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Crystal structure of 1-ferrocenyl-(3-methyl-1-phenyl-aziridin-2-yl)-methanone, $(C_5H_5)Fe(C_{16}H_{16}NO)$

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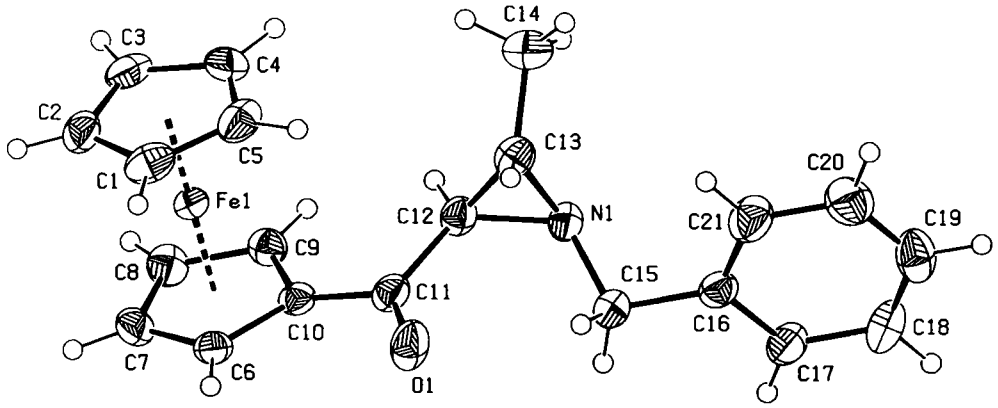

Abstract

$C_{21}H_{21}FeNO$, monoclinic, $P12_1/a1$ (no. 14), $a = 7.329(7)$ Å, $b = 17.343(3)$ Å, $c = 13.618(5)$ Å, $\beta = 95.02°$, $V = 1724.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.044$, $wR_{ref}(F^2) = 0.111$, $T = 293$ K.

Source of material

The title compound was prepared by Gabriel-Cromwell method [1]. Bromine (1.5 eq, 1 M in CCl_4) was added over 30 min to a stirred solution of crotonoylferrocene (1.0 eq) in pentane at room temperature [2]. After the addition the reaction was judged to complete by TLC. The pure dibromo compound was obtained in 75 % yield by standard work-up and purification by chromatography on silica gel. The compound (167 mg, 0.50 mmol) was stirred with Et_3Ny (0.75 mmol, 105 µL) and benzylamine (1.00 mmol, 105 µL) in $CHCl_3$ for 10 h at room temperature. The crude product was applied to a flash column for purification. From the column, aziridine (162 mg, 0.450 mmol) was isolated in 90 % yield as a 1:1 mixture of *cis*- and *trans*-isomers [1]. The second one was recrystallized from chloroform.

Discussion

The lengths of the C—N bonds, d(C12—N1) = 1.482(3) Å, d(C13—N1) = 1.446(4) Å, which constituted the aziridine ring in the title structure are in agreement with corresponding values of another similar structure related to ferrocenyl aziridines [3]. One molecule is connected with two other molecules via one C–H···O and two C–H···N hydrogen bonds. The C–H···X bond lengths and angles range from 2.638 Å to 2.654 Å and from 142° to 170°, respectively. There is one intramolecular C–H···O interaction with d(H15B···O1) = 2.376 Å and ∠C15–H15B···O1 = 115.2°.

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Table 1. Data collection and handling.

Crystal:	reddish prism, size 0.20 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	8.81 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7-S, $\omega/2\theta$
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5390, 5028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2574
$N(param)_{refined}$:	217
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	−0.4818	0.2575	0.6147	0.059
H(2)	4e	−0.6456	0.3517	0.4988	0.054
H(3)	4e	−0.8455	0.4335	0.5960	0.056
H(4)	4e	−0.8074	0.3880	0.7713	0.062
H(5)	4e	−0.5838	0.2803	0.7828	0.061
H(6)	4e	−0.1444	0.3734	0.6810	0.044
H(7)	4e	−0.2752	0.4608	0.5468	0.050
H(8)	4e	−0.4865	0.5544	0.6176	0.051
H(9)	4e	−0.4904	0.5261	0.7978	0.044
H(12)	4e	−0.4897	0.4554	0.9260	0.044
H(13)	4e	−0.4352	0.2997	0.9960	0.049
H(14A)	4e	−0.7145	0.3104	1.0624	0.094
H(14B)	4e	−0.7608	0.3558	0.9638	0.094
H(14C)	4e	−0.7062	0.4007	1.0618	0.094
H(15A)	4e	−0.0862	0.4192	1.0823	0.056
H(15B)	4e	−0.1444	0.3334	1.0590	0.056
H(17)	4e	0.0116	0.4427	1.2472	0.056
H(18)	4e	0.0093	0.4199	1.4149	0.067
H(19)	4e	−0.1786	0.3265	1.4700	0.066
H(20)	4e	−0.3688	0.2581	1.3595	0.072
H(21)	4e	−0.3663	0.2803	1.1927	0.066

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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(1)	4*e*	–0.50389(5)	0.40460(2)	0.67549(3)	0.0254(2)	0.0342(2)	0.0313(2)	–0.0017(2)	–0.0001(1)	0.0001(2)
O(1)	4*e*	–0.1573(3)	0.3436(1)	0.8845(1)	0.057(1)	0.073(2)	0.038(1)	0.033(1)	–0.004(1)	–0.004(1)
N(1)	4*e*	–0.3613(3)	0.4066(1)	1.0540(2)	0.038(1)	0.043(1)	0.031(1)	0.001(1)	0.0003(9)	–0.002(1)
C(1)	4*e*	–0.5655(4)	0.2945(2)	0.6313(2)	0.041(2)	0.039(2)	0.064(2)	–0.007(1)	–0.006(2)	–0.008(2)
C(2)	4*e*	–0.6575(4)	0.3477(2)	0.5661(2)	0.046(2)	0.051(2)	0.038(2)	–0.012(2)	–0.005(1)	–0.002(1)
C(3)	4*e*	–0.7701(4)	0.3935(2)	0.6205(2)	0.028(1)	0.054(2)	0.057(2)	–0.002(1)	–0.008(1)	0.005(2)
C(4)	4*e*	–0.7481(4)	0.3678(2)	0.7194(2)	0.035(2)	0.073(2)	0.048(2)	–0.017(2)	0.010(1)	–0.003(2)
C(5)	4*e*	–0.6228(4)	0.3075(2)	0.7259(2)	0.049(2)	0.048(2)	0.052(2)	–0.019(2)	–0.013(2)	0.017(2)
C(6)	4*e*	–0.2265(3)	0.4136(2)	0.6880(2)	0.028(1)	0.043(2)	0.039(1)	–0.003(1)	0.004(1)	–0.008(1)
C(7)	4*e*	–0.3009(4)	0.4629(2)	0.6124(2)	0.042(2)	0.048(2)	0.036(1)	–0.012(1)	0.009(1)	0.001(1)
C(8)	4*e*	–0.4204(4)	0.5158(2)	0.6522(2)	0.048(2)	0.036(2)	0.043(2)	–0.006(1)	0.004(1)	0.004(1)
C(9)	4*e*	–0.4222(4)	0.5000(2)	0.7539(2)	0.037(2)	0.036(2)	0.038(1)	–0.001(1)	0.005(1)	–0.004(1)
C(10)	4*e*	–0.3005(3)	0.4366(2)	0.7776(2)	0.028(1)	0.034(2)	0.035(1)	–0.004(1)	0.001(1)	–0.007(1)
C(11)	4*e*	–0.2740(4)	0.3935(2)	0.8711(2)	0.034(1)	0.043(2)	0.036(1)	0.005(1)	–0.003(1)	–0.010(1)
C(12)	4*e*	–0.4046(4)	0.4135(2)	0.9460(2)	0.036(1)	0.040(2)	0.034(1)	0.009(1)	–0.001(1)	–0.003(1)
C(13)	4*e*	–0.4851(4)	0.3515(2)	1.0041(2)	0.043(2)	0.038(2)	0.042(2)	0.001(1)	–0.001(1)	–0.003(1)
C(14)	4*e*	–0.6850(4)	0.3549(2)	1.0250(3)	0.041(2)	0.082(3)	0.065(2)	–0.009(2)	0.007(2)	0.005(2)
C(15)	4*e*	–0.1766(4)	0.3799(2)	1.0930(2)	0.036(2)	0.069(2)	0.034(1)	0.003(1)	–0.002(1)	–0.001(1)
C(16)	4*e*	–0.1786(4)	0.3641(2)	1.2017(2)	0.032(1)	0.041(2)	0.039(2)	0.004(1)	–0.001(1)	–0.001(1)
C(17)	4*e*	–0.0659(4)	0.4052(2)	1.2693(2)	0.045(2)	0.049(2)	0.043(2)	–0.008(2)	–0.007(1)	0.005(2)
C(18)	4*e*	–0.0666(5)	0.3915(2)	1.3701(2)	0.066(2)	0.058(2)	0.040(2)	0.002(2)	–0.016(2)	–0.002(2)
C(19)	4*e*	–0.1788(5)	0.3362(2)	1.4028(2)	0.072(2)	0.053(2)	0.040(2)	0.022(2)	0.006(2)	0.009(2)
C(20)	4*e*	–0.2911(5)	0.2953(2)	1.3371(2)	0.063(2)	0.058(2)	0.061(2)	–0.006(2)	0.011(2)	0.012(2)
C(21)	4*e*	–0.2900(5)	0.3090(2)	1.2369(2)	0.057(2)	0.055(2)	0.050(2)	–0.014(2)	–0.006(2)	–0.002(2)

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