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Crystallographic report

(η^5-C_5Me_5)Fe(CO)$_2$(BOCH$_2$CH$_2$CH$_2$O): an organoiron complex containing the (trimethyleneglycolato)boryl ligand

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The structure of the pentamethylcyclopentadienyliron boryl complex (η^5-C_5Me_5)Fe(CO)$_2$B(tmg) (tmg = trimethylene glycolato, –OCH$_2$CH$_2$CH$_2$O–), reveals a planar three-coordinate boryl ligand and an Fe–B distance significantly in excess of that reported for related catecholboryl complexes. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; iron; boryl; boron

COMMENT

The title complex, (η^5-C_5Me_5)Fe(CO)$_2$B(tmg) (**I**; Fig. 1), represents only the second reported example of a complex containing the B(tmg) ligand.[1] Of interest are (i) the near orthogonal alignment of BO_2 and centroid(η^5-C_5Me_5)–Fe–B planes (∠centroid–Fe–B–O3 = 92.9(4)°) presumably adopted on steric grounds and (ii) the Fe–B distance (2.024(4) Å), which is essentially identical to that found in the related dinuclear complex (η^5-C_5H_5)Fe(CO)$_2$BO$_2$C$_5$H$_8$O$_2$BFe(CO)$_2$(η^5-C_5H_5) (2.030(5) Å), but significantly greater than that found in the corresponding catecholboryl complex (η^5-C_5Me_5)Fe(CO)$_2$BO$_2$C$_6$H$_4$ (1.980(2) Å).[2,3] This observation is consistent with weaker π acceptor properties for alkoxoboryl ligands compared with aryloxo analogues, as reported previously.[2]

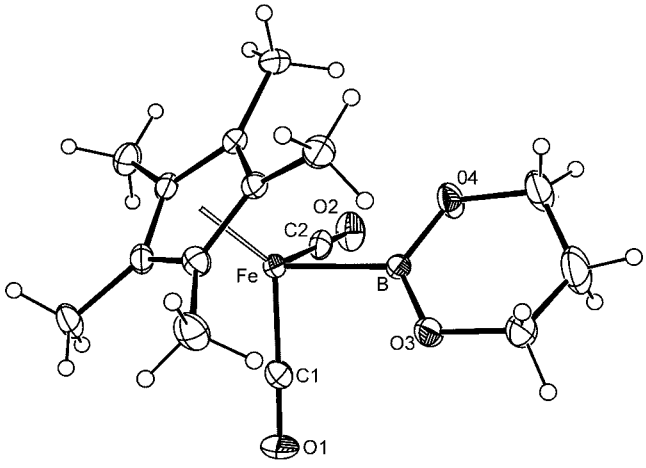

Figure 1. Molecular structure of **I**. Key geometric parameters: Fe–B 2.024(4), Fe–C1 1.737(4), Fe–C2 1.742(4), centroid(η^5-C_5Me_5)–Fe 1.728(5), B–O3 1.357(5), B–O4 1.364(5) Å; C1–Fe–C2 95.4(2), Fe–B–O3 119.9(3), Fe–B–O4 118.9(3), O3–B–O4 121.1(3)°.

EXPERIMENTAL

I was prepared from Na[(η^5-C_5Me_5)Fe(CO)$_2$] and *B*-chloro(trimethylene glycolato)borane by a method analogous to that reported previously,[2,4] and isolated as pale-yellow crystals from a hexane solution cooled to −50 °C. Salient spectroscopic data: ^{1}H NMR (C_6D_6), δ 1.37 (m, 2H, OCH$_2$C*H*$_2$CH$_2$O), 1.68 (s, 15H, η^5-C_5Me_5), 3.64 (m, 4H, OC*H*$_2$CH$_2$C*H*$_2$O). ^{11}B NMR (96 MHz, C_6D_6), δ 47.0. IR (KBr disk, cm^{-1}) ν(CO) 1971 s, 1910 s. Mass spectrometry (electron impact): M^+ = 332 (15%). Exact mass: calc. 332.0877, meas. 332.0882. Data were collected on a Nonius Kappa CCD diffractometer for a pale-yellow crystal 0.10 × 0.30 × 0.43 mm^3. $C_{15}H_{21}BFeO_4$, M = 331.98,

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monoclinic, $P2_1/n$, $a = 13.1516(3)$, $b = 8.9576(2)$, $c = 14.0546(4)$ Å, $\beta = 105.164(1)°$, $V = 1598.08(7)$ Å^3, $Z = 4$, $T = 180(2)$ K, 3649 unique data (θ_{max} 27.5°), 2526 data $I \geq 2\sigma(I)$, $R = 0.059$, $wR = 0.148$, $\rho_{max} = 1.08\ e^-$ Å^{-3}. Programs used: SHELXS-97, SHELXL-97, X-seed and ORTEP. CCDC deposition number: 241273.

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