Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[2,6-Dichloro-4-(trifluoromethyl)phenyl]-5-[(2-furyl)methyleneamino]-1*H*-pyrazole-3-carbonitrile

Ping Zhong,* Zhiping Yang,‡ Qian Shi, Maolin Hu, Shuyan Li and Riyuan Tang

Department of Chemistry, Wenzhou Normal College, 325027 Wenzhou, People's Republic of China

‡ Present address: Zhangzhou Vocational and Technical College, 363000 Zhangzhou, People's Republic of China

Correspondence e-mail: zhongp0512@163.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.069
wR factor = 0.204
Data-to-parameter ratio = 12.8

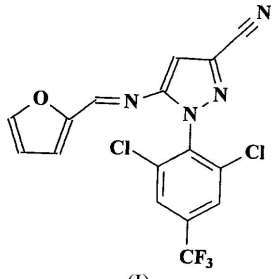
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 28 February 2005
Accepted 11 August 2005
Online 17 August 2005

The title compound, $C_{16}H_7Cl_2F_3N_4O$, is a tricyclic imide with an overall U-shape. There are π–π interactions between the pyrazole and furyl rings.

Comment

The title compound, (I), is an important starting material for the synthesis of 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]- 4-(trifluoromethyl)thiopyrazole, 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethylsulfenyl)pyrazole and 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethylsulfonyl)pyrazole, which are all good insecticides (Hatton *et al.*, 1993).



(I)

The structure of (I) is shown in Fig. 1, with the atom-numbering scheme. The molecule contains three planar moieties, forming an overall U-shape. The dihedral angles between the pyrazole and the furyl and benzene rings are

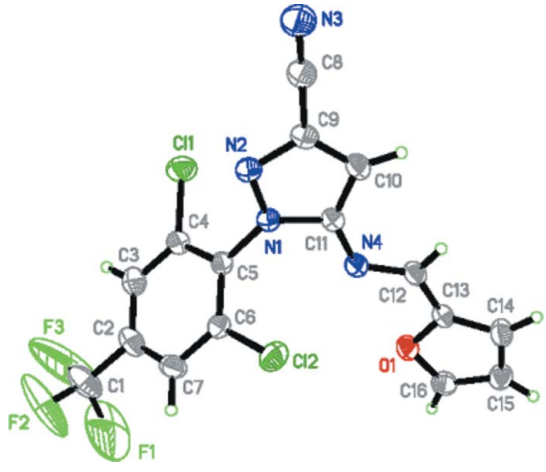


Figure 1
The structure of (I), showing the atomic numbering scheme and displacement ellipsoids drawn at the 50% probability level.

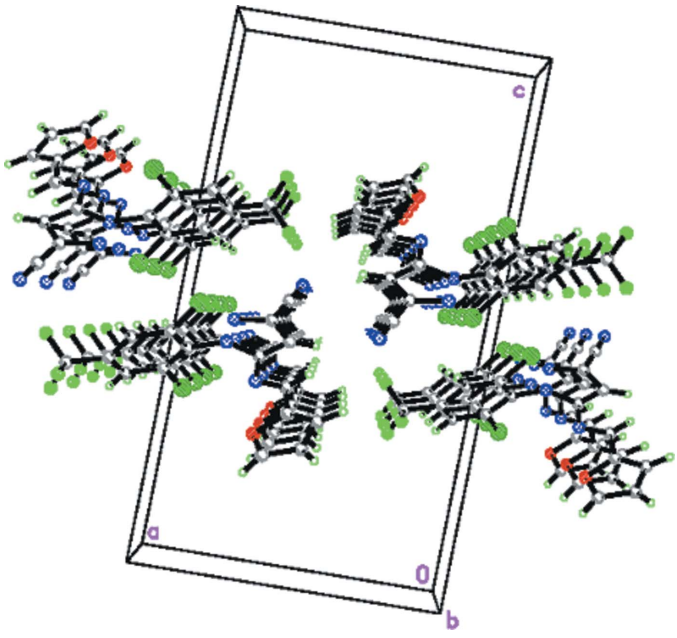


Figure 2
The packing of (I), viewed down the *b* axis.

19.8 (2) and 67.9 (1)°, respectively. The plane-to-plane separation of 3.8411 (1) Å between the furyl and pyrazole rings indicates the presence of a weak π–π interaction. In the crystal structure, the molecules are stacked along the *b* axis, as shown in Fig. 2.

Experimental

Following the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-(trifluoromethyl)amine with a suspension of nitrosyl sulfuric acid, followed by reaction with a solution of ethyl 2,3-dicyanopropionate in acetic acid, gave 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]pyrazole, which was then reacted with 2-furanal to give (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution (m.p. 449–451 K). IR (KBr, ν cm^{-1}): 3129, 2240, 1611, 1558, 1395, 1310, 1133, 873, 818; ^{1}H NMR ($CDCl_3$): δ 8.81 (*s*, 1H), 8.12 (*s*, 2H), 7.83 (*s*, 1H), 7.24 (*m*, 2H), 6.69 (*m*, 1H); ^{13}C NMR ($CDCl_3$): 154.2 (1C), 153.3 (1C), 152.1 (1C), 149.2 (1C), 136.6 (1C), 134.4 (*q*, *J* = 34.3 Hz, 1C), 128.2 (1C), 127.05 (1C), 127.01 (1C), 126.95 (1C), 126.91 (1C), 123.3 (*q*, *J* = 271.6 Hz, 1C), 122.0 (1C), 114.2 (1C), 114.0 (1C), 98.4 (1C).

Crystal data

$C_{16}H_7Cl_2F_3N_4O$
M_r = 399.16
Monoclinic, $P2_1/n$
a = 11.8828 (9) Å
b = 6.7072 (5) Å
c = 21.1191 (16) Å
β = 92.084 (1)°
V = 1682.1 (2) Å^3
Z = 4
D_x = 1.576 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3298 reflections
θ = 3.2–25.0°
μ = 0.43 mm^{-1}
T = 298 (2) K
Block, colorless
0.38 × 0.31 × 0.29 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.854, T_{max} = 0.885
8571 measured reflections
3013 independent reflections
2571 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.2°
$h = -13 \rightarrow 14$
$k = -8 \rightarrow 6$
$l = -24 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.204
S = 1.05
3013 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1164P)^2 + 2.0094P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.724 (4)	N4—C12	1.273 (4)
F1—C1	1.271 (9)	N4—C11	1.382 (4)
O1—C16	1.352 (4)	C9—C10	1.389 (5)
O1—C13	1.362 (4)	C10—C11	1.374 (5)
N1—N2	1.348 (4)	C13—C14	1.350 (5)
N1—C11	1.374 (4)	C14—C15	1.403 (5)
N2—C9	1.337 (5)	C15—C16	1.340 (6)
N3—C8	1.147 (5)		
C16—O1—C13	106.2 (3)	C11—C10—C9	104.9 (3)
N2—N1—C11	113.0 (3)	C10—C11—N1	105.7 (3)
C9—N2—N1	103.2 (3)	C14—C13—O1	109.3 (3)
F3—C1—F2	111.0 (7)	C13—C14—C15	107.6 (3)
N3—C8—C9	179.1 (5)	C16—C15—C14	105.6 (3)
N2—C9—C10	113.1 (3)	C15—C16—O1	111.4 (3)

All H atom were initially observed in a difference Fourier map and were then placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.22$_{eq}$(C). The low U_{eq} value of atom C1 compared with its neighbours may be attributed to the three possibly disordered F atoms. The highest peak is located 1.27 Å from atoms C1 and F.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

This work was supported by the National Nature Science Foundation of China (No. 20272043) and the Nature Science Foundation of Zhejiang Province (No. M203001).

References

Bruker (2002). *SMART*, *SAINT*, *SADABS*, *SHELXL* and *XP*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hatton, L. R., Buntain, I. G., Hawkins, D. W., Parnell, E. W., Pearson C. J. & Roberts, D. A. (1993). *US Patent No.* 5232940.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.