Crystal structure of methyl ferrocenemonocarboxylate, [Fe(C₅H₅)(C₅H₄C₂O₂H₃)]

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Abstract

C₁₂H₁₂FeO₂, monoclinic, *P*12₁/*c*1 (No. 14), *a* = 16.064(1) Å, *b* = 5.9250(3) Å, *c* = 23.105(2) Å, β = 108.79(1)°, *V* = 2081.9 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.029, *wR*_{ref}(*F*²) = 0.053, *T* = 200 K.

Source of material

The title complex [1] was synthesized from ferrocenecarboxylic acid and thionylchloride in methanol and extracted with *n*-pentane. Crystals were obtained by sublimation in vacuum.

Discussion

Ferrocenecarboxylic acid esters are well studied compounds [2] and can be used, e.g., for the synthesis of ferrocenyl substituted 1,3-diketones [3–5]. The crystallographic data of ethyl and phenyl ferrocenecarboxylate were reported [6]. In the title molecule the ferrocene unit deviates from the eclipsed conformation by δ = 4.6°. The C6—C11 bond distance between the Cp ring and the carboxyl group is with 1.464(3) Å shorter than a typical C—C single bond which corresponds to somewhat longer C6—C7 and C6—C10 bond lengths (1.43 Å) in the Cp ring compared to C7—C8, C8—C9 and C9—C10 (1.42 Å). All the bond lengths (C6—C11, C—C_{ring}, C=O, C—O) are quite similar to that of ferrocenecarboxylic acid [7] and of ferrocene-1,1'-dicarboxylic acid [8]. The ester unit is coplanar with the C₅H₄ ring.

Table 1. Data collection and handling.

Crystal:	orange rod, size 0.06 × 0.12 × 0.43 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	14.22 cm ⁻¹
Diffractometer, scan mode:	Stoe IPDS, φ
2θ _{max} :	51.84°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	10733, 3857
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2825
<i>N</i> (<i>param</i>) _{refined} :	367
Programs:	SIR97 [9], SHELXL-97 [10], ORTEP-III [11]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.814(2)	0.224(5)	0.740(1)	0.045(8)
H(2)	4e	0.857(2)	-0.144(5)	0.791(1)	0.031(7)
H(3)	4e	0.841(2)	-0.126(5)	0.895(1)	0.036(8)
H(4)	4e	0.792(2)	0.257(5)	0.912(1)	0.043(8)
H(5)	4e	0.776(2)	0.479(5)	0.817(1)	0.036(8)
H(7)	4e	0.646(2)	-0.297(5)	0.730(1)	0.032(7)
H(8)	4e	0.644(2)	-0.312(5)	0.837(1)	0.043(8)
H(9)	4e	0.593(2)	0.069(5)	0.861(1)	0.035(7)
H(10)	4e	0.570(2)	0.326(5)	0.771(1)	0.022(7)
H(12A)	4e	0.586(2)	0.601(7)	0.604(2)	0.06(1)
H(12B)	4e	0.534(2)	0.380(6)	0.569(2)	0.058(9)
H(12C)	4e	0.637(2)	0.410(5)	0.592(1)	0.026(7)
H(13)	4e	0.799(2)	-0.463(7)	0.670(2)	0.08(1)
H(14)	4e	0.969(2)	-0.466(7)	0.700(2)	0.07(1)
H(15)	4e	1.015(2)	-0.079(5)	0.679(1)	0.046(8)
H(16)	4e	0.885(2)	0.156(6)	0.638(1)	0.040(9)
H(17)	4e	0.757(2)	-0.084(6)	0.635(1)	0.051(9)
H(19)	4e	0.802(2)	-0.626(5)	0.532(1)	0.027(7)
H(20)	4e	0.964(2)	-0.615(5)	0.567(1)	0.028(7)
H(21)	4e	1.010(2)	-0.223(5)	0.545(1)	0.029(7)
H(22)	4e	0.872(1)	0.007(5)	0.503(1)	0.022(7)
H(24A)	4e	0.608(2)	0.241(6)	0.467(1)	0.051(9)
H(24B)	4e	0.579(2)	0.030(6)	0.498(2)	0.07(1)
H(24C)	4e	0.575(2)	0.019(5)	0.428(1)	0.039(8)

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Fe(1)	4e	0.71287(2)	0.06234(6)	0.80378(1)	0.0185(2)	0.0228(2)	0.0197(2)	-0.0030(2)	0.0044(1)	-0.0035(2)
Fe(2)	4e	0.88075(2)	-0.26311(6)	0.59627(1)	0.0227(2)	0.0238(2)	0.0147(1)	0.0044(1)	0.0052(1)	-0.0005(2)
O(1)	4e	0.6054(1)	0.0045(3)	0.62554(7)	0.0384(9)	0.026(1)	0.0287(9)	0.0041(7)	0.0057(7)	-0.0056(8)
O(2)	4e	0.5940(1)	0.3519(3)	0.66031(8)	0.048(1)	0.022(1)	0.030(1)	0.0035(8)	0.0097(8)	-0.0012(8)
O(3)	4e	0.6482(1)	-0.3544(3)	0.48333(9)	0.0296(9)	0.026(1)	0.052(1)	-0.0068(8)	0.0053(8)	0.0005(9)
O(4)	4e	0.6963(1)	-0.0006(3)	0.47972(7)	0.0247(8)	0.023(1)	0.0378(9)	0.0002(7)	0.0028(7)	0.0083(8)
C(1)	4e	0.8153(2)	0.1959(5)	0.7813(1)	0.023(1)	0.042(2)	0.029(1)	-0.010(1)	0.007(1)	0.005(1)
C(2)	4e	0.8391(1)	-0.0176(5)	0.8086(1)	0.018(1)	0.034(2)	0.038(1)	0.001(1)	0.007(1)	-0.003(1)
C(3)	4e	0.8312(2)	-0.0091(5)	0.8677(1)	0.023(1)	0.048(2)	0.027(1)	-0.001(1)	-0.001(1)	0.009(1)
C(4)	4e	0.8032(2)	0.2094(6)	0.8770(1)	0.028(1)	0.061(2)	0.026(1)	-0.013(1)	0.004(1)	-0.020(2)
C(5)	4e	0.7930(2)	0.3366(6)	0.8234(1)	0.026(1)	0.022(2)	0.058(2)	-0.006(1)	0.004(1)	-0.006(1)
C(6)	4e	0.6049(1)	0.0508(5)	0.7273(1)	0.016(1)	0.023(2)	0.028(1)	-0.002(1)	0.0034(9)	-0.006(1)
C(7)	4e	0.6289(1)	-0.1726(5)	0.7504(1)	0.021(1)	0.024(2)	0.030(1)	-0.004(1)	0.003(1)	-0.005(1)
C(8)	4e	0.6252(2)	-0.1801(5)	0.8109(1)	0.025(1)	0.034(2)	0.034(1)	-0.011(1)	0.010(1)	-0.000(1)
C(9)	4e	0.5989(2)	0.0355(5)	0.8252(1)	0.026(1)	0.042(2)	0.035(1)	-0.007(1)	0.016(1)	-0.009(1)
C(10)	4e	0.5867(1)	0.1792(5)	0.7742(1)	0.017(1)	0.028(2)	0.039(1)	0.002(1)	0.007(1)	-0.008(1)
C(11)	4e	0.6022(1)	0.1275(4)	0.6665(1)	0.014(1)	0.025(2)	0.030(1)	0.0008(9)	0.0007(9)	-0.004(1)
C(12)	4e	0.5863(2)	0.4397(7)	0.6003(1)	0.052(2)	0.029(2)	0.037(2)	0.001(2)	0.010(1)	0.002(1)
C(13)	4e	0.8397(2)	-0.3375(6)	0.6687(1)	0.075(2)	0.053(2)	0.028(2)	-0.008(2)	0.029(2)	0.001(1)
C(14)	4e	0.9313(2)	-0.3381(7)	0.6868(1)	0.076(2)	0.055(2)	0.016(1)	0.030(2)	0.006(1)	0.005(1)
C(15)	4e	0.9596(2)	-0.1258(7)	0.6755(1)	0.032(2)	0.075(3)	0.025(1)	-0.001(2)	0.003(1)	-0.024(2)
C(16)	4e	0.8855(2)	0.0117(6)	0.6501(1)	0.065(2)	0.027(2)	0.026(1)	0.006(1)	0.017(1)	-0.008(1)
C(17)	4e	0.8114(2)	-0.1225(6)	0.6462(1)	0.032(2)	0.071(3)	0.026(1)	0.014(2)	0.012(1)	-0.012(1)
C(18)	4e	0.8016(1)	-0.2875(4)	0.50824(9)	0.028(1)	0.021(2)	0.015(1)	0.001(1)	0.0049(9)	-0.0024(9)
C(19)	4e	0.8340(2)	-0.5052(5)	0.5308(1)	0.035(1)	0.022(2)	0.023(1)	0.000(1)	0.009(1)	-0.005(1)
C(20)	4e	0.9269(2)	-0.4963(5)	0.5487(1)	0.035(1)	0.032(2)	0.026(1)	0.011(1)	0.011(1)	-0.005(1)
C(21)	4e	0.9522(2)	-0.2776(5)	0.5376(1)	0.028(1)	0.040(2)	0.025(1)	0.001(1)	0.013(1)	-0.004(1)
C(22)	4e	0.8754(2)	-0.1448(5)	0.5127(1)	0.031(1)	0.027(2)	0.016(1)	-0.001(1)	0.0096(9)	0.002(1)
C(23)	4e	0.7078(2)	-0.2233(5)	0.4894(1)	0.030(1)	0.026(2)	0.018(1)	-0.002(1)	0.0018(9)	0.001(1)
C(24)	4e	0.6065(2)	0.0773(6)	0.4653(2)	0.027(1)	0.035(2)	0.051(2)	0.005(1)	0.002(1)	0.002(2)

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