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

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The title compound, $C_{18}H_{11}Cl_2F_3N_4O_2S$, is a tricyclic amide with an overall 'Y' shape. The dihedral angle between the pyrazole and attached benzene rings is 97.6 (2)°. Intermolecular N—H···O hydrogen bonds, with an N(amide)···O(sulfonyl) separation of 2.929 (4) Å, link the molecules into centrosymmetric dimers.

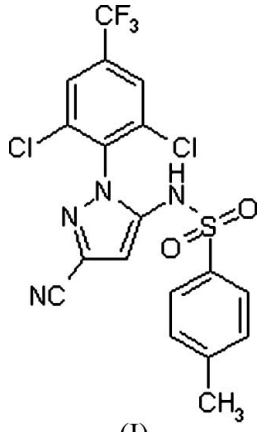
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.067
wR factor = 0.199
Data-to-parameter ratio = 13.2

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

Comment

The title compound, (I), is an intermediate for the synthesis of 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylthiopyrazole, 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulphenylpyrazole and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonylpyrazole, which are all good insecticides (Hatton *et al.*, 1993).



(I)

The structure of (I) is shown in Fig. 1. The molecule has an an overall Y shape, formed by the three groups, *viz*. 2,6-dichloro-4-(trifluoromethyl)phenyl, 4-methylbenzenesulfonyl and a pyrazole ring. The bond lengths and angles are normal (Table 1; Zhang *et al.*, 2005; Zhong *et al.*, 2005). The dihedral angles between the pyrazole and C2–C7 and C12–C17 benzene rings are 97.6 (2) and 74.2 (1)°, respectively.

In the crystal structure, an intermolecular N4—H4···O1 hydrogen bond, with an N(amide)···O(sulfonyl) separation of 2.929 (4) Å (Table 2) links the molecules into centrosymmetric dimers (Fig. 2).

Experimental

Following the method of Hatton *et al.* (1993), the 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 (10 ml), gave 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole (about 0.005 mol), which was then stirred with 4-methylbenzenesulfonyl chloride (0.005 mol) in pyridine (5 ml) at room temperature overnight to give the title compound, (I) (Xu *et al.*, 1999). Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a mixed acetone–ethanol solution (1:1) (m.p. 500–501 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3222, 3091, 2246, 1562, 1509, 1464, 1381, 1309, 1176, 1133, 1027; ^{1}H NMR (CD_3COCD_3, δ, p.p.m.): 9.70 (*s*, 1H), 8.02 (*s*, 2H), 7.73 (*d*, 2H, J = 8.6 Hz), 7.42 (*d*, 2H, J = 8.6 Hz,), 6.85 (*s*, 1H), 2.43 (*s*, 3H); ^{13}C NMR (CD_3COCD_3, δ, p.p.m.): 145.8 (1C), 140.6 (1C), 137.2 (1C), 137.0 (1C), 136.5 (1C), 134.9 (*q*, J = 33.8 Hz, 1C), 130.8 (2C), 128.7 (2C), 128.6 (2C), 127.2 (2C), 123.2 (*q*, J = 271.4 Hz, 1C), 113.8 (1C), 103.6 (1C), 21.5 (1C).

Crystal data

$C_{18}H_{11}Cl_2F_3N_4O_2S$
M_r = 475.27
Triclinic, $P\overline{1}$
a = 9.4636 (10) Å
b = 10.0256 (11) Å
c = 11.6641 (13) Å
α = 102.699 (2)°
β = 102.171 (2)°
γ = 102.984 (2)°
V = 1011.81 (19) Å^3
Z = 2
D_x = 1.560 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2895 reflections
θ = 2.7–25.0°
μ = 0.47 mm^{-1}
T = 298 (2) K
Block, colourless
0.43 × 0.40 × 0.24 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.822, T_{max} = 0.895
5392 measured reflections
3585 independent reflections
3041 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 25.2°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 11$
$l = -13 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.199
S = 1.07
3585 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1091P)^2 + 0.809P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.421 (3)	N1—C5	1.430 (4)
S1—N4	1.640 (3)	N4—C10	1.387 (4)
F1—C1	1.256 (7)	C1—C2	1.485 (5)
N1—C10	1.348 (4)	C4—C5	1.381 (5)
N1—N2	1.359 (4)	C12—C13	1.383 (6)
O2—S1—O1	120.19 (18)	C10—N4—H4	119.1
O2—S1—N4	108.35 (18)	F2—C1—F3	105.7 (6)
O2—S1—C12	108.68 (19)	C4—C5—C6	119.5 (3)
N4—S1—C12	105.57 (17)	N2—C8—C9	113.8 (3)
C10—N1—N2	112.5 (3)	C14—C15—C18	120.2 (5)
C10—N1—C5	127.6 (3)		
Cl1—C4—C5—C6	178.3 (3)	N4—S1—C12—C17	116.4 (3)
N1—C5—C6—Cl2	−1.4 (4)	O2—S1—C12—C13	−178.8 (3)
N2—N1—C10—C9	−1.7 (4)	N4—S1—C12—C13	−62.8 (3)
C5—N1—C10—N4	3.2 (6)	C15—C16—C17—C12	0.8 (7)
O1—S1—C12—C17	−132.8 (3)		

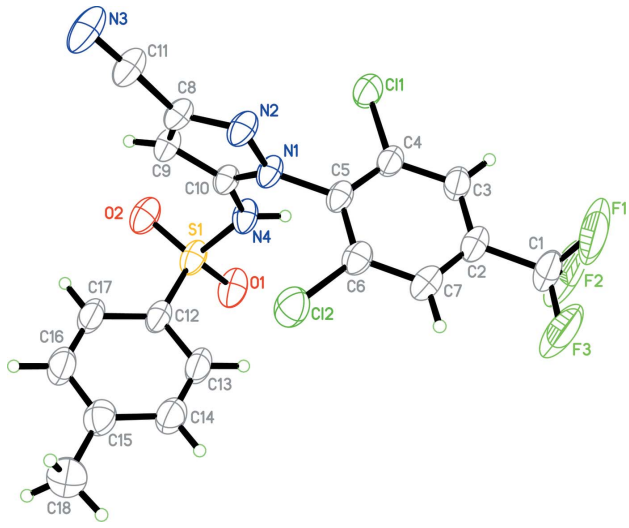


Figure 1
A view of (I), showing the atom-numbering scheme and with displacement ellipsoids at the 50% probability level.

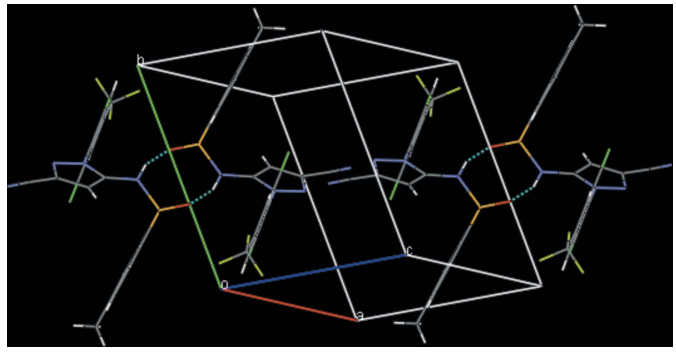


Figure 2
The crystal packing of (I), showing the hydrogen-bonded (dashed lines) dimers.

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O1^{i}	0.86	2.17	2.929 (4)	148

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

The H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å with U_{iso}(H) = 1.2U_{eq}(C), Csp^3—H = 0.96 Å with U_{iso}(H) = 1.5U_{eq}(C), and N—H = 0.86 with U_{iso}(H) = 1.2U_{eq}(N). High values of the displacement parameters for atoms F1, F2 and F3 indicate either large thermal motion or rotational disorder of the trifluoromethyl group. However, attempts to represent the CF_3 group using a disordered model were unsuccessful. The inability to account fully for the electron-density distribution in the vicinity of the CF_3 group is the most likely reason for the rather limited overall precision of the structure.

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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