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[{$Me_4C_2(C_5H_4)_2$}Zr(BH_4)$_2$], a bis(tetrahydroborate) complex of a bridged zirconocene

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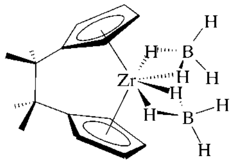
Bis(tetrahydroborato)[1,1,2,2-tetramethyl-1,2-ethylenebis(η^5-cyclopentadienyl)]zirconium, (I), was synthesized by the reaction of the zirconocene dichloride with lithium tetrahydroborate. Crystals suitable for X-ray structure analysis were obtained by recrystallization from toluene. The molecule adopts an appproximate C_{2v} symmetry. Both tetrahydroborate ligands are η^2-coordinated and tilted by 18–19° out of the equatorial plane; the angle B1—Zr1—B2 is 104.7°. The cyclopentadienyl rings show a normal η^5-coordinaton, with a centroid–Zr–centroid angle of 124.3°.



(I)

Experimental

$C_2Me_4(C_5H_4)_2ZrCl_2$ (1.1 g, 2.9 mmol) and lithium tetrahydroborate (0.25 g, 11.5 mmol) were stirred in tetrahydrofuran at room temperature for 12 h (James *et al.*, 1967). The solvent was removed and the residue extracted three times with 70 ml toluene. The yellow solution was concentrated until precipitation started. After warming and addition of 20 ml pentane, the solution was cooled to 193 K. Filtration and washing with pentane yielded 570 mg of a colourless powder (59% yield). NMR (250 MHz, C_6D_6): 6.18 (*t*, 4H), 5.59 (*t*, 4 H), 0.76 (*s*, 12H), 1.0 [*bq*, *J*(BH) = 60 Hz, 8H]. Elemental analysis: calculated C 57.7, H 8.7%; found C 57.4, H 8.3% All operations were carried out under exclusion of air and moisture. Solvents were dried by standard techniques.

Crystal data

[Zr(BH_4)$_2$($C_{16}H_{20}$)]
M_r = 333.22
Triclinic, $P\bar{1}$
a = 7.699 (2) Å
b = 9.341 (2) Å
c = 12.934 (3) Å
α = 77.77 (3)°
β = 78.52 (3)°
γ = 69.09 (3)°
V = 841.4 (3) Å^3
Z = 2
D_x = 1.315 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14.0–15.9°
μ = 0.638 mm^{-1}
T = 153 K
Block cut from a needle, colourless
0.4 × 0.4 × 0.4 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*MolEN*; Fair 1990)
$T_{\min}$ = 0.696, $T_{\max}$ = 0.775
5012 measured reflections
3653 independent reflections
3360 reflections with$I > 2\sigma(I)$
$R_{\rm int}$ = 0.014
$\theta_{\max}$ = 26.94°
$h = 0 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.058
S = 1.037
3653 reflections
284 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 0.3682P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.81 e Å^{-3}
$\Delta\rho_{\min}$ = −0.69 e Å^{-3}

Data collection: *MolEN* (Fair, 1990); cell refinement: *MolEN*; data reduction: *MolEN*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

References

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