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

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.050
wR factor = 0.120
Data-to-parameter ratio = 11.3

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

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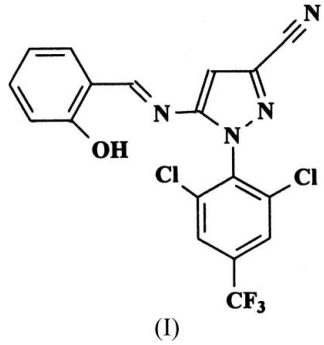
The title compound, $C_{18}H_9Cl_2F_3N_4O$, is a tricyclic amide with an overall U-shaped molecule.

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-(trifluoromethylsulfanyl)pyrazole, 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, all of which are good insecticides (Hatton *et al.*, 1993).



(I)

The structure of (I) is shown in Fig. 1. The molecule contains three planar groups, forming an overall U-shape, *viz*. a 2,6-dichloro-4-(trifluoromethyl)phenyl, a pyrazole and a 2-hydroxyphenyl ring. The dihedral angles between the pyrazole and the C1–C6 and C12–C17 aromatic rings are 17.9 (3) and 64.3 (1)°, respectively. In the crystal structure, the molecules stack along the *c* axis, as shown in Fig. 2.

Experimental

Following the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-trifluoromethylamine 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-(trifluoromethyl)phenyl]pyrazole, which was then reacted with 2-hydroxybenzaldehyde and hydrochloric acid in anhydrous ethanol to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate/petroleum ether (1/2 *v*/*v*) solution (m.p. 445–447 K). IR (KBr, ν cm^{-1}): 3355, 3145, 3064, 2359, 2241, 1606, 1568, 1522, 1313, 887, 860; ^{1}H NMR ($CDCl_3$): δ 11.15 (*s*, 1H), 9.25 (*s*, 1H), 8.19 (*s*, 2H), 7.62 (*d*, J = 7.6 Hz, 1H), 7.50 (*m*, 1H), 7.40 (*s*, 1H), 7.01 (*m*, 1H), 6.88 (*d*, J = 8.3 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 168.4 (1C), 162.1 (1C), 151.6 (1C), 137.3 (1C), 136.6 (1C), 135.2 (1C), 134.3 (1C), 128.5 (2C), 126.3 (2C), 123.3 (*q*, J = 270 Hz, 1C), 121.9 (2C), 117.0 (1C), 114.1 (1C), 100.7 (1C), 99.3 (1C).

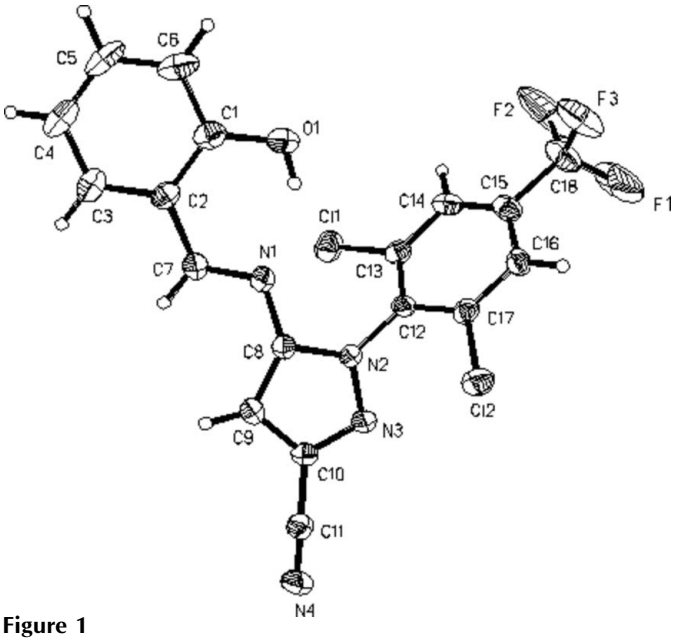


Figure 1
The structure of (I), showing the atomic numbering scheme and displacement ellipsolids at the 50% probability level.

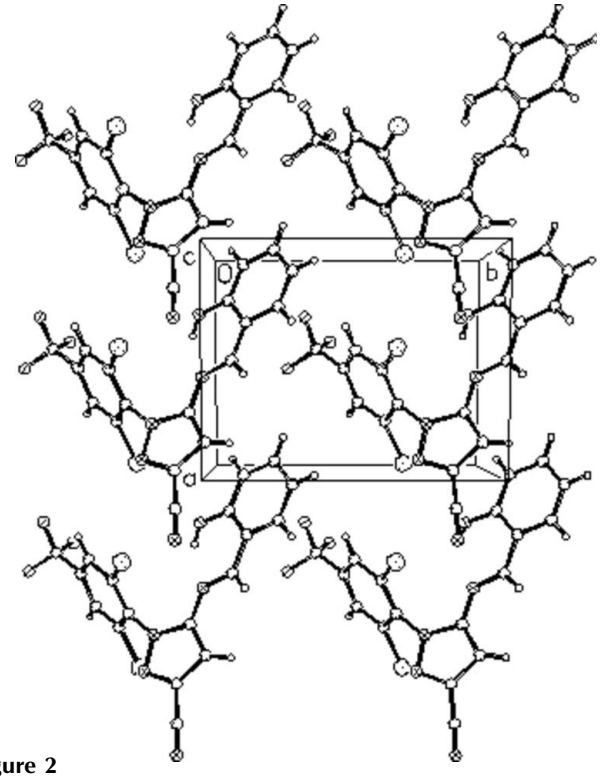


Figure 2
Packing diagram viewed down the *c* axis.

Crystal data

$C_{18}H_9Cl_2F_3N_4O$
M_r = 425.19
Monoclinic, $P2_1$
a = 7.4482 (9) Å
b = 9.3760 (11) Å
c = 13.1365 (16) Å
β = 98.820 (2)°
V = 906.53 (19) Å^3
Z = 2
D_x = 1.558 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3113 reflections
θ = 1.6–25.2°
μ = 0.41 mm^{-1}
T = 298 (2) K
Block, colorless
0.28 × 0.22 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.899, T_{max} = 0.930
4838 measured reflections
2928 independent reflections
2687 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.2°
$h = -8 \rightarrow 7$
$k = -8 \rightarrow 11$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.120
S = 1.04
2928 reflections
258 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 0.4065P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 3100 Friedel pairs
Flack parameter: 0.15 (9)

Table 1
Selected geometric parameters (Å, °).

Cl1—C13	1.728 (4)	N2—N3	1.352 (4)
F1—C18	1.260 (8)	N2—C8	1.372 (5)
F2—C18	1.294 (7)	N2—C12	1.417 (5)
F3—C18	1.290 (6)	N3—C10	1.332 (5)
O1—C1	1.351 (6)	C8—C9	1.372 (6)
N1—C7	1.286 (5)	C9—C10	1.392 (6)
N1—C8	1.385 (5)		
C7—N1—C8	118.1 (4)	C8—C9—C10	104.3 (4)
N3—N2—C8	112.7 (3)	N3—C10—C9	113.5 (3)
N3—N2—C12	119.8 (3)	N3—C10—C11	118.9 (4)
C8—N2—C12	127.3 (3)	C9—C10—C11	127.6 (4)
C10—N3—N2	103.1 (3)	N4—C11—C10	178.4 (5)
C9—C8—N2	106.3 (4)	F1—C18—F3	105.7 (7)
C9—C8—N1	135.1 (4)	F1—C18—F2	106.5 (6)
N2—C8—N1	118.5 (4)	F3—C18—F2	104.0 (5)

The H atom on atom O1 was located in a difference Fourier synthesis and refined with the restraint O—H = 0.82 (1) Å. Other H atoms were positioned geometrically (C—H = 0.96 Å), and allowed to ride on their respective parent C atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$. The highly anisotropic displacements of the F atoms indicate possible disorder, which was not resolved. The absolute configuration of the title compound is unknown and there is no firm chemical evidence for its assignment. Refinement of the Flack (1983) parameter tends to support the current assignment.

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: *SHELXTL* (Bruker, 2002).

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References

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