Crystal structure of *rac*-2-(methylthio)propanoylferrocene, $C_{14}H_{16}FeOS$

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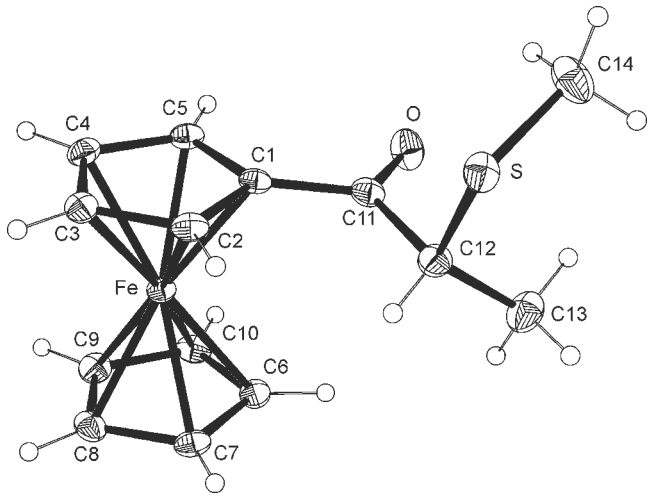

Abstract

$C_{14}H_{16}FeOS$, monoclinic, $P12_1/n1$ (no. 14), a = 12.6757(9) Å, b = 5.7043(5) Å, c = 18.062(1) Å, β = 103.151(7)°, V = 1271.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.032, $wR_{ref}(F^2)$ = 0.083, T = 173 K.

Source of material

The title compound was synthesized from 2-(methylthio)-propanoyl chloride, which was prepared *in situ* from 2-(methylthio)propanoic acid and PCl_3, and ferrocene in the presence of $AlCl_3$ by a one-pot procedure in CH_2Cl_2 as described previously [1]. Suitable crystals were obtained by slow evaporation of a solution in CH_2Cl_2/heptane at room temperature.

Experimental details

Positions of hydrogen atoms bonded to carbon were generated in idealized geometries using a riding model with U_{iso}(H) = 1.2 U_{eq}(C) for CH and U_{iso}(H) = 1.5 U_{eq}(C) for CH_3. The fractional coordinates of the H atom linked to C12 were identified from difference Fourier maps and refined freely. The corresponding isotropic temperature factor U_{iso}(H) was constrained as described above.

Discussion

Replacement of a phenyl ring by a ferrocene unit often leads to products with new desirable properties [2-4]. Thus, ferrocene analogues of sulfur analogues [5] of phenacyl amines with amphetamine-type activity were prepared. Although this approach may seem far-fetched, it is yet worth trying.

The ferrocene in the title crystal structure adopts an almost eclipsed conformation with an average torsion angle of 1.58°. The planes of the cyclopentadienide (Cp) rings are tilted by 3.24°. The atoms of the carbonyl group deviate from the plane of the adjacent Cp ring by only 0.18 Å (C) and 0.14 Å (O), whereas the sulfur atom is fully rotated out of the plane. The molecules are linked by weak C7–H7···O^{i} interactions into infinite chains in the direction of the [010] (symmetry code i: x,–1+y,z). The H···acceptor distance is 2.57 Å, donor···acceptor distance 3.414(3) Å, donor–H···acceptor angle 148°, respectively.

Table 1. Data collection and handling.

Crystal:	red-brown needle, size 0.04 × 0.04 × 0.24 mm
Wavelength:	Cu K_α radiation (1.5418 Å)
μ:	108.65 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Ruby, Gemini ultra, ω
$2\theta_{max}$:	134.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11551, 2266
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1948
$N(param)_{refined}$:	159
Programs:	SHELXS-97 [6], SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.6791	0.0062	0.3092	0.032
H(12)	4*e*	0.524(2)	0.115(4)	0.351(2)	0.037
H(5)	4*e*	0.7934	0.6642	0.3811	0.032
H(3)	4*e*	0.8830	0.0069	0.3265	0.033
H(6)	4*e*	0.6377	0.2454	0.5150	0.035
H(7)	4*e*	0.6987	–0.1573	0.4822	0.035
H(4)	4*e*	0.9527	0.4107	0.3713	0.035
H(10)	4*e*	0.8042	0.4968	0.5601	0.035
H(9)	4*e*	0.9690	0.2495	0.5567	0.038
H(8)	4*e*	0.9041	–0.1549	0.5087	0.037
H(13A)	4*e*	0.4493	0.3039	0.4420	0.066
H(13B)	4*e*	0.3563	0.1925	0.3764	0.066
H(13C)	4*e*	0.3823	0.4669	0.3755	0.066
H(14A)	4*e*	0.3112	0.5440	0.2473	0.075
H(14B)	4*e*	0.3463	0.5638	0.1678	0.075
H(14C)	4*e*	0.4251	0.6597	0.2439	0.075

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	4*e*	0.79594(3)	0.22019(6)	0.43561(2)	0.0248(2)	0.0239(2)	0.0155(2)	–0.0015(2)	0.0042(1)	0.0002(1)
S	4*e*	0.43618(5)	0.2546(1)	0.23845(3)	0.0306(3)	0.0420(4)	0.0249(3)	–0.0012(3)	0.0026(3)	–0.0061(2)
O	4*e*	0.5697(2)	0.6465(3)	0.3684(1)	0.035(1)	0.034(1)	0.048(1)	0.0024(8)	0.0038(9)	–0.0088(9)
C(1)	4*e*	0.6938(2)	0.3651(4)	0.3445(1)	0.029(1)	0.023(1)	0.016(1)	–0.002(1)	0.0019(9)	0.0023(9)
C(2)	4*e*	0.7260(2)	0.1354(4)	0.3254(1)	0.036(1)	0.027(1)	0.015(1)	–0.004(1)	0.005(1)	–0.0017(9)
C(12)	4*e*	0.4958(2)	0.2634(5)	0.3410(1)	0.030(1)	0.038(2)	0.024(1)	–0.002(1)	0.003(1)	0.002(1)
C(5)	4*e*	0.7901(2)	0.5044(4)	0.3658(1)	0.034(1)	0.026(1)	0.020(1)	–0.005(1)	0.006(1)	0.0015(9)
C(3)	4*e*	0.8400(2)	0.1359(5)	0.3352(1)	0.032(1)	0.032(1)	0.018(1)	0.001(1)	0.007(1)	–0.001(1)
C(6)	4*e*	0.7105(2)	0.1972(5)	0.5191(1)	0.032(1)	0.036(2)	0.021(1)	0.003(1)	0.009(1)	0.003(1)
C(7)	4*e*	0.7445(2)	–0.0285(5)	0.5009(1)	0.039(1)	0.032(1)	0.019(1)	–0.006(1)	0.008(1)	0.003(1)
C(4)	4*e*	0.8791(2)	0.3627(5)	0.3602(1)	0.028(1)	0.039(2)	0.022(1)	–0.005(1)	0.008(1)	0.005(1)
C(10)	4*e*	0.8036(2)	0.3376(5)	0.5445(1)	0.044(2)	0.027(1)	0.018(1)	–0.000(1)	0.006(1)	–0.002(1)
C(9)	4*e*	0.8957(2)	0.1995(5)	0.5425(1)	0.031(1)	0.043(2)	0.019(1)	–0.003(1)	0.001(1)	0.002(1)
C(11)	4*e*	0.5865(2)	0.4419(4)	0.3529(1)	0.032(1)	0.031(2)	0.018(1)	0.000(1)	0.001(1)	0.002(1)
C(8)	4*e*	0.8594(2)	–0.0268(5)	0.5156(1)	0.037(1)	0.036(2)	0.021(1)	0.010(1)	0.007(1)	0.005(1)
C(13)	4*e*	0.4137(2)	0.3108(6)	0.3878(2)	0.036(2)	0.068(2)	0.029(1)	–0.006(1)	0.010(1)	–0.001(1)
C(14)	4*e*	0.3724(3)	0.5378(6)	0.2226(2)	0.052(2)	0.064(2)	0.030(1)	0.020(2)	–0.000(1)	–0.002(1)

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