Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-[4-Ferrocenyl-5-(1*H*-1,2,4-triazol-1-yl)-1,3-thiazol-2-yl]-2-nitrobenzamide

Ling Shao, Xin Zhou, Yan Hu and Jian-Xin Fang*

State Key Laboratory and Institute of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail: shaoling1999@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.049
wR factor = 0.115
Data-to-parameter ratio = 14.6

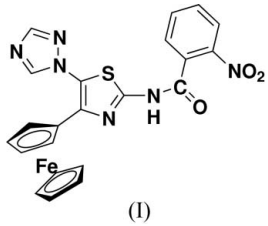
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 November 2005
Accepted 1 December 2005
Online 7 December 2005

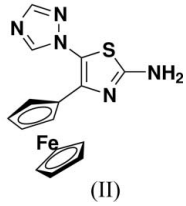
The title compound, $[Fe(C_5H_5)(C_{17}H_{11}N_6O_3S)]$, has been synthesized as a potential anticancer agent. There are two molecules in the asymmetric unit. In the crystal structure, the molecules are associated *via* N—H···N and C—H···O interactions.

Comment

2-Amino-1,3-thiazoles are known as synthetic intermediates and therapeutic agents. Two examples are 5-arylthio-2-acylamino-1,3-thiazoles, known to be antitumour agents (Matsuo *et al.*, 1990), and 2-(arylmethylcarbonylamino)-1,3-thiazole derivatives, known to be cyclin-dependent kinase inhibitors (Pevarello *et al.*, 2004). The ferrocenyl group has been invoked as a bonus in the design of new biologically active molecules, as it is neutral, chemically stable, non-toxic and able to cross cell membranes (Dombrowski *et al.*, 1986). In our search for novel thiazole compounds with potential anticancer activity, we intend to synthesize thiazole compounds incorporating the ferrocenyl unit. In this paper, we report the crystal structure of the title compound, (I).



(I)



(II)

The asymmetric unit of (I) consists of two molecules, *A* and *B* (Figs. 1 and 2). The molecules have five planar subunits, namely the thiazole ring (*P*1), the 2-nitrophenyl ring, C17–C22 (*P*2), the triazole ring (*P*3), the substituted cyclopentadienyl ring, C4–C8 (*P*4), and the cyclopentadienyl ring, C9–C13 (*P*5). The dihedral angles between *P*1 and *P*2, *P*2 and *P*3, *P*3 and *P*4 in molecule *A* are 66.7 (2), 51.7 (3) and 15.5 (2)°, respectively. The corresponding angles in molecule *B* are 76.3 (3), 80.0 (2) and 20.8 (3)°, respectively.

The Fe—C (Table 1) are comparable with those in 2-amino-4-(ferrocenyl)-5-(1*H*-1,2,4-triazol-1-yl)-1,3-thiazole, (II) [2.015 (4)–2.048 (3) Å; Shao *et al.*, 2005]. The cyclopentadienyl rings are in the eclipsed geometry, as evidenced by the C4—*Cg*1—*Cg*2—C9 torsion angle of 2.2 (3)° in molecule *A*, where *Cg*1 and *Cg*2 are the centroids of the C_5H_4 and C_5H_5 rings, respectively. The corresponding angle in molecule *B*, C26—*Cg*3—*Cg*4—C31, is −9.0 (4)°. However, in compound (II), they are not eclipsed (Shao *et al.*, 2005).

In compound (I), the molecules are associated *via* N—H···N and C—H···O interactions (Table 2 and Fig. 3).

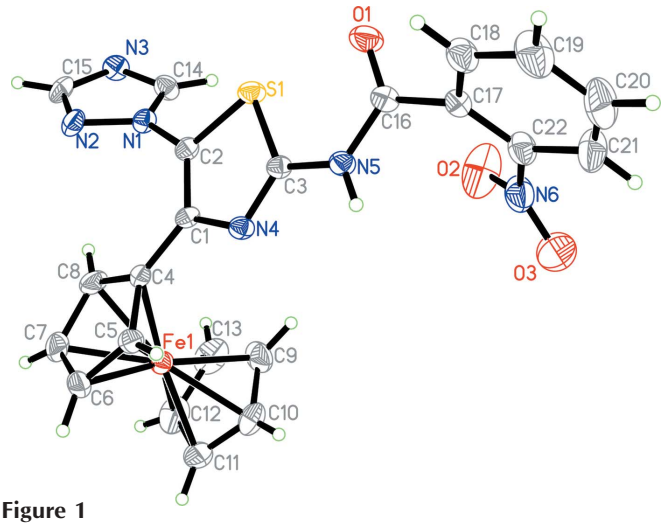


Figure 1
One of the two independent molecules of (I), showing displacement ellipsoids at the 30% probability level.

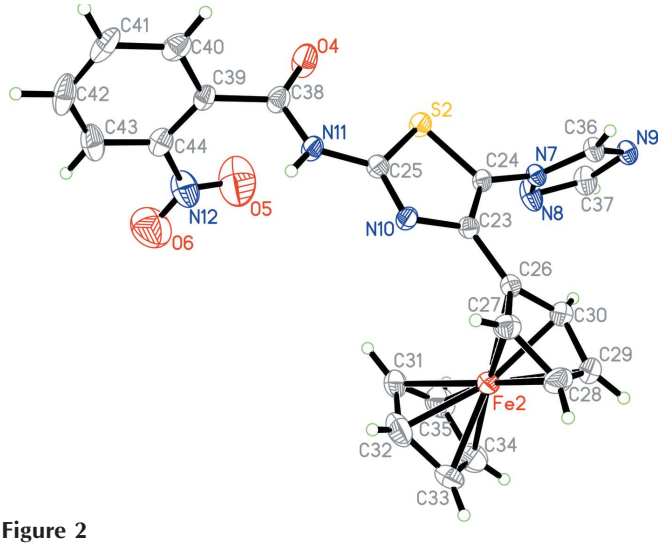


Figure 2
The second of the two independent molecules of (I), showing displacement ellipsoids at the 30% probability level.

Experimental

2-Nitrobenzoyl chloride (2 mmol) was added to a solution of 2-amino-4-(ferrocenyl)-5-(1*H*-1,2,4-triazol-1-yl)-1,3-thiazole (1.8 mmol) in dry pyridine (15 ml). After stirring for 8 h, the reaction mixture was poured into water (200 ml). After 2 d, the precipitate was collected, washed with water and dried. After recrystallization from ethanol, the title compound was obtained (yield 46%). Analysis calculated for $C_{22}H_{16}FeN_6O_3S$: C 53.71, H 3.53, N 16.34%; found: C53.68, H 3.73, N 16.63%.

Crystal data

$[Fe(C_5H_5)(C_{17}H_{11}N_6O_3S)]$
M_r = 500.32
Monoclinic, $P2_1/c$
a = 11.9851 (15) Å
b = 28.541 (4) Å
c = 13.4467 (17) Å
β = 111.046 (2)°
V = 4292.8 (9) Å^3
Z = 8
D_x = 1.548 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3749 reflections
θ = 2.7–22.5°
μ = 0.84 mm^{-1}
T = 294 (2) K
Block, red
0.26 × 0.20 × 0.16 mm

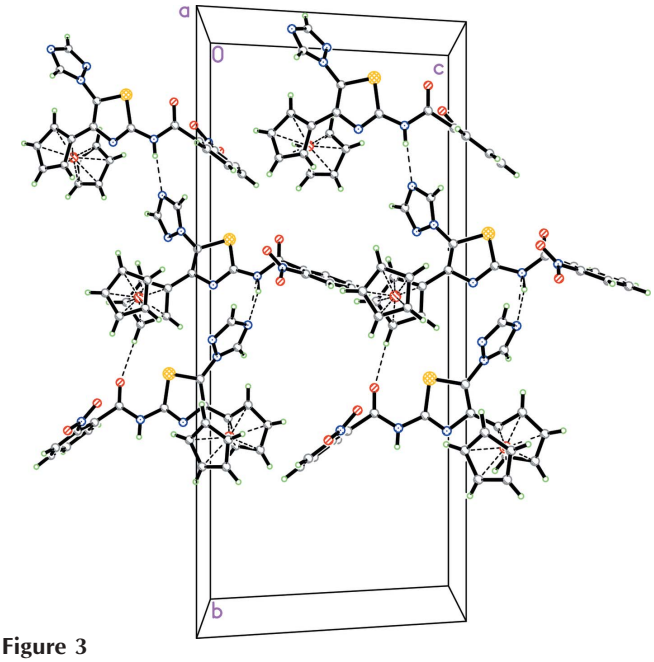


Figure 3
A packing diagram for (I), viewed down the *a* axis. Dashed lines indicate the hydrogen bonds.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.802, T_{max} = 0.878
24210 measured reflections
8783 independent reflections
4708 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 26.5°
$h = -14 \rightarrow 14$
$k = -35 \rightarrow 29$
$l = -16 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.115
S = 0.99
8783 reflections
601 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—C13	2.023 (4)	Fe2—C27	2.015 (4)
Fe1—C7	2.024 (4)	Fe2—C28	2.022 (4)
Fe1—C8	2.025 (4)	Fe2—C32	2.024 (5)
Fe1—C12	2.025 (5)	Fe2—C33	2.028 (4)
Fe1—C9	2.032 (4)	Fe2—C31	2.029 (5)
Fe1—C6	2.033 (4)	Fe2—C34	2.032 (5)
Fe1—C10	2.038 (4)	Fe2—C26	2.043 (4)
Fe1—C11	2.042 (4)	Fe2—C29	2.043 (4)
Fe1—C5	2.045 (4)	Fe2—C35	2.046 (5)
Fe1—C4	2.052 (4)	Fe2—C30	2.057 (4)
C14—N1—C2—S1	49.5 (5)	C36—N7—C24—S2	98.6 (4)
N2—N1—C2—S1	−126.5 (3)	N8—N7—C24—S2	−77.6 (4)
C16—N5—C3—S1	1.8 (5)	C38—N11—C25—S2	3.1 (6)
N4—C1—C4—C5	14.9 (5)	N10—C23—C26—C27	−20.3 (5)
C3—N5—C16—O1	4.2 (6)	C25—N11—C38—O4	−13.4 (7)
N5—C16—C17—C22	66.1 (5)	N11—C38—C39—C44	−76.6 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···N9^{i}	0.85 (3)	2.13 (4)	2.952 (4)	160 (4)
N11—H11A···N3^{ii}	0.85 (3)	2.10 (4)	2.932 (4)	165 (3)
C6—H6···O4^{i}	0.93	2.48	3.333 (3)	153

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x+1, y, z$.

The amino H atoms were located in a difference Fourier map and refined isotropically, with the distance restraint N—H = 0.85 (3) Å. Other H atoms were placed in calculated positions, with C—H = 0.93 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China (grant Nos. 29872022 and 20172030) and the Key Project of the Chinese Ministry of Education (grant No. 105046).

References

Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dombrowski, K. E., Baldwin, W. & Sheats, J. E. (1986). *J. Organomet. Chem.* **302**, 281–306.
Matsuo, M., Ogino, T., Igari, N., Seno, H. & Shimomura, K. (1990). EP Patent 0 412 404.
Pevarello, P., Amici, R., Villa, M., Salom, B., Vulpetti, A. & Varasi, M. (2004). US Patent 2004 6 784 198.
Shao, L., Hu, Y., Zhou, X., Zhang, Q. & Fang, J. X. (2005). *Acta Cryst.* E**61**, m1269–m1271.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.