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1-[2,6-Dichloro-4-(trifluoromethyl)phenyl]-5-(4-methoxyphenylsulfonamido)-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 = 0.078
wR factor = 0.196
Data-to-parameter ratio = 13.0

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

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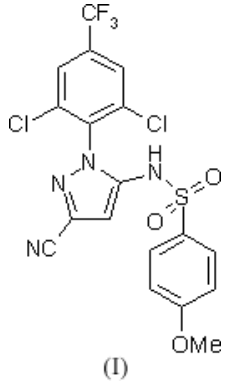
The title molecule, $C_{18}H_{11}Cl_2F_3N_4O_3S$, is a tricyclic amide with an overall Y shape. The dihedral angle between the pyrazole and attached benzene rings is 75.0 (1)°. In the crystal structure, an intermolecular N—H···O hydrogen bond with an $N_{amide}\cdots O_{sulfonyl}$ separation of 2.924 (5) Å links the molecules into linear chains along the *a* axis.

Comment

The title compound, (I), is an intermediate in 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-trifluoromethylsulphenyl-pyrazole and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonyl pyrazole, which are all good insecticides (Hatton *et al.*, 1993). The structure of (I) is shown in Fig. 1. The molecule is a tricyclic amide with an overall Y shape. All bond lengths and angles are normal (Table 1). The dihedral angles between the pyrazole and the C2–C7 and C12–C17 benzene rings are 75.0 (1) and 80.0 (1)°, respectively. In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 2) link the molecules into linear chains along the *a* axis (Fig. 2).



(I)

Experimental

In accordance with the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-trifluoromethylamine (0.01 mol) with a suspension of nitrosylsulfuric acid (0.01 mol), followed by reaction with a solution of ethyl 2,3-dicyanopropionate (0.01 mol) in acetic acid, gave 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole (approximateely 0.005 mol), which was then refluxed with 2-chloro-benzoyl chloride (0.005 mol) and pyridine (5 ml) in chloroform (10 ml) for about 12 h to give the title compound (I) (Xu *et al.*,1999). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an acetone/ethanol (1:1) solution (m.p. 492–493 K). IR (KBr, ν cm^{-1}): 3215, 2247, 1592, 1500, 1380, 1311, 1266, 1166, 1093; ^{1}H NMR ($CDCl_3$, p.p.m.): δ 9.72 (*s*,1H), 8.04 (*s*, 2H), 7.12 (*d*, 4H), 6.85

(*s*, 1H), 3.91 (*s*, 3H); ^{13}C NMR ($CDCl_3$, p.p.m.): δ 164.8 (1C), 140.0 (1C), 137.1 (1C), 135.1 (1C), 134.6 (1C), 132.7 (1C), 131.2 (1C), 130.5 (1C), 128.7 (1C), 128.2 (1C), 127.1 (1C), 125.1 (1C), 121.5 (1C), 115.4(1C), 114.8 (1C), 113.8 (1C), 103.2 (1C), 56.3(1C).

Crystal data

$C_{18}H_{11}Cl_2F_3N_4O_3S$
M_r = 491.27
Triclinic, $P\overline{1}$
a = 9.3444 (9) Å
b = 10.1558 (10) Å
c = 11.6007 (11) Å
α = 99.651 (2)°
β = 100.645 (2)°
γ = 102.014 (2)°
V = 1033.65 (17) Å^3
Z = 2
D_x = 1.578 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1737 reflections
θ = 2.7–24.1°
μ = 0.47 mm^{-1}
T = 298 (2) K
Block, colourless
0.23 × 0.20 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.900, T_{max} = 0.929
5556 measured reflections
3659 independent reflections
2845 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.2°
$h = -11 \rightarrow 8$
$k = -10 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.196
S = 1.08
3659 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0844P)^2 + 1.3536P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.422 (3)	O3—C15	1.353 (6)
S1—O1	1.430 (3)	N1—N2	1.361 (4)
S1—N4	1.642 (4)	N1—C5	1.428 (5)
S1—C12	1.742 (5)	C4—C5	1.386 (6)
F1—C1	1.245 (8)	C10—C11	1.447 (6)
O2—S1—O1	120.2 (2)	C6—C5—N1	120.5 (4)
O2—S1—N4	108.1 (2)	C4—C5—N1	120.3 (4)
N4—S1—C12	106.1 (2)	N1—C8—C9	106.8 (4)
C8—N1—N2	112.9 (3)	N1—C8—N4	120.9 (3)
C8—N1—C5	127.5 (3)	N2—C10—C9	113.7 (4)
C8—N4—S1	121.8 (3)	N2—C10—C11	119.1 (4)
F3—C1—C2	116.6 (5)	C17—C12—S1	119.6 (4)
C3—C2—C1	119.8 (5)	C16—C17—C12	119.9 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O1^{i}	0.86	2.20	2.924 (5)	141

Symmetry code: (i) $-x, -y+1, -z+1$.

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.96 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2–1.5U_{eq}(parent atom). The low U_{eq} value of C1 compared with that of the neighbouring atoms can be attributed to the three disordered F atoms. Attempts to find an appropriate approximation for the disordered CF_3 group failed, resulting in the limited overall precision of the structure.

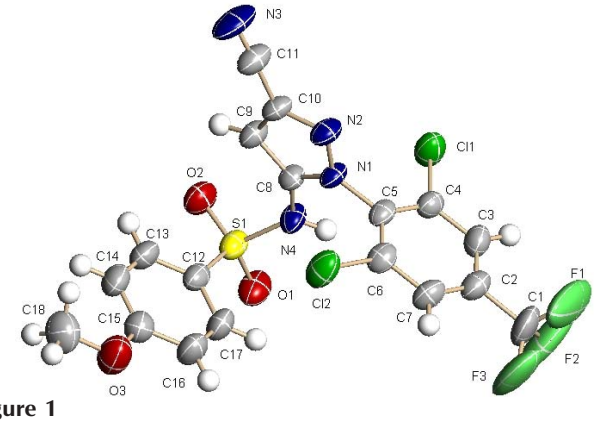


Figure 1
View of (I), with the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

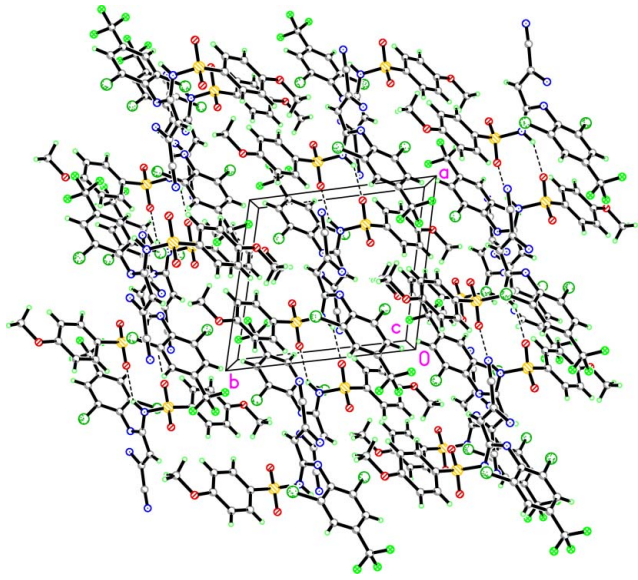


Figure 2
Crystal packing, viewed along the *c* axis. Intermolecular N—H···O hydrogen bonds are shown as dashed lines.

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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