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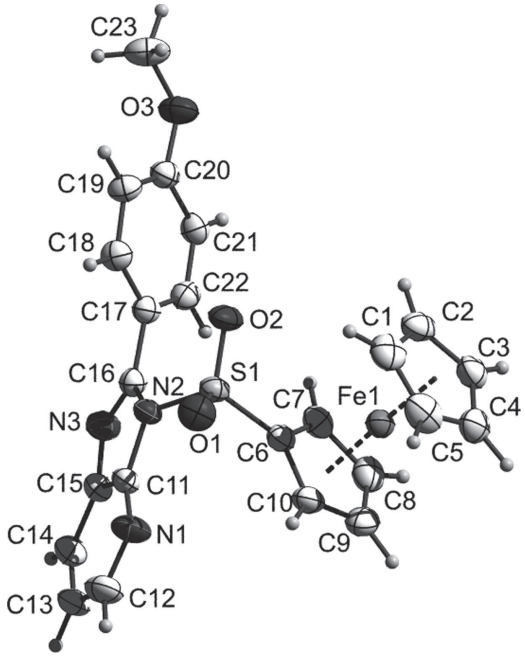
Crystal structure of 3-ferrocenylsulfonyl-2-(4-methoxyphenyl)-3*H*-imidazo[4,5-*b*]pyridine, $C_{23}H_{19}FeN_3O_3S$



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Abstract

$C_{23}H_{19}FeN_3O_3S$, monoclinic, $P2_1/n$ (no. 14), $a = 13.1372(6)$ Å, $b = 9.6866(5)$ Å, $c = 15.9700(8)$ Å, $\beta = 92.499(2)°$, $V = 2030.33(17)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0294$, $wR_{ref}(F^2) = 0.0807$, $T = 296(2)$K

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Red, block, size 0.10×0.15×0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.78 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω scans
$2\theta_{max}$:	56.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15379, 4844
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4223
$N(param)_{refined}$:	281
Programs:	Bruker programs [6], SHELX [7]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	0.6543	0.5328	0.2112	0.059
H(2A)	4*e*	0.5697	0.4389	0.0746	0.053
H(3A)	4*e*	0.3807	0.5082	0.0715	0.052
H(4A)	4*e*	0.3498	0.6447	0.2061	0.059
H(5A)	4*e*	0.5175	0.6590	0.2916	0.064
H(7A)	4*e*	0.5952	0.7318	−0.0351	0.046
H(8A)	4*e*	0.4135	0.8175	−0.0115	0.056
H(9A)	4*e*	0.4131	0.9440	0.1286	0.059
H(10A)	4*e*	0.5946	0.9397	0.1954	0.050
H(12A)	4*e*	0.7635	1.3003	0.1576	0.062
H(13A)	4*e*	0.7790	1.4139	0.0345	0.061
H(14A)	4*e*	0.8061	1.2929	−0.0886	0.057
H(18A)	4*e*	0.9754	0.7009	−0.0255	0.044
H(19A)	4*e*	1.0105	0.5020	−0.0986	0.045
H(21A)	4*e*	0.7599	0.5674	−0.2472	0.043
H(22A)	4*e*	0.7278	0.7673	−0.1750	0.040
H(23A)	4*e*	0.9845	0.2343	−0.2472	0.087
H(23B)	4*e*	0.9872	0.2804	−0.1530	0.087
H(23C)	4*e*	1.0477	0.3645	−0.2184	0.087

Source of material

All reagents and solvents were purchased form commercial sources and used as received. A solution of 2-(4-methoxyphenyl)-1*H*-imidazo[4,5-*b*]pyridine (0.6 g, 2.7 mmol) in acetonitrile (18 mL) was added to a mixture of ferrocenylsulfonylchloride (0.72 g, 2.5 mmol) and anhydrous sodium

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Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	4*e*	0.52184(2)	0.70195(2)	0.12198(1)	0.0380(1)	0.0290(1)	0.0332(1)	−0.00085(9)	0.00811(9)	0.00099(9)
S(1)	4*e*	0.76180(3)	0.78695(4)	0.10082(2)	0.0398(2)	0.0247(2)	0.0306(2)	0.0016(1)	0.0053(1)	0.0022(1)
N(1)	4*e*	0.7836(1)	1.1106(2)	0.1189(1)	0.0620(9)	0.0321(7)	0.0448(8)	0.0043(7)	−0.0021(7)	−0.0087(6)
N(2)	4*e*	0.8163(1)	0.9037(1)	0.03665(8)	0.0390(6)	0.0225(6)	0.0325(6)	−0.0014(5)	0.0040(5)	−0.0004(5)
N(3)	4*e*	0.8264(1)	1.0044(2)	−0.09075(9)	0.0541(8)	0.0325(7)	0.0405(7)	0.0034(6)	0.0116(6)	0.0064(6)
O(1)	4*e*	0.7953(1)	0.8216(1)	0.18387(7)	0.0544(7)	0.0491(7)	0.0331(6)	0.0008(6)	−0.0022(5)	0.0034(5)
O(2)	4*e*	0.7851(1)	0.6557(1)	0.06683(8)	0.0574(7)	0.0230(5)	0.0534(7)	0.0045(5)	0.0186(6)	0.0033(5)
O(3)	4*e*	0.8995(1)	0.3976(1)	−0.22688(8)	0.0583(7)	0.0371(7)	0.0524(7)	0.0094(6)	−0.0045(6)	−0.0134(6)
C(1)	4*e*	0.5825(2)	0.5435(2)	0.1930(1)	0.057(1)	0.043(1)	0.048(1)	−0.0009(8)	0.0015(8)	0.0153(8)
C(2)	4*e*	0.5360(1)	0.4919(2)	0.1177(1)	0.058(1)	0.0306(8)	0.0449(9)	0.0014(7)	0.0151(8)	0.0024(7)
C(3)	4*e*	0.4320(1)	0.5302(2)	0.1159(1)	0.0489(9)	0.0362(9)	0.0462(9)	−0.0093(7)	0.0089(7)	−0.0001(7)
C(4)	4*e*	0.4149(2)	0.6052(2)	0.1903(1)	0.054(1)	0.044(1)	0.052(1)	−0.0088(8)	0.0251(8)	−0.0010(8)
C(5)	4*e*	0.5070(2)	0.6126(2)	0.2374(1)	0.079(1)	0.051(1)	0.0326(8)	−0.013(1)	0.0144(8)	0.0011(8)
C(6)	4*e*	0.6342(1)	0.8240(2)	0.0856(1)	0.0389(7)	0.0265(7)	0.0350(8)	−0.0016(6)	0.0065(6)	0.0001(6)
C(7)	4*e*	0.5727(1)	0.7833(2)	0.0136(1)	0.0452(8)	0.0346(8)	0.0343(8)	−0.0052(7)	0.0026(6)	0.0046(7)
C(8)	4*e*	0.4732(1)	0.8325(2)	0.0264(1)	0.0436(9)	0.043(1)	0.054(1)	−0.0020(8)	−0.0012(8)	0.0148(8)
C(9)	4*e*	0.4729(1)	0.9024(2)	0.1040(1)	0.0478(9)	0.0344(9)	0.067(1)	0.0070(8)	0.0142(9)	0.0065(9)
C(10)	4*e*	0.5723(1)	0.8986(2)	0.1416(1)	0.0505(9)	0.0283(8)	0.0459(9)	0.0010(7)	0.0126(7)	−0.0045(7)
C(11)	4*e*	0.8021(1)	1.0469(2)	0.0479(1)	0.0339(7)	0.0230(7)	0.0408(8)	−0.0014(6)	−0.0009(6)	−0.0013(6)
C(12)	4*e*	0.7757(2)	1.2485(2)	0.1100(1)	0.057(1)	0.0345(9)	0.063(1)	0.0045(8)	−0.0071(9)	−0.0157(9)
C(13)	4*e*	0.7844(1)	1.3182(2)	0.0355(2)	0.0451(9)	0.0240(8)	0.083(2)	0.0006(7)	−0.0033(9)	−0.0034(9)
C(14)	4*e*	0.8011(1)	1.2476(2)	−0.0376(1)	0.0471(9)	0.0298(8)	0.067(1)	−0.0002(7)	0.0074(8)	0.0110(8)
C(15)	4*e*	0.8099(1)	1.1053(2)	−0.0308(1)	0.0366(7)	0.0281(8)	0.0471(9)	−0.0011(6)	0.0062(6)	0.0030(7)
C(16)	4*e*	0.8290(1)	0.8876(2)	−0.05051(9)	0.0324(7)	0.0305(7)	0.0328(7)	0.0004(6)	0.0058(6)	0.0017(6)
C(17)	4*e*	0.8478(1)	0.7545(2)	−0.09188(9)	0.0364(7)	0.0287(7)	0.0300(7)	0.0004(6)	0.0076(6)	0.0013(6)
C(18)	4*e*	0.9321(1)	0.6741(2)	−0.0702(1)	0.0377(8)	0.0386(8)	0.0342(8)	0.0030(7)	−0.0025(6)	−0.0028(7)
C(19)	4*e*	0.9534(1)	0.5546(2)	−0.1137(1)	0.0377(7)	0.0369(8)	0.0389(8)	0.0082(7)	0.0000(6)	0.0006(7)
C(20)	4*e*	0.8883(1)	0.5143(2)	−0.1802(1)	0.0405(8)	0.0289(7)	0.0344(8)	−0.0005(6)	0.0061(6)	−0.0004(6)
C(21)	4*e*	0.8035(1)	0.5944(2)	−0.2027(1)	0.0362(7)	0.0369(8)	0.0342(8)	−0.0033(6)	−0.0007(6)	−0.0004(7)
C(22)	4*e*	0.7841(1)	0.7135(2)	−0.1592(1)	0.0327(7)	0.0336(8)	0.0346(8)	0.0024(6)	0.0029(6)	0.0045(6)
C(23)	4*e*	0.9869(2)	0.3122(2)	−0.2100(1)	0.069(1)	0.043(1)	0.061(1)	0.0195(9)	0.005(1)	−0.0085(9)

carbonate (0.26 g, 2.5 mmol) with constant stirring. The mixture was heated to reflux temperature for 48 h. The acetonitrile was evaporated under vacuum yielding a brown solid. The crude product was purified by column chromatography (SiO_2, ethyl acetate petroleum ether v/v = 1:9) to give the title compound (0.25 g, 52% yield) as dark red crystals, mp: 483–485 K.

Experimental details

The crystal structure was solved by the direct methods. The hydrogen atoms were placed on calculated positions using the AFIX 43, 13 or 33 option of the SHELX-97 programm [6].

Discussion

Imidazo[4,5-*b*]pyridine and their derivatives possess diverse pharmacological properties, including anticancer, antiviral, and other important biological activities [1–3]. The asymmetric unit of the title compound contains one title complex. There is a imidazo[4,5-*b*]pyridine and a sandwich ferrocene moiety in the structure. The imidazo[4,5-*b*]pyridine ring system (C_{11}—C_{16},N_1—N_3) is planar, and its dihedral angle to the substituted cyclopentadienyl plane (C_6—C_{10}) is 102.03(7)°. There are some differences in bond lengths and bond angles between the substituted and unsubstituted cyclopentadienyl ring [4, 5]. The bond length of substituted cyclopentadienyl range from 1.412(3) to 1.431(2) Å, whereas in unsubstituted one the range is 1.397(3) to 1.418(3) Å.

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