Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Received 31 January 2005
Accepted 21 February 2005
Online 26 February 2005

Key indicators

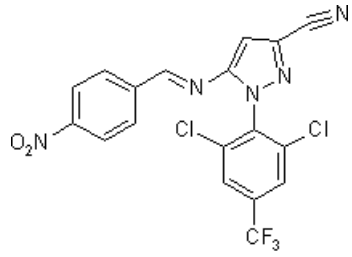
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.162
Data-to-parameter ratio = 11.4

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

The title compound, $C_{18}H_8Cl_2F_3N_5O_2$, is a tricyclic imide with an overall U-shape, each of the the three rings being planar.

Comment

The title compound, (I), is an important starting material for the synthesis of 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-(trifluoromethyl)thiopyrazole, 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-(trifluoromethyl)sulfenylpyrazole and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-(trifluoromethyl)sulfonylpyrazole, 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 groups, forming an overall U-shape, *viz*. a 2,6-dichloro-4-(trifluoromethyl)phenyl group, a pyrazole ring and a 4-nitrophenyl ring. The C—F bond lengths and F—C—F angles are in normal ranges (Hassall & White, 2004), allowing for the possibility of unresolved disorder. The dihedral angles between the pyrazole and the C1–C6 and C12–C17 benzene rings are 28.5 (1) and 74.7 (1)°, respectively. There are π–π interactions between the pyrazole ring and the C1–C6 benzene ring. In the crystal structure, the molecules stack along the *a* axis, as shown in Fig. 2.

Experimental

Following the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-(trifluoromethyl)aniline with a suspension of nitrosylsulfuric 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-trifluoromethylphenyl)pyrazole, which then reacted with 4-nitrobenzaldehyde to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution (m.p. 464–465 K). IR (KBr, ν cm^{-1}): 3133, 2360, 2241, 1620, 1596, 1523, 1345, 1315, 861, 823; ^{1}H NMR ($CDCl_3$): δ 9.26 (*s*, 1H), 8.30 (*d*, J = 8.8 Hz, 2H), 8.14 (*s*, 2H), 8.08 (*d*, J = 8.8 Hz, 2H), 7.39 (*s*, 1H); ^{13}C NMR ($CDCl_3$): δ 166.6 (1C), 152.9 (1C), 141.3 (1C), 137.7 (1C), 136.5 (1C), 134.5 (*q*, J = 34.1 Hz, 1C), 132.4 (2C), 130.1 (1C), 128.4 (2C), 126.8 (2C), 123.8 (2C), 123.3 (*q*, J = 269.6 Hz, 1C), 114.1 (1C), 100.9 (1C).

Crystal data

$C_{18}H_8Cl_2F_3N_5O_2$	$Z = 2$
$M_r = 454.19$	$D_x = 1.561$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.8108$ (8) Å	Cell parameters from 2373 reflections
$b = 11.0797$ (12) Å	$\theta = 1.9$–$25.3°$
$c = 13.3766$ (15) Å	$\mu = 0.39$ mm^{-1}
$\alpha = 75.862$ (2)°	$T = 298$ (2) K
$\beta = 84.841$ (2)°	Block, colorless
$\gamma = 81.296$ (2)°	0.28 × 0.22 × 0.17 mm
$V = 966.09$ (19) Å^3	

Data collection

Bruker SMART APEX area-detector diffractometer	3449 independent reflections
φ and ω scans	2726 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$R_{int} = 0.014$
$T_{min} = 0.902$, $T_{max} = 0.936$	$\theta_{max} = 25.3°$
5202 measured reflections	$h = -7 \rightarrow 8$
	$k = -13 \rightarrow 13$
	$l = -12 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0803P)^2 + 0.4474P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.058$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.162$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.41$ e Å^{-3}
3449 reflections	
303 parameters	
All H-atom parameters refined	

All H atoms were located in a difference Fourier map and were refined freely. The CF_3 group may be subject to unresolved disorder.

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* in *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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

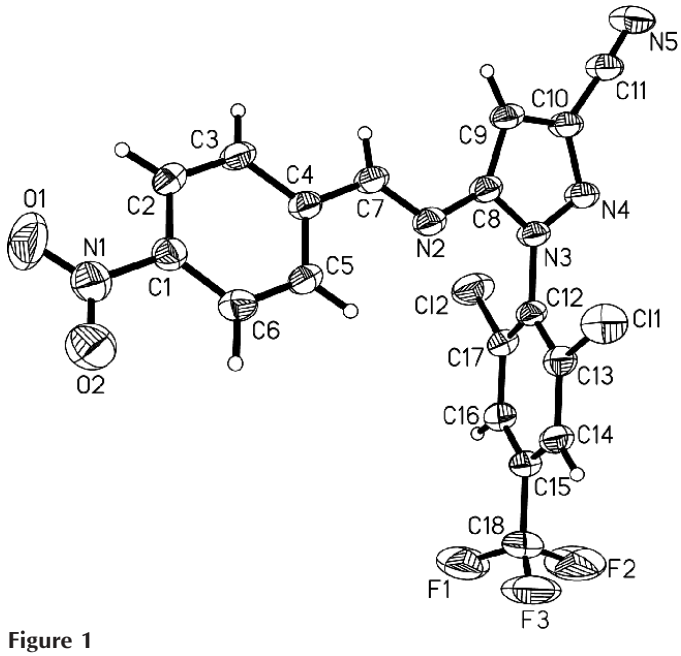


Figure 1
The structure of (I), showing the atomic numbering scheme and displacement ellipsoids drawn at the 50% probability level.

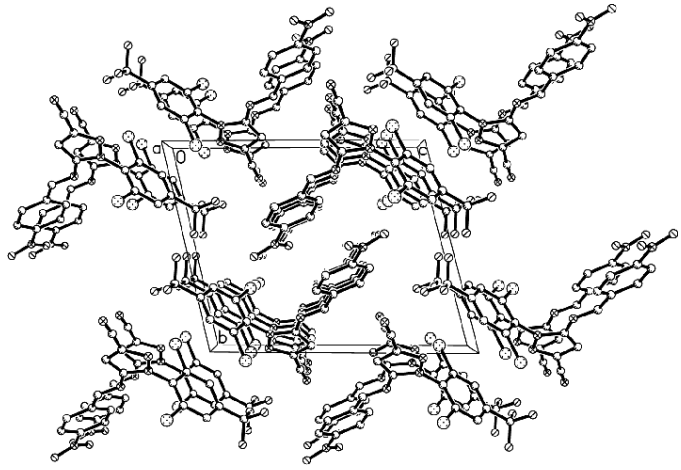


Figure 2
Packing diagram for (I), viewed down the *a* axis.

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