

Crystal structure of 1-ferrocenyl-(3-methyl-1-phenyl-aziridin-2-yl)-methanone, (C₅H₅)Fe(C₁₆H₁₆NO)

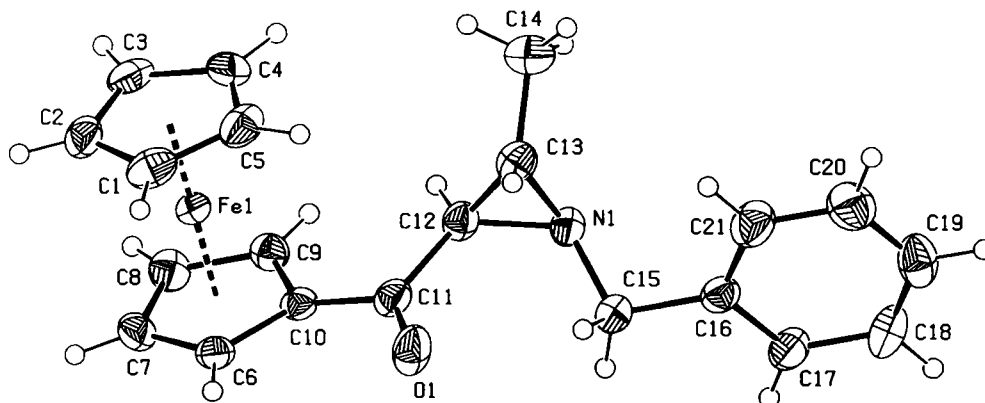
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Abstract

C₂₁H₂₁FeNO, monoclinic, *P*2₁/*a*1 (no. 14), *a* = 7.329(7) Å, *b* = 17.343(3) Å, *c* = 13.618(5) Å, β = 95.02°, *V* = 1724.3 Å³, *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 CCl₄) was added over 30 min to a stirred solution of crotonylferrocene (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 Et₃Ny (0.75 mmol, 105 µL) and benzylamine (1.00 mmol, 105 µL) in CHCl₃ 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 $\angle$ C15—H15B...O1 = 115.2°.

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
Diffraction, scan mode:	Rigaku AFC7-S, $\omega/2\theta$
$2\theta_{\max}$:	60°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5390, 5028
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2574
<i>N</i> (<i>param</i>) _{refined} :	217
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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	-0.4818	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 Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Fe(1)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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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