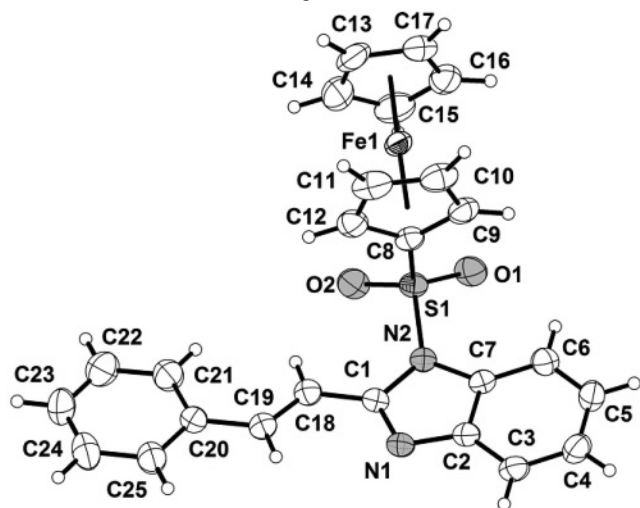


Crystal structure of 2-cinnameryl-1-ferrocenylsulfonyl-1*H*-1,3-benzimidazole, C₂₅H₂₀FeN₂O₂S

Lu-Yao Wang*, Dong Meng, Lu Li and Bing-Sen Zhang

Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China

Received November 29, 2013, accepted March 17, 2014, available online April 10, 2014, CCDC no. 967702



Abstract

C₂₅H₂₀FeN₂O₂S, monoclinic, *C*2/*c* (no. 15), *a* = 24.0837(6) Å, *b* = 9.9163(2) Å, *c* = 19.9314(5) Å, β = 117.055(1)°, *V* = 4239.2 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0301, *wR*_{ref}(*F*²) = 0.0848, *T* = 296 K.

Table 1. Data collection and handling.

Crystal:	red blocks, size 0.20×0.30×0.40 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ :	8.35 cm ^{−1}
Diffractionmeter, scan mode:	Bruker APEXII CCD, φ and ω
2 θ _{max} :	56.16°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	26624, 5143
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 4353
<i>N</i> (<i>param</i>) _{refined} :	280
Programs:	SHELX [15]

Source of material

All reagents and solvents were purchased from commercial sources and used as received. The title compound (Fig.) is obtained from a series of reactions involving the synthesis of 2-cinnameryl-1-ferrocenylsulfonyl-benzimidazole from 2-cinnamerylbenzimidazole and ferrocenyl sulfonyl chloride. A solution of 2-cinnamerylbenzimidazole (1.21 g, 5.5 mmol) in acetonitrile 20 mL was added to a mixture of ferrocenylsulfonyl chloride (1.43 g, 5.0 mmol) and anhydrous sodium carbonate (0.53 g, 5.0 mmol) with constantly stirring [1,2]. The mixture was heated to reflux for 24 h [3]. The acetonitrile was evaporated under vacuum to leave a brown solid. The crude product was purified by column chromatography (SiO₂, ethyl acetate: petroleum ether *v/v* = 3:7) to give the title compound (2 g, 78% yield) as dark red crystals, mp: 495–497 K.

Discussion

Benzimidazoles and their derivatives have been associated with various biological activities. Compounds containing benzimidazole moieties have shown good bactericidal, fungicidal and antimalarial activities [4]. The asymmetric unit of title structure contains one 2-cinnameryl-1-ferrocenylsulfonyl-1*H*-1,3-benzimidazole molecule. There is a benzimidazole ring and a ferrocene in the structure. The benzimidazole ring (C2–C7, N1 and N2) and 2-sited cinnameryl (C18–C25) are almost coplanar. The dihedral angles between the benzimidazole plane and the substituted cyclopentadienyl plane (C8–C12) is 85.89(6)°. There are some differences in bond length and bond angle between the substituted and unsubstituted (C13–C17) cyclopentadienyl ring. The bond lengths of substituted cyclopentadienyl range from 1.403(3)–1.432(2) Å, whereas the bond lengths in the unsubstituted one are 1.397(3)–1.419(3) Å. The bond angle of substituted cyclopentadienyl range from 106.06(17)–109.47(17)° which in unsubstituted one are from 107.3(2)–108.52(49)°.

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(3A)	8 <i>f</i>	0.4463	0.4527	0.7535	0.062
H(4A)	8 <i>f</i>	0.3756	0.5983	0.7651	0.068
H(5A)	8 <i>f</i>	0.3038	0.7151	0.6628	0.065
H(6A)	8 <i>f</i>	0.2995	0.6929	0.5449	0.058
H(9A)	8 <i>f</i>	0.2095	0.4968	0.4462	0.059
H(10A)	8 <i>f</i>	0.1561	0.2647	0.4132	0.073
H(11A)	8 <i>f</i>	0.2186	0.1113	0.3708	0.077
H(12A)	8 <i>f</i>	0.3128	0.2427	0.3766	0.063
H(13A)	8 <i>f</i>	0.1379	0.2051	0.1982	0.082
H(14A)	8 <i>f</i>	0.2269	0.3489	0.2007	0.091
H(15A)	8 <i>f</i>	0.2166	0.5872	0.2451	0.082
H(16A)	8 <i>f</i>	0.1213	0.5876	0.2692	0.077
H(17A)	8 <i>f</i>	0.0730	0.3534	0.2405	0.077
H(18A)	8 <i>f</i>	0.4414	0.3884	0.4521	0.054
H(19A)	8 <i>f</i>	0.5126	0.2370	0.5765	0.057
H(21A)	8 <i>f</i>	0.4769	0.2912	0.3837	0.077
H(22A)	8 <i>f</i>	0.5166	0.1696	0.3164	0.098
H(23A)	8 <i>f</i>	0.5867	−0.0021	0.3729	0.091
H(24A)	8 <i>f</i>	0.6203	−0.0478	0.4981	0.087
H(25A)	8 <i>f</i>	0.5809	0.0711	0.5664	0.072

* Correspondence author (e-mail: xdwly@163.com)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Fe(1)	8 <i>f</i>	0.19979(1)	0.37100(2)	0.31891(1)	0.0443(1)	0.0430(1)	0.0313(1)	0.00087(9)	0.0113(1)	−0.00381(8)
S(1)	8 <i>f</i>	0.32819(2)	0.53998(4)	0.42788(2)	0.0406(2)	0.0415(2)	0.0336(2)	0.0022(2)	0.0136(2)	0.0070(1)
O(1)	8 <i>f</i>	0.29902(6)	0.6658(1)	0.42529(7)	0.0563(7)	0.0409(6)	0.0525(7)	0.0081(5)	0.0151(6)	0.0096(5)
O(2)	8 <i>f</i>	0.35903(6)	0.5185(1)	0.38258(7)	0.0541(7)	0.0738(8)	0.0406(6)	0.0058(6)	0.0244(5)	0.0145(6)
N(1)	8 <i>f</i>	0.45124(6)	0.4054(2)	0.61978(7)	0.0367(7)	0.0563(8)	0.0362(7)	0.0055(6)	0.0106(5)	0.0003(6)
N(2)	8 <i>f</i>	0.38190(6)	0.5201(1)	0.51846(7)	0.0368(6)	0.0430(7)	0.0343(6)	0.0027(5)	0.0124(5)	0.0004(5)
C(1)	8 <i>f</i>	0.43118(7)	0.4245(2)	0.54770(8)	0.0324(7)	0.0437(8)	0.0381(7)	0.0004(6)	0.0119(6)	−0.0011(6)
C(2)	8 <i>f</i>	0.41460(7)	0.4860(2)	0.64093(9)	0.0343(7)	0.0494(8)	0.0365(7)	−0.0019(6)	0.0117(6)	−0.0039(6)
C(3)	8 <i>f</i>	0.41725(8)	0.5000(2)	0.71207(9)	0.0440(9)	0.069(1)	0.0352(8)	0.0002(8)	0.0122(7)	−0.0030(8)
C(4)	8 <i>f</i>	0.37496(9)	0.5869(2)	0.7185(1)	0.055(1)	0.073(1)	0.0450(9)	−0.0068(9)	0.0255(8)	−0.0149(9)
C(5)	8 <i>f</i>	0.33161(9)	0.6576(2)	0.6565(1)	0.052(1)	0.055(1)	0.059(1)	−0.0006(8)	0.0277(9)	−0.0148(8)
C(6)	8 <i>f</i>	0.32850(8)	0.6451(2)	0.5860(1)	0.0454(9)	0.0456(9)	0.0495(9)	0.0037(7)	0.0169(7)	−0.0040(7)
C(7)	8 <i>f</i>	0.37068(7)	0.5577(2)	0.57946(8)	0.0362(7)	0.0403(7)	0.0363(7)	−0.0039(6)	0.0128(6)	−0.0050(6)
C(8)	8 <i>f</i>	0.27655(7)	0.4104(2)	0.41451(8)	0.0419(8)	0.0431(8)	0.0306(7)	−0.0006(6)	0.0102(6)	0.0022(6)
C(9)	8 <i>f</i>	0.22339(8)	0.4179(2)	0.42821(9)	0.0495(9)	0.062(1)	0.0331(8)	−0.0082(8)	0.0168(7)	−0.0040(7)
C(10)	8 <i>f</i>	0.19461(9)	0.2895(2)	0.4109(1)	0.060(1)	0.071(1)	0.0442(9)	−0.019(1)	0.0188(8)	0.0019(9)
C(11)	8 <i>f</i>	0.2292(1)	0.2049(2)	0.3875(1)	0.072(1)	0.0450(9)	0.052(1)	−0.0097(9)	0.0070(9)	0.0076(8)
C(12)	8 <i>f</i>	0.28068(9)	0.2772(2)	0.3894(1)	0.054(1)	0.0446(9)	0.0462(9)	0.0074(7)	0.0107(7)	0.0015(7)
C(13)	8 <i>f</i>	0.1459(1)	0.3001(2)	0.2131(1)	0.081(1)	0.064(1)	0.0378(9)	0.004(1)	0.0082(9)	−0.0160(9)
C(14)	8 <i>f</i>	0.1946(1)	0.3791(3)	0.2144(1)	0.079(2)	0.112(2)	0.039(1)	0.023(1)	0.028(1)	0.004(1)
C(15)	8 <i>f</i>	0.1891(1)	0.5104(2)	0.2390(1)	0.078(1)	0.071(1)	0.041(1)	−0.006(1)	0.0140(9)	0.0132(9)
C(16)	8 <i>f</i>	0.1367(1)	0.5105(2)	0.2517(1)	0.068(1)	0.060(1)	0.047(1)	0.017(1)	0.0104(9)	−0.0013(8)
C(17)	8 <i>f</i>	0.1102(1)	0.3818(2)	0.2361(1)	0.050(1)	0.077(1)	0.047(1)	0.0022(9)	0.0051(8)	−0.0079(9)
C(18)	8 <i>f</i>	0.45594(8)	0.3600(2)	0.50170(9)	0.0411(8)	0.0547(9)	0.0387(8)	0.0032(7)	0.0166(7)	−0.0005(7)
C(19)	8 <i>f</i>	0.49802(8)	0.2630(2)	0.52655(9)	0.0450(9)	0.0562(9)	0.0375(8)	0.0050(7)	0.0158(7)	−0.0016(7)
C(20)	8 <i>f</i>	0.52383(7)	0.1929(2)	0.48234(9)	0.0384(8)	0.0538(9)	0.0443(8)	−0.0010(7)	0.0180(7)	−0.0054(7)
C(21)	8 <i>f</i>	0.5054(1)	0.2223(2)	0.4072(1)	0.060(1)	0.085(1)	0.047(1)	0.015(1)	0.0249(9)	0.004(1)
C(22)	8 <i>f</i>	0.5293(1)	0.1494(3)	0.3669(1)	0.077(2)	0.126(2)	0.049(1)	0.008(2)	0.035(1)	−0.007(1)
C(23)	8 <i>f</i>	0.5715(1)	0.0476(3)	0.4006(1)	0.068(1)	0.096(2)	0.077(2)	0.002(1)	0.046(1)	−0.023(1)
C(24)	8 <i>f</i>	0.5910(1)	0.0198(2)	0.4748(1)	0.069(1)	0.075(1)	0.082(2)	0.018(1)	0.042(1)	−0.004(1)
C(25)	8 <i>f</i>	0.56740(9)	0.0914(2)	0.5158(1)	0.059(1)	0.067(1)	0.054(1)	0.0141(9)	0.0257(9)	0.0006(9)

Acknowledgments. This work is financially supported by the Ministry of Education of Beijing's University Key Teacher (2010).

References

- Wang, L.Y.; Gao, Y.; Yang, B.Q.; Shi, Z.: Synthesis, Structure and Bioactivity of Nitrobenzoimidazole Derivatives. *Chem. J. Chin. University*. **26** (2005) 2241-2246.
- Slocum, D. W.; Achermann, W.: The Preparation of Several N-Substituted and N,N-Disubstituted Ferrocenesulfonamides. *Syn. React. Inorg. Met. Chem.* **12** (1982) 397-405.
- Li, M.; Bai, Y. J.; Lu, J.: Ferrocene derivatives (I). Synthesis and X-ray structure determination of *N*-*o*-methylphenylferrocenesulfonamide. *J. Organomet. Chem.* **637-639** (2001) 738-741.
- Wang, L. Y.; Tian, M.; Chen, B.; Shi, Z.: Synthesis, Characterization and Inhibition Activities of Cyclopentylmethylbenzimidazole Derivatives. *Chin. J. Appl. Chem.* **22** (2005) 1087-1091.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112-122.