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The crystal structure of butyrylferrocene, $C_{14}H_{16}FeO$

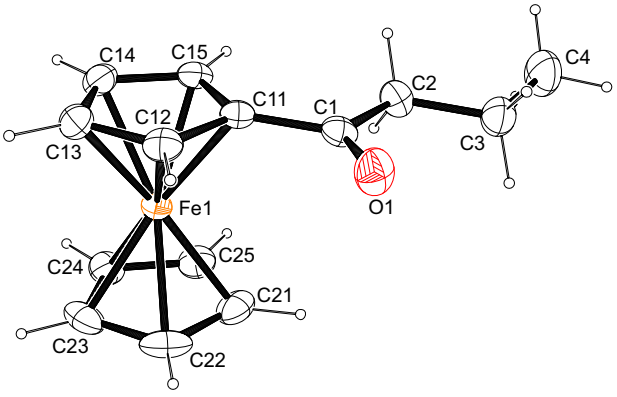

Table 1: Data collection and handling.

Crystal:	Yellow rods
Size:	0.38 × 0.17 × 0.14 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.27 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.3°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11166, 2910, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2579
$N(param)_{refined}$:	147
Programs:	Bruker [1, 2], SHELX [3], WinGX/ORTEP [4], Mercury [5], PLATON [6]

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Abstract

$C_{14}H_{16}FeO$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.6954(3) Å, b = 10.0307(6) Å, c = 20.4422(14) Å, V = 1167.84(12) Å^3, Z = 4, $R_{gt}(F)$ = 0.0267, $wR_{ref}(F^2)$ = 0.0559, T = 200 K.

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The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The compound was obtained commercially (Strem Chemicals).

Experimental details

H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms, C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C). The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the *SHELX* program suite [3]), with U(H) set to 1.5U_{eq}(C).

Comment

Ferrocenes have been a busy playing ground for theoretical and synthetic chemists alike since the 1940s and 1950s which may be due to the intriguing bonding situation that opened up the field for organometallic chemistry as an exciting new field of study [7, 8]. Especially its behaviour typical for aromatic compounds [9] as well as its successful structure elucidation [10] gave rise to the synthesis and characterization of novel metallocene compounds [11, 12]. The archaetype – ferrocene itself – and its derivatives have been the subject of structure-relationship studies for several decades, and ample information is apparent in the literature (e.g. the asymmetric formyl [13] and acetyl [14] derivatives).

The structure shows the presence of the expected ferrocenyl "sandwich" with one of the two cyclopentadienyl moieties featuring a butyryl side chain. The iron atom is displaced by 1.6452(13) Å and 1.6484(14) Å from the

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.42289 (6)	0.62657 (3)	0.33982 (2)	0.02002 (9)
O1	0.0151 (3)	0.4883 (2)	0.46802 (10)	0.0367 (5)
C1	0.2117 (5)	0.5357 (3)	0.47104 (12)	0.0239 (6)
C2	0.3968 (5)	0.4830 (3)	0.51682 (13)	0.0279 (6)
H2A	0.445923	0.556098	0.546357	0.033*
H2B	0.535585	0.456717	0.490679	0.033*
C3	0.3220 (5)	0.3651 (3)	0.55817 (13)	0.0347 (6)
H3A	0.292790	0.287160	0.529562	0.042*
H3B	0.173859	0.386636	0.581195	0.042*
C4	0.5112 (6)	0.3304 (3)	0.60807 (15)	0.0449 (8)
H4A	0.658626	0.310904	0.585295	0.067*
H4B	0.462207	0.252024	0.633245	0.067*
H4C	0.534307	0.405949	0.637773	0.067*
C11	0.2785 (4)	0.6510 (2)	0.42986 (11)	0.0220 (5)
C12	0.1339 (5)	0.7116 (3)	0.38137 (13)	0.0258 (6)
H12	−0.033118	0.688035	0.371685	0.031*
C13	0.2677 (5)	0.8105 (3)	0.34919 (13)	0.0285 (6)
H13	0.212071	0.868165	0.312406	0.034*
C14	0.4961 (5)	0.8118 (3)	0.37691 (13)	0.0291 (6)
H14	0.628660	0.870789	0.363070	0.035*
C15	0.5055 (5)	0.7141 (3)	0.42670 (12)	0.0249 (6)
H15	0.644446	0.692852	0.454728	0.030*
C21	0.4573 (5)	0.4266 (3)	0.32356 (14)	0.0333 (7)
H21	0.409075	0.353999	0.354260	0.040*
C22	0.3145 (5)	0.4850 (3)	0.27458 (15)	0.0370 (7)
H22	0.147909	0.460806	0.264717	0.044*
C23	0.4482 (5)	0.5840 (3)	0.24228 (13)	0.0340 (7)
H23	0.392898	0.641961	0.205558	0.041*
C24	0.6728 (5)	0.5870 (3)	0.27126 (14)	0.0303 (6)
H24	0.804696	0.647477	0.258571	0.036*
C25	0.6796 (5)	0.4895 (3)	0.32177 (14)	0.0301 (6)
H25	0.816594	0.468995	0.350650	0.036*

respective center of gravity of the two cyclopentadiene rings with the shorter distance established towards the functionalized carbocycle. The stacking of the "sandwich" is close to linearity with the pertaining C_g–Fe–C_g angle measured at 178.94(7). The rings adopt an almost ecliptic conformation as becomes apparent by the planes defined by the iron atom, the *ipso* carbon atom as well as the carbonyl-type carbon atom on the one hand and the iron atom as well as the atoms of the CH group below the side chain intersecting at an angle of only 1.472(13). The butyryl side chain is vastly co-planar with its carrier carbocycle with the least-squares plane defined by the pertaining ten non-hydrogen atoms showing the terminal carbon atom deviating most with a value of only 0.059(3) Å. The C=O bond length was found at 1.218(3) Å which is in good agreement with other keto compounds whose metrical parameters have been deposited with the Cambridge Structural Database [15].

In the crystal, no strong interatomic interactions whose range falls below the sum of van-der-Waals radii of the atoms participating in them are observed. The only notable interaction in this regard is a potential C–H···π contact supported by a hydrogen atom of the functionalized cyclopentadiene moiety as donor and the aromatic system of the non-functionalized carbocycle in the neighbouring molecule as acceptor. While the two aromatic systems within a molecule of the title compound are found at a distance of only 3.2933(18) Å, π-Stacking between two separate molecules is not a prominent feature with the shortest pertaining distance between two centers of gravity measured at 4.6663(18) Å only.

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