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1-[(*Z*)-3-Ferrocenyl-1-(4-fluorophenyl)-1-methoxyprop-2-en-2-yl]-1*H*-1,2,4-triazole

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
R factor = 0.042
wR factor = 0.099
Data-to-parameter ratio = 13.5

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

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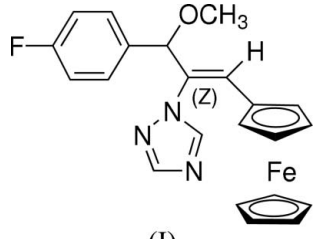
The title compound, $[Fe(C_5H_5)(C_{17}H_{15}FN_3O)]$, was synthesized unexpectedly during a search for potent fungicidal and plant growth regulatory agents. In the crystal structure, the molecules form centrosymmetric dimers *via* weak intermolecular C—H···F interactions. The planes of the substituted cyclopentadienyl and benzene rings make dihedral angles of 93.9 (3) and 15.1 (2)°, respectively, with the triazole ring.

Comment

Ferrocene is stable, non-toxic, can cross cell membranes (Dombrowsk *et al.*, 1986; Yeav, 1969), and its derivatives offer exciting possibilities in insecticide and drug development (Motohashi *et al.*, 1990; Biot *et al.*, 2000). There are many studies reporting that higher activity can be achieved by introducing the ferrocenyl group into biologically active compounds (Huang & Wang, 2001; Molina *et al.*, 1999; Fang *et al.*, 2003). However, the concept of introducing ferrocenyl into triazole derivatives, which possess fungicidal, insecticidal, herbicidal and plant growth regulatory activites (Czollner *et al.*, 1990; Chu *et al.*, 1999), has not received much attention. In order to investigate novel biological triazole molecules containing the ferrocenyl group, we designed and synthesized some 1-aryl-3-ferrocenyl-2-(1*H*-1,2,4-triazole-1-yl)prop-2-en-1-ol derivatives. In this study, the crystal structure of an unexpected product, the title compound, (I), was determined.



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule contains four essentially planar ring systems, *viz*. two cyclopentadienyl rings [*p*1 (C1–C5) and *p*2 (C6–C10)], a triazole ring (*p*3) and a substituted benzene ring (*p*4). The dihedral angles between *p*3 and *p*1, and between *p*3 and *p*4, are 93.9 (3) and 15.1 (2)°, respectively. The Fe—C distances in the ferrocenyl ring are normal and the distance of the cyclopentadienyl ring centroids *Cg*1 (C1–C5) and *Cg*2 (C6–C10) from atom Fe1 are not significantly different [1.638 (2) and 1.650 (3) Å, respectively]. The cyclopentadienyl rings are slightly staggered, as evidenced by the C1···*Cg*1···*Cg*2···C6 and C3···*Cg*1···*Cg*2···C8 pseudo-torsion angles of −12.0 (2) and −14.0 (3)°, respectively.

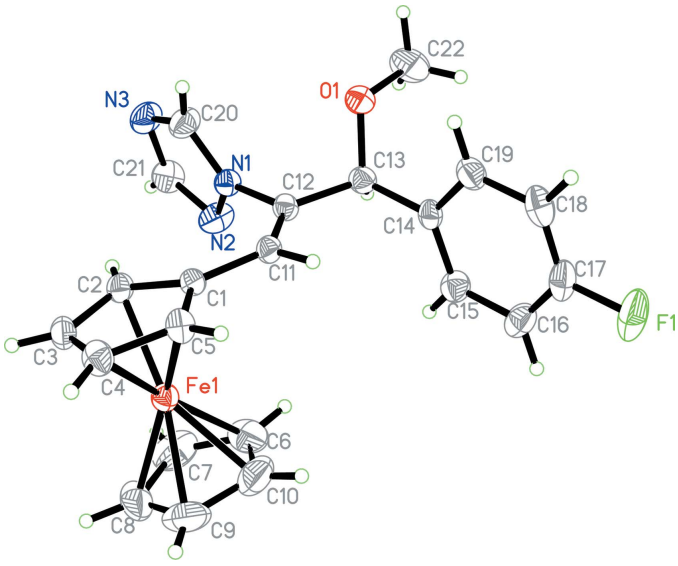


Figure 1
A view of (I), with displacement ellipsoids drawn at the 30% probability level.

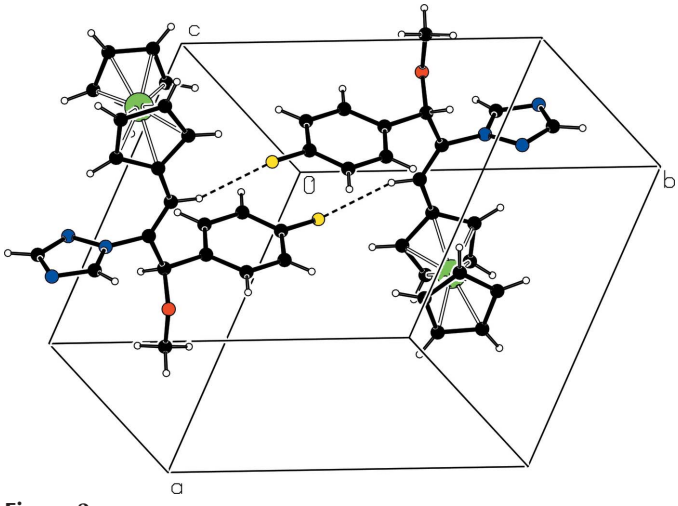


Figure 2
A view (Spek, 2003) of a centrosymmtric dimer of (I). Dashed lines indicate weak C—H···F interactions.

In the crystal structure of (I), weak intermolecular C—H···F interactions [H···F^{i} = 2.511 Å, C···F^{i} = 3.386 (3) Å and C11—H11···F^{i} = 156.8 (2)°; symmetry code: (i) $1-x, 1-y, 2-z$] link the molecules into centrosymmetric dimers (Fig. 2).

Experimental

1-(4-Fluorophenyl)-3-ferrocenyl-2-(1*H*-1,2,4-triazol-1-yl)prop-2-en-1-one (4.2 g, 10 mmol) was dissolved in methanol (15 ml) and water (20 ml). Sodium borohydride (0.076 g, 20 mmol) was then added in six batches below 283 K. The mixture was stirred for 24 h at room temperature, then adjusted to pH 6 using 10% (*w*/*w*) sulfuric acid. The solution was extracted with diethyl ether (3 × 20 ml), and the combined organic layer was washed with water (3 × 20 ml) and then dried over anhydrous magnesium sulfate. After removal of the solvent, the residue was recrystallized from petroleum ether–ethyl acetate (4:1 *v*/*v*) to give yellow crystals of (I) (yield 56%).

Crystal data

$[Fe(C_5H_5)(C_{17}H_{15}FN_3O)]$	$Z = 2$
$M_r = 417.26$	$D_x = 1.421$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 10.116$ (2) Å	Cell parameters from 1579 reflections
$b = 11.045$ (3) Å	$\theta = 2.2$–$22.8°$
$c = 11.239$ (3) Å	$\mu = 0.80$ mm^{-1}
$\alpha = 100.554$ (4)°	$T = 294$ (2) K
$\beta = 110.267$ (4)°	Block, yellow
$\gamma = 115.736$ (4)°	0.24 × 0.22 × 0.18 mm
$V = 975.0$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3417 independent reflections
φ and ω scans	2445 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.025$
$T_{min} = 0.821$, $T_{max} = 0.866$	$\theta_{max} = 25.0°$
4998 measured reflections	$h = -12 \rightarrow 10$
	$k = -7 \rightarrow 13$
	$l = -12 \rightarrow 13$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	$(\Delta/\sigma)_{max} = 0.004$
$S = 1.02$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
3417 reflections	$\Delta\rho_{min} = -0.24$ e Å^{-3}
254 parameters	

Table 1
Selected geometric parameters (Å, °).

N1—C20	1.319 (4)	O1—C22	1.428 (4)
N1—N2	1.360 (3)	C11—C12	1.321 (4)
N1—C12	1.440 (3)	C12—C13	1.510 (4)
N2—C21	1.317 (4)	C13—C14	1.513 (4)
N3—C20	1.311 (4)	C14—C19	1.380 (4)
N3—C21	1.338 (4)	C14—C15	1.383 (4)
O1—C13	1.417 (3)		
C20—N1—N2	108.8 (2)	C11—C12—N1	121.1 (2)
C20—N1—C12	129.2 (3)	C11—C12—C13	126.8 (3)
N2—N1—C12	121.7 (2)	N1—C12—C13	112.2 (2)
C21—N2—N1	101.5 (3)	O1—C13—C12	105.2 (2)
C20—N3—C21	101.3 (3)	O1—C13—C14	112.6 (2)
C13—O1—C22	113.6 (2)	C12—C13—C14	113.9 (2)
C2—C1—C11	130.9 (3)	N3—C20—N1	112.2 (3)
C5—C1—C11	122.5 (3)	N2—C21—N3	116.2 (3)
C20—N1—N2—C21	−0.2 (3)	C12—N1—N2—C21	−175.1 (3)

All H atoms were placed in calculated positions, with C—H = 0.93 Å, or 0.96 Å for methyl H, and included in the refinement using a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl H.

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*.

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