Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(2,5-Dimethoxyphenyl)-3-ferrocenyl-2-(1*H*-1,2,4-triazol-1-yl)prop-2-en-1-one

Jian-Bing Liu, Hong Dai, Li-Chun Li, Wei-Feng Tao 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: liu_jianbing65@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.129
Data-to-parameter ratio = 15.3

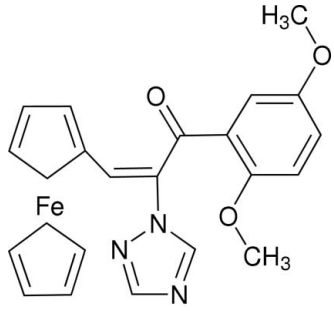
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[Fe(C_5H_5)(C_{18}H_{16}N_3O_3)]$, the propene plane is tilted from its parent cyclopentadienyl ring by a small dihedral angle [17.9 (5)°], whereas the triazole and dimethoxyphenyl groups are nearly perpendicular to the propene plane [dihedral angles 75.9 (2) and 67.9 (2)°, respectively]. The cyclopentadienyl rings exhibit an almost eclipsed geometry.

Received 21 September 2005
Accepted 22 November 2005
Online 7 December 2005

Comment

We are interested in 1*H*-1,2,4-triazole derivatives because of their biological activities (Moreno-Manas *et al.*, 1992; Chu *et al.*, 1999; Liu *et al.*, 1998). As part of an investigation on ferrocenyl-triazole derivatives (Fang *et al.*, 2003; Jin *et al.*, 2005), we report here the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The Fe—C bond lengths range from 2.021 (3) to 2.042 (3) Å. The Fe1···*Cg*1 and Fe1···*Cg*2 distances are 1.634 (2) and 1.647 (2) Å, respectively (where *Cg*1 and *Cg*2 are the centroids of rings C14–C18 and C19–C23, respectively). The cyclopentadienyl rings of the ferrocene are in an almost

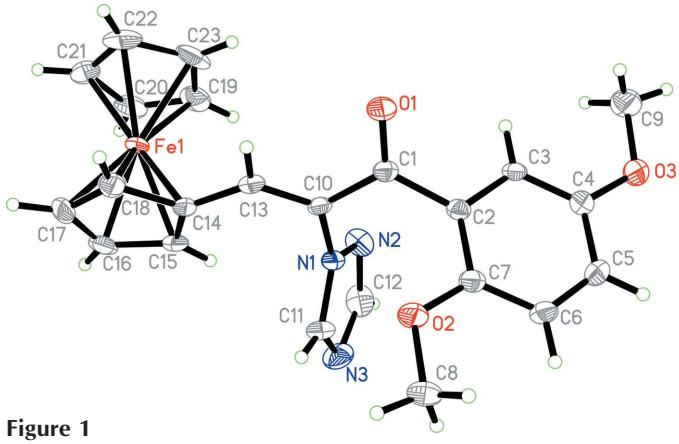


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

eclipsed geometry. While the propene plane from the C14–C18 ring of the ferrocene by a small dihedral angle of 17.9 (5)°, the dimethoxyphenyl and triazole rings are nearly perpendicular to the propene plane, the dihedral angles being 75.9 (2) and 67.9 (2)°, respectively.

Experimental

1-(2,5-Dimethoxyphenyl)-2-(1*H*-1,2,4-triazol-1-yl)-ethanone (10 mmol) and ferrocenecarboxaldehyde (11 mmol) were dissolved in a dry toluene solution (50 ml). To the solution five drops of piperidine and five drops of glacial acetic acid were added at room temperature under a nitrogen atmosphere. The mixture was refluxed for 6 h, during which time the water generated in the reaction was evaporated. The toluene was evaporated under reduced pressure, and the residue was then purified by column chromatography on silica gel with petroleum ether/ethyl acetate (*v*/*v* = 4:1). Recrystallization from a petroleum ether/ethyl acetate solution (*v*/*v* = 3:1) gave single crystals of (I) (yield 70.0%).

Crystal data

$[Fe(C_5H_5)(C_{18}H_{16}N_3O_3)]$
M_r = 443.28
Monoclinic, $P2_1/n$
a = 7.292 (2) Å
b = 20.226 (5) Å
c = 13.956 (4) Å
β = 93.669 (5)°
V = 2054.2 (10) Å^3
Z = 4
D_x = 1.433 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2484 reflections
θ = 2.5–24.3°
μ = 0.76 mm^{-1}
T = 294 (2) K
Block, black
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.826, T_{max} = 0.878
11212 measured reflections
4181 independent reflections
2551 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 26.5°
$h = -8 \rightarrow 9$
$k = -22 \rightarrow 25$
$l = -11 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.129
S = 0.99
4181 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.216 (4)	C1—C10	1.478 (4)
C1—C2	1.489 (4)	C10—C13	1.332 (4)
O1—C1—C10	120.2 (3)	C13—C10—N1	121.7 (3)
O1—C1—C2	120.2 (3)	C13—C10—C1	121.7 (3)
C10—C1—C2	119.5 (3)	N1—C10—C1	116.6 (3)

Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angles refined to fit the electron density, with $U_{iso}(H) = 1.5U_{eq}(C)$. 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*; data reduction: *SAINT* (Bruker,1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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

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