Crystal structure of 2-ethyl-1-ferrocenylsulfonyl-1*H*-imidazo[4,5-*b*]-pyridine, $C_{18}H_{17}FeN_3O_2S$

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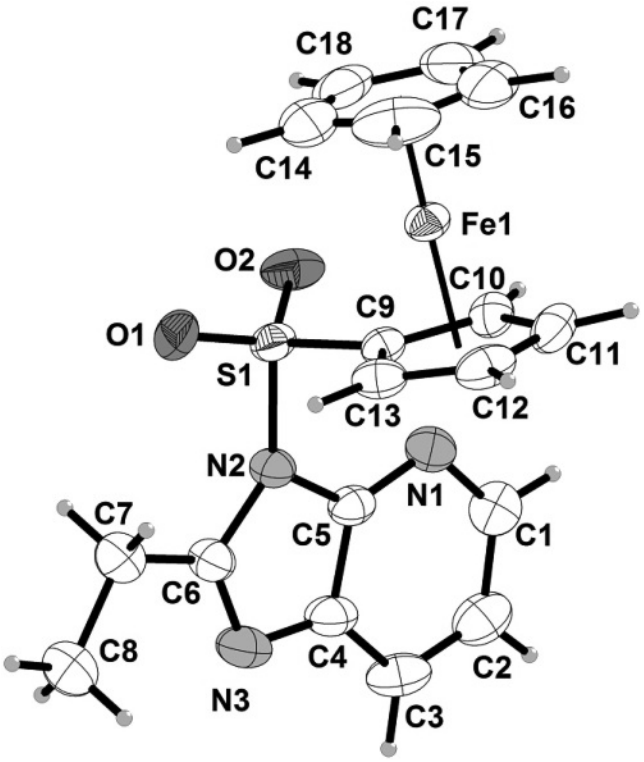

Abstract

$C_{18}H_{17}FeN_3O_2S$, triclinic, $P\bar{1}$ (no. 2), a = 7.8088(2) Å, b = 10.5763(3) Å, c = 11.9799(3) Å, α = 109.277(1)°, β = 107.856(1)°, γ = 97.832(1)°, V = 857.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.0280, $wR_{ref}(F^2)$ = 0.0755, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.20×0.30×0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.18 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII CCD, φ and ω
$2\theta_{max}$:	56.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11513, 4101
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3842
$N(param)_{refined}$:	228
Programs:	SHELX [4]

Source of material

All reagents and solvents were purchased from commercial sources and used as received. The title compound is obtained from a series of reactions involving the synthesis of 2-ethyl-1-ferrocenylsulfonyl-1*H*-imidazo[4,5-*b*]pyridine from 2-ethyl-1*H*-imidazo[4,5-*b*]pyridine and ferrocenylsulfonyl chloride. A solution of 2-ethyl-1*H*-imidazo[4,5-*b*]pyridine (0.4 g, 2.7 mmol) in acetonitrile (18 mL) was added to a mixture of ferrocenylsulfonylchloride (0.72 g, 2.5 mmol) and anhydrous sodium carbonate (0.26 g, 2.5 mmol) with constant stirring. The mixture was heated to reflux temperatures for 48 h. The acetonitrile was evaporated under vacuum to leave a brown solid. The crude product was purified by column chromatography (SiO_2, ethyl acetate:petroleum ether, v/v = 3:7) to give the title compound (0.16 g, 16% yield) as yellow crystals, m.p.: 452-453K.

Discussion

Imidazo[4,5-*b*]pyridine and their derivatives possess diverse pharmacological properties, including anticancer, antiviral, and other important biological activities [1, 2]. The asymmetric unit of the title compound contains one 2-ethyl-1-ferrocenylsulfonyl-1*H*-imidazo[4,5-*b*]pyridine molecule. There is a imidazo[4,5-*b*] pyridine moiety and a sandwich ferrocene unit in the structure. The two rings in the imidazo[4,5-*b*]pyridine part of the title molecule (C1-C6, N1-N3) are coplanar, and the dihedral angles between the imidazo[4,5-*b*]pyridine plane and the substituted cyclopentadienyl plane (C9-C13) is 102.73(7)°. There are some differences in bond lengts and bond angles between the substituted and unsubstituted cyclopentadienyl ring [3]. The bond lengths of the substituted cyclopentadienyl ligand range from 1.428(2)-1.427(2) Å, whereas in the unsubstituted one they range from 1.388(4)-1.369(4) Å.

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.5756	0.5085	0.6368	0.056
H(2)	2*i*	0.6608	0.4310	0.7971	0.066
H(3)	2*i*	0.5365	0.2013	0.7604	0.069
H(7A)	2*i*	0.0757	–0.1907	0.2419	0.065
H(7B)	2*i*	–0.0837	–0.1396	0.2824	0.065
H(8A)	2*i*	–0.0569	–0.3422	0.3158	0.098
H(8B)	2*i*	–0.0044	–0.2195	0.4479	0.098
H(8C)	2*i*	0.1531	–0.2720	0.4058	0.098
H(10)	2*i*	0.0984	0.4309	0.3548	0.043
H(11)	2*i*	–0.1964	0.4890	0.3871	0.052
H(12)	2*i*	–0.4392	0.2635	0.3173	0.055
H(13)	2*i*	–0.3018	0.0588	0.2395	0.046
H(14)	2*i*	–0.3788	0.0458	–0.0569	0.080
H(15)	2*i*	–0.6018	0.1861	0.0015	0.095
H(16)	2*i*	–0.4298	0.4460	0.0874	0.096
H(17)	2*i*	–0.1056	0.4577	0.0781	0.078
H(18)	2*i*	–0.0789	0.2115	–0.0100	0.073

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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)	2*i*	–0.23926(3)	0.27477(2)	0.16646(2)	0.0308(1)	0.0396(1)	0.0237(1)	0.01022(8)	0.00566(8)	0.01277(8)
S(1)	2*i*	0.09067(5)	0.12106(4)	0.23724(3)	0.0367(2)	0.0428(2)	0.0262(2)	0.0158(1)	0.0123(1)	0.0159(1)
O(1)	2*i*	–0.0162(2)	–0.0139(1)	0.1452(1)	0.0677(8)	0.0441(6)	0.0304(6)	0.0197(6)	0.0111(5)	0.0075(5)
O(2)	2*i*	0.2262(2)	0.2021(1)	0.2145(1)	0.0402(6)	0.0781(8)	0.0461(6)	0.0223(6)	0.0224(5)	0.0387(6)
N(1)	2*i*	0.4034(2)	0.3357(1)	0.4994(1)	0.0373(6)	0.0453(7)	0.0409(7)	0.0126(5)	0.0118(5)	0.0209(6)
N(2)	2*i*	0.2074(2)	0.0984(1)	0.3709(1)	0.0394(6)	0.0406(6)	0.0320(6)	0.0160(5)	0.0116(5)	0.0191(5)
N(3)	2*i*	0.2752(2)	0.0113(2)	0.5222(1)	0.0607(9)	0.0530(8)	0.0438(8)	0.0173(7)	0.0159(7)	0.0297(7)
C(1)	2*i*	0.5239(2)	0.4161(2)	0.6188(2)	0.0367(8)	0.0512(9)	0.0467(9)	0.0109(7)	0.0124(7)	0.0162(7)
C(2)	2*i*	0.5760(3)	0.3700(2)	0.7167(2)	0.0463(9)	0.069(1)	0.0366(9)	0.0111(8)	0.0066(7)	0.0152(8)
C(3)	2*i*	0.5031(3)	0.2340(2)	0.6958(2)	0.059(1)	0.078(1)	0.0358(9)	0.018(1)	0.0086(8)	0.0305(9)
C(4)	2*i*	0.3777(2)	0.1488(2)	0.5738(2)	0.0467(8)	0.0545(9)	0.0358(8)	0.0192(7)	0.0141(7)	0.0255(7)
C(5)	2*i*	0.3376(2)	0.2063(2)	0.4829(1)	0.0327(7)	0.0461(8)	0.0307(7)	0.0166(6)	0.0119(5)	0.0188(6)
C(6)	2*i*	0.1741(2)	–0.0162(2)	0.4045(2)	0.0494(9)	0.0417(8)	0.0422(8)	0.0190(7)	0.0208(7)	0.0229(7)
C(7)	2*i*	0.0396(3)	–0.1530(2)	0.3142(2)	0.069(1)	0.0432(9)	0.050(1)	0.0131(8)	0.0215(9)	0.0206(8)
C(8)	2*i*	0.0322(4)	–0.2561(2)	0.3767(2)	0.088(2)	0.048(1)	0.069(1)	0.016(1)	0.035(1)	0.029(1)
C(9)	2*i*	–0.0556(2)	0.2162(1)	0.2834(1)	0.0266(6)	0.0365(7)	0.0231(6)	0.0077(5)	0.0063(5)	0.0119(5)
C(10)	2*i*	–0.0176(2)	0.3641(2)	0.3354(1)	0.0321(7)	0.0374(7)	0.0279(7)	0.0065(5)	0.0036(5)	0.0085(5)
C(11)	2*i*	–0.1789(2)	0.3956(2)	0.3550(1)	0.0438(8)	0.0519(9)	0.0280(7)	0.0210(7)	0.0096(6)	0.0087(6)
C(12)	2*i*	–0.3132(2)	0.2707(2)	0.3163(2)	0.0342(7)	0.076(1)	0.0370(8)	0.0210(7)	0.0169(6)	0.0273(8)
C(13)	2*i*	–0.2395(2)	0.1577(2)	0.2715(1)	0.0299(7)	0.0509(8)	0.0333(7)	0.0042(6)	0.0080(6)	0.0216(6)
C(14)	2*i*	–0.3521(4)	0.1472(2)	–0.0221(2)	0.088(2)	0.060(1)	0.0278(8)	0.013(1)	–0.0023(9)	0.0138(8)
C(15)	2*i*	–0.4743(3)	0.2233(3)	0.0103(2)	0.045(1)	0.136(2)	0.049(1)	0.010(1)	–0.0051(9)	0.053(1)
C(16)	2*i*	–0.3800(4)	0.3666(3)	0.0576(2)	0.119(2)	0.104(2)	0.049(1)	0.081(2)	0.033(1)	0.047(1)
C(17)	2*i*	–0.2025(4)	0.3729(2)	0.0522(2)	0.089(2)	0.065(1)	0.044(1)	0.010(1)	0.020(1)	0.0326(9)
C(18)	2*i*	–0.1887(3)	0.2379(2)	0.0033(2)	0.075(1)	0.087(1)	0.0316(8)	0.036(1)	0.0221(9)	0.0279(9)

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