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

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.135
Data-to-parameter ratio = 13.3

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

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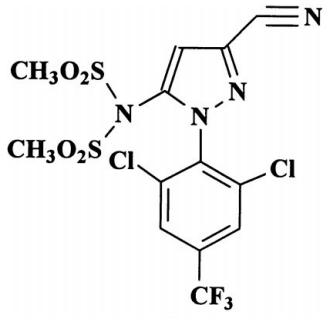
The title compound, $C_{13}H_9Cl_2F_3N_4O_4S_2$, is a bicyclic sulfonated amide with an overall U-shape, each of the two rings being planar.

Comment

The title compound, (I), has been used to synthesize 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethyl)thiopyrazole, 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 these compounds were found to be good insecticides (Hatton *et al.*, 1993).



(I)

The molecular structure of (I) is shown in Fig. 1, and selected geometric parameters are given in Table 1. The molecule has an overall U-shape. The dihedral angle between the mean planes through the pyrazole ring and the benzene ring is 73.2 (1)°.

In the crystal structure of (I), the molecules stack along the *a* axis, as shown in Fig. 2.

Experimental

Following the method of Hatton *et al.* (1993), 2,6-dichloro-4-trifluoromethylamine was reacted with a suspension of nitrosyl sulfuric acid, followed by reaction with a solution of ethyl 2,3-dicyanopropionate in acetic acid. This reaction gave 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]pyrazole, which was then reacted with two equivalents of methanesulfonyl chloride in pyridine to give (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an acetone solution (m.p. 505–507 K). IR (KBr, cm^{-1}): 3146, 3075, 2252, 1537, 1378, 1365, 1319, 1171, 1136; ^{1}H NMR ($CDCl_3$, p.p.m.): 8.12 (*s*, 2H), 7.70 (*s*, 1H), 3.54 (*s*, 6H); ^{13}C NMR ($CDCl_3$, p.p.m.): 137.1 (1 C), 136.6 (1 C), 135.5 (1C), 127.9 (1C), 127.5 (2C), 127.4 (2C), 123.2 (1C), 114.6 (1C), 113.2 (1C), 42.8 (2C).

Crystal data

$C_{13}H_9Cl_2F_3N_4O_4S_2$
M_r = 477.26
Triclinic, $P\overline{1}$
a = 8.4681 (7) Å
b = 8.9652 (8) Å
c = 14.3497 (12) Å
α = 75.198 (2)°
β = 87.918 (1)°
γ = 65.395 (1)°
V = 954.57 (14) Å^3
Z = 2
D_x = 1.660 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2990 reflections
θ = 2.6–25.2°
μ = 0.62 mm^{-1}
T = 298 (2) K
Block, colourless
0.41 × 0.28 × 0.24 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.786, T_{max} = 0.866
5126 measured reflections
3400 independent reflections
3039 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 25.3°
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 7$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.136
S = 1.04
3400 reflections
255 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0723P)^2 + 0.9462P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ <0.001
$\Delta\rho_{max}$ = 1.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.724 (3)	N1—C5	1.427 (3)
S1—N4	1.729 (2)	N2—C10	1.330 (4)
S1—C12	1.757 (3)	N3—C11	1.136 (4)
S2—N4	1.705 (2)	C1—C2	1.496 (5)
S2—C13	1.752 (4)	C8—C9	1.360 (4)
F1—C1	1.254 (5)	C8—N4	1.414 (4)
N1—N2	1.341 (3)	C9—C10	1.398 (4)
N1—C8	1.375 (3)	C10—C11	1.435 (4)
O1—S1—N4	105.04 (12)	C9—C8—N1	106.9 (2)
O1—S1—C12	110.46 (16)	C9—C8—N4	130.4 (2)
N4—S1—C12	102.93 (15)	N1—C8—N4	122.7 (2)
N2—N1—C8	111.8 (2)	C8—C9—C10	104.3 (2)
N2—N1—C5	117.6 (2)	N2—C10—C9	112.7 (3)
C8—N1—C5	130.5 (2)	N2—C10—C11	119.8 (3)
C10—N2—N1	104.2 (2)	C9—C10—C11	127.5 (3)
F1—C1—F3	111.1 (4)	N3—C11—C10	178.6 (4)
F1—C1—C2	113.8 (3)	C8—N4—S2	116.78 (19)
C3—C2—C1	118.3 (3)	S2—N4—S1	117.75 (13)
C4—C3—C2	119.2 (3)		

All H atoms were initially located in a difference Fourier map and were then placed in geometrically idealized position and constrained to ride on their parent atom, with C—H distances in the range 0.93–0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C). Although the F atoms display large displacement parameters, no disorder model could be defined.

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*.

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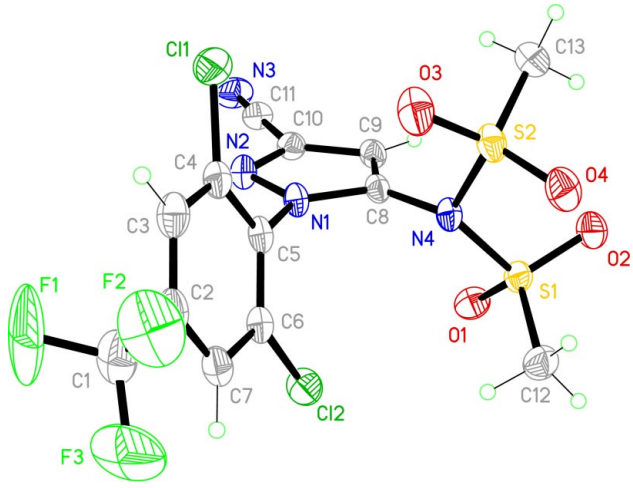

Figure 1
The structure (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

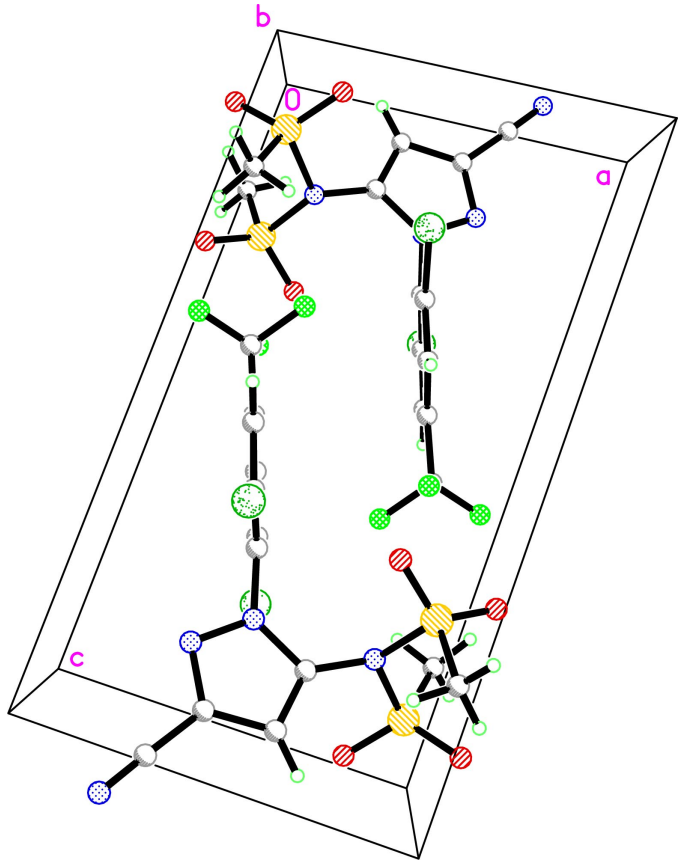

Figure 2
The unit-cell contents for (I), viewed down the *b* axis.

References

Bruker (2002). *SMART*, *SAINT*, *SADABS*, *XP* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hatton, L. R., Bunain, B. G., Hawkins, D. W., Parnell, E. W., Pearson C. J. & Roberts, D. A. (1993). US Patent No. 5232940.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.