarrow 26$
 Intensity decay: none

Refinement

Refinement on F^2
 $R(F) = 0.044$
 $wR(F^2) = 0.100$
 $S = 1.016$
 13 434 reflections
 715 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0403P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.77$ e Å⁻³
 $\Delta\rho_{\min} = -1.15$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C24'—Zr1'	2.632 (2)	C31—Zr1	2.643 (2)
C17—C16—Zr1	119.93 (16)	C24'—C23'—Zr1'	85.48 (16)
C17'—C16'—Zr1'	103.08 (16)	C31—C30—Zr1	86.43 (16)
C24—C23—Zr1	103.91 (17)	C31'—C30'—Zr1'	122.36 (17)

The methylene H atoms of the benzylic groups were refined isotropically and the remaining H atoms were included in the refinement using a riding model. The maximum residual electron density is 1.08 Å⁻³ from Zr1.

Data collection: SMART (Siemens, 1995); cell refinement: SMART; data reduction: SAINT (Siemens, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997).

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