Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-Amino-4-(ferrocenyl)-5-(1*H*-1,2,4-triazol-1-yl)-1,3-thiazole

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

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

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

Received 11 May 2005
Accepted 31 May 2005
Online 10 June 2005

In the title compound, $[Fe(C_5H_5)(C_{10}H_8N_5S)]$, the Fe—C bond lengths are in the range 2.015 (4)–2.048 (3) Å. The thiazole and triazole rings make dihedral angles of 84.2 (1) and 10.7 (2)°, respectively, with the substituted cyclopentadienyl ring (C_5H_4). The crystal packing is stabilized by intermolecular N—H···N hydrogen bonds involving the amino H atoms.

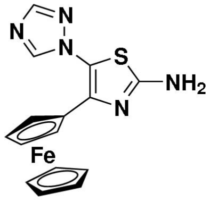
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.031
wR factor = 0.092
Data-to-parameter ratio = 14.5

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

Comment

Ferrocene-containing organic compounds often exhibit biological activity (Biot *et al.*, 2000; Fang *et al.*, 2003*a*,*b*). Thiazoles and their derivatives are found to be associated with various biological activities, such as antibacterial, antifungal and anti-inflammatory activities (Gusmeroli *et al.*, 2003; Wilson *et al.*, 2001). Triazole antifungals are known as potent inhibitors of cytochrome P450 monooxygenase in the process of fungal biosynthesis of ergosterol, which is an important constituent of fungal cell membranes (Hiroshi *et al.*, 1995). In our search for novel aminothiazole compounds with potent fungicidal activities, we intend to synthesize 2-aminothiazole compounds incorporating ferrocene and 1*H*-1,2,4-triazole units. We have investigated the crystal structure of the title compound, (I) (Fig. 1), and present the results here.



(I)

In the molecule of (I), the Fe—C bond lengths are in the range 2.015 (4)–2.048 (3) Å (Table 1). The Fe1···*Cg*1 and Fe1···*Cg*2 distances are 1.650 (2) and 1.640 (2) Å, respectively, and the *Cg*1···Fe1···*Cg*2 angle is 179.4 (2)°, where *Cg*1 and *Cg*2 are the centroids of the C_5H_5 and C_5H_4 rings, respectively. The cyclopentadienyl rings are not in the eclipsed geometry, as evidenced by the C1—*Cg*1—*Cg*2—C6 and C3—*Cg*1—*Cg*2—C8 torsion angles of 39.7 (2)° and 32.5 (2)°, respectively. The C—C bond lengths in both cyclopentadienyl rings are normal (Anderson *et al.*, 2003).

The thiazole (C11/C12/S1/C15/N4) and triazole (N1/N2/C14/N3/C13) rings make dihedral angles of 84.2 (1) and 10.7 (2)°, respectively, with the substituted cyclopentadienyl ring (C6–C10).

The crystal packing of (I) (Fig. 2) is stabilized by intermolecular N—H···N hydrogen bonds (Table 2) involving the amino H atoms.

doi:10.1107/S1600536805017186

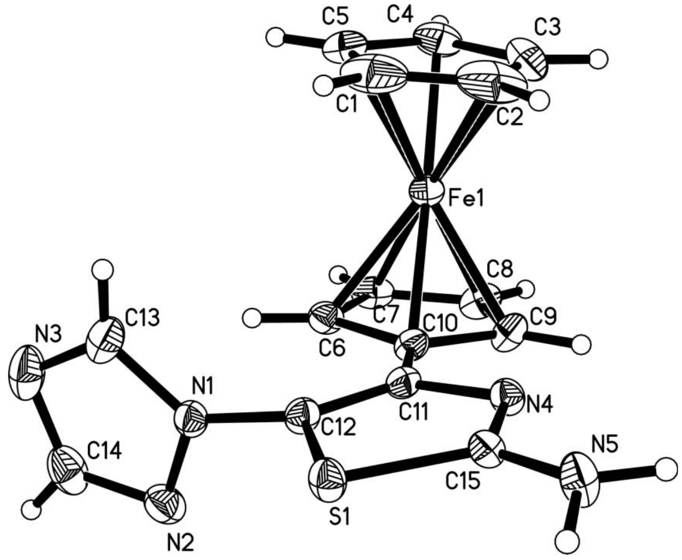


Figure 1
A view of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

Experimental

Preparation of the title compound was based on a Hantzsch reaction (Hantzsch & Weber, 1887). 2-Bromo-2–1*H*-1,2,4-triazole-1-acetylferrocene (1 mmol) and thiourea (2 mmol) were dissolved in warm ethanol (100 ml). The mixture was refluxed for 10 h and ammonia solution (5%, 20 ml) was then added. The red crystals that formed were filtered and dried. After recrystallization from methanol, the title compound was obtained. Analysis, calculated for $C_{15}H_{13}FeN_5S$: C 51.30, H 3.73, N 19.94%; found: C 51.35, H 3.61, N 19.72%. Yield 46%.

Crystal data

$[Fe(C_5H_5)(C_{10}H_8N_5S)]$
$M_r = 351.21$
Monoclinic, $P2_1/n$
$a = 7.507\ (5)$ Å
$b = 20.018\ (14)$ Å
$c = 9.600\ (7)$ Å
$\beta = 103.330\ (11)^\circ$
$V = 1403.8\ (17)$ Å^3
$Z = 4$

$D_x = 1.662$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3711 reflections
$\theta = 2.4$–26.4°
$\mu = 1.23$ mm^{-1}
$T = 293\ (2)$ K
Block, red
$0.22 \times 0.16 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.759$, $T_{max} = 0.885$
7935 measured reflections

2885 independent reflections
2262 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.5^\circ$
$h = -9 \rightarrow 8$
$k = -22 \rightarrow 25$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.092$
$S = 1.09$
2885 reflections
199 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.9147P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

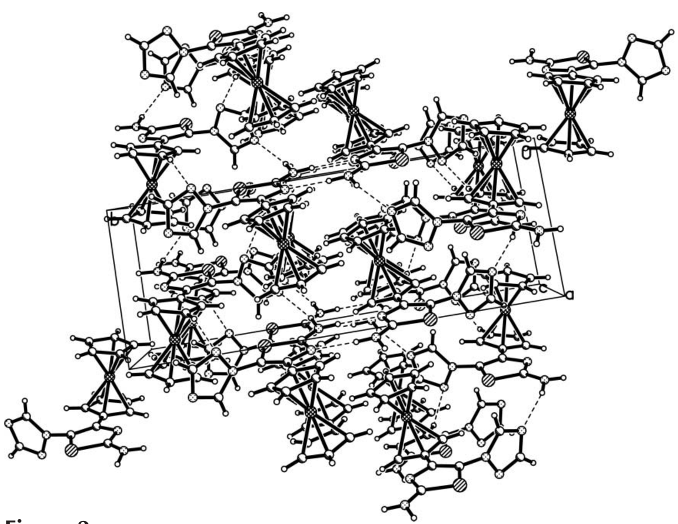


Figure 2
A packing diagram for (I), viewed down the *c* axis Dashed lines indicate the hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

Fe1—C2	2.015 (4)	S1—C15	1.736 (3)
Fe1—C1	2.018 (4)	S1—C12	1.737 (3)
Fe1—C6	2.022 (3)	N1—C13	1.336 (4)
Fe1—C3	2.028 (3)	N1—N2	1.364 (3)
Fe1—C9	2.029 (3)	N1—C12	1.410 (3)
Fe1—C10	2.029 (3)	N4—C15	1.308 (3)
Fe1—C5	2.040 (3)	N4—C11	1.385 (3)
Fe1—C4	2.042 (3)	N5—C15	1.341 (3)
Fe1—C7	2.043 (3)	C10—C11	1.458 (4)
Fe1—C8	2.048 (3)	C11—C12	1.350 (4)
C15—S1—C12	87.67 (12)	C12—C11—C10	126.2 (2)
C13—N1—N2	109.4 (2)	C11—C12—N1	126.6 (2)
C13—N1—C12	129.2 (2)	C11—C12—S1	111.73 (19)
C14—N2—N1	101.6 (3)	N3—C13—N1	110.3 (3)
C13—N3—C14	102.7 (3)	N3—C13—H13	124.9
C15—N4—C11	110.9 (2)	N3—C14—H14	122.0
C2—C1—C5	108.5 (4)	N4—C15—S1	115.45 (19)
C12—C11—N4	114.2 (2)		
C12—N1—N2—C14	−175.4 (2)	N2—N1—C12—S1	−94.5 (3)
C6—C10—C11—C12	11.2 (4)	C15—S1—C12—C11	1.2 (2)
C6—C10—C11—N4	−173.2 (3)	N1—N2—C14—N3	0.3 (3)
N2—N1—C12—C11	77.0 (3)	C12—S1—C15—N5	178.7 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N4^{i}	0.93	2.08	3.016 (4)	176
N5—H5*B*···N3^{ii}	0.92	2.27	3.136 (4)	157

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

The amino H atoms were located in a difference Fourier map and refined isotropically, with the distance restraint N—H = 0.92 (1) Å. The C-bound H atoms were placed in calculated positions, with C—H = 0.93 Å, and included in the final cycles of refinement 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 Research Fund for the Doctoral Programme of Higher Education (grant No. 9805520).

References

Anderson, F. P., Gallagher, J. F., Kenny, P. T. M., Ryan, C. & Savage, D. (2003). *Acta Cryst.* C**59**, m13–m15

Biot, C., Delhaes, L., Maciejewski, L. A., Mortuaire, M., Camus, D., Divid, S. & Brocard, S. S. (2000). *Eur. J. Med. Chem.* **35**, 707–714.

Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Fang, J., Jin, Z., Li, Z. & Liu, W. (2003*a*). *J. Organomet. Chem.* **674**, 1–9.

Fang, J., Jin, Z., Li, Z. & Liu, W. (2003*b*). *Appl. Organomet. Chem.* **17**, 145–153.

Gusmeroli, M., Ciapessoni A., Bettarini, F., Osi, S., Mirenna, L., Camaggi, G. & Elmini, A. (2003). WO Patent No. 03 050 096.

Hantzsch, A. & Weber, J. H. (1887). *Ber. Dtsch. Chem. Ges.* **20**, 3118–3132.

Hiroshi M., Koichi K., Tomoharu T. & Naohito O. (1995). *Bioorg. Med. Chem. Lett.* **5**, 1479–1482.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Wilson, K. J., Utig, N., Subhasinghe, N., Hoffman, J. B., Rudolph, N. J., Soll, R., Molloy, C. J., Bone, R., Green, D. & Randall, J. (2001). *Bioorg. Med. Chem. Lett.* **11**, 915–918.