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5-(3-Bromobenzylideneamino)-4-chloro-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-1*H*-pyrazole-3-carbonitrile

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Key indicators

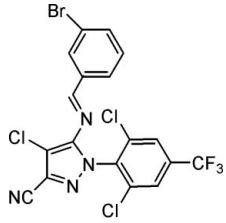
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.058
wR factor = 0.153
Data-to-parameter ratio = 13.8

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

The title compound, $C_{18}H_7BrCl_3F_3N_4$, is a tricyclic imine with an overall Y-shape. The dihedral angles between the pyrazole ring and the two benzene rings are 121.43 (2) and 20.48 (1)°.

Comment

The title compound, (I), is similar to the very effective insecticides used to treat animals such as cows and sheep (Philippe, 1997, 2000) and its structure is reported here (Fig. 1). The molecule contains three essentially planar rings. The dihedral angles between the pyrazole ring and the benzene rings are 121.43 (2)° for C2–C7 and 20.48 (1)° for C12–C17.



(I)

Experimental

According to the method of Zhong *et al.* (2005), using 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]pyrazole (2.5 mmol), followed by reaction with 3-bromobenzaldehyde (2.5 mmol) and hydrochloric acid (2 ml) in anhydrous ethanol (5 ml), we obtained 1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-3-cyano-5-benzylideneamino-1*H*-pyrazole, which was then reacted with sulfinyl dichloride (0.36 ml) in ethyl acetate (7 ml) under ice cooling and a stream of nitrogen (Okui, 2005). After stirring for 1 h, the vessel was left at room temperature with further stirring. The reaction was monitored at intervals by thin-layer chromatography until completion. Saturated aqueous sodium hydrogen carbonate solution (2 ml) was added and the organic layer was washed with saturated aqueous sodium hydrogen carbonate solution (15 ml) and water (15 ml) several times. The organic layer was then purified by silica-gel column chromatography. Removal of the solvent gave the title compound (61.4% yield). Colourless single crystals suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol–acetone (2:1) solution of (I) (m.p. 470–471 K).

Crystal data

$C_{18}H_7BrCl_3F_3N_4$
M_r = 522.54
Triclinic, $P\overline{1}$
a = 7.8683 (8) Å
b = 11.5269 (12) Å
c = 11.8954 (12) Å
α = 92.974 (2)°
β = 107.609 (2)°
γ = 98.142 (2)°
V = 1012.82 (18) Å^3
Z = 2
D_x = 1.713 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.46 mm^{-1}
T = 298 (2) K
Block, colourless
0.21 × 0.17 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.626, T_{max} = 0.756
5472 measured reflections
3614 independent reflections
2463 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.153
S = 1.04
3614 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 0.403P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

All H atoms were initially located in a difference Fourier map but 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) = 1.2U_{eq}(C). The high displacement parameters for the F atoms indicate either large thermal motion or rotational disorder of the trifluoromethyl group. However, attempts to find a suitable disorder model were unsuccessful.

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: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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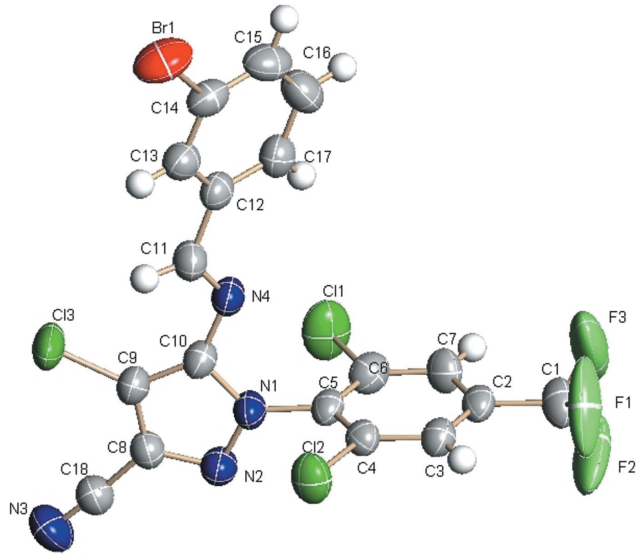

Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

References

Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Okui, S. (2005). US Patent No. 6 849 633.
Philippe, J. (1997). US Patent No. 6 001 384.
Philippe, J. (2000). US Patent No. 6 096 329.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhong, P., Yang, Z. & Shi, Q. (2005). *Acta Cryst.* E**61**, o786–o787.