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Methyl 5-chloro-2-{[(trifluoromethyl)sulfonyl]-amino}benzoate

Masaharu Kimura[a]* and Shinji Hourai[b]

[a]Organic Synthesis Research Laboratory, Sumitomo Chemical Co. Ltd, Kasugade-naka 3-1-98, Konohana-ku, Osaka 554-8558, Japan, and [b]Environmental Health Science Laboratory, Sumitomo Chemical Co. Ltd, Kasugade-naka 3-1-98, Konohana-ku, Osaka 554-8558, Japan

Correspondence e-mail: kimuram5@sc.sumitomo-chem.co.jp

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.011 Å
R factor = 0.105
wR factor = 0.284
Data-to-parameter ratio = 11.7

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

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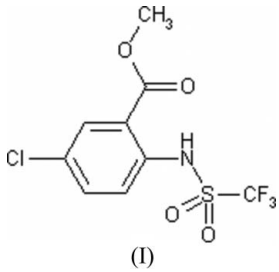
The title compound [systematic name: methyl 5-chloro-2-(trifluoromethylsulfonamido)benzoate], $C_9H_7ClF_3NO_4S$, is a novel acaricide commonly named amidoflumet. The orientations of two side chains are such that the sulfonamide H atom and the carbonyl O atom of the ester substituent are coplanar with the aromatic ring, forming an intramolecular N—H···O hydrogen bond.

Comment

The title compound, amidoflumet, (I) (Fig. 1), is a novel acaricide developed by Sumitomo Chemical Co. Ltd (Mori *et al.*, 2004). Compound (I) dissolves well in several organic solvents (*N*,*N*-dimethylformamide, chloroform, acetonitrile, methanol, ethanol *etc*) but is hardly soluble in water.



(I)

There is an intramolecular hydrogen bond (Table 2) between the sulfonamide H atom and the carbonyl O atom of the ester substituent. The crystal used was a little fragile and structural anisotropy was observed. These characteristics are a consequence of the lack of major intermolecular interactions along the *c* axis (Fig. 2).

Experimental

The analytical standard of (I) used for this study was manufactured and purified by Sumitomo Chemical Co. Ltd. The purity was determined by high-performance liquid chromatography and differential thermal analysis to be 100%. Crystallization was performed by gradual cooling of a supersaturated solution of (I) in chloroform at 300 K after mild heating to dissolve the residue completely. Elemental analysis calculated for $C_9H_7ClF_3NO_4S$: C 34.0, H 2.2, N 4.4, F 17.9, Cl 11.2, S 10.1%; found C 33.9, H 2.4, N 4.2, F 17.8, Cl 11.1, S 10.5%. IR (KBr, cm^{-1}): 2900–3100 (*w*), 1695 (*ms*), 1490 (*ms*), 1310 (*ms*), 1200 (*s*), 1150 (*ms*); ^{1}H NMR (400 MHz, $CDCl_3$, p.p.m.): δ 4.00 (3H, *s*), 7.54 (1H, *dd*), 7.72 (1H, *d*), 8.05 (1H, *d*), 11.2 (1H, *s*); ^{13}C NMR (100 MHz, $CDCl_3$, p.p.m.): δ 53.27, 117.68, 119.63 (splitting to four peaks because of C—F coupling), 120.58, 130.24, 130.95, 134.82, 136.96, 167.55; EI–MS fragmentation: *m*/*z* = 317, 285, 248, 216, 184, 154.

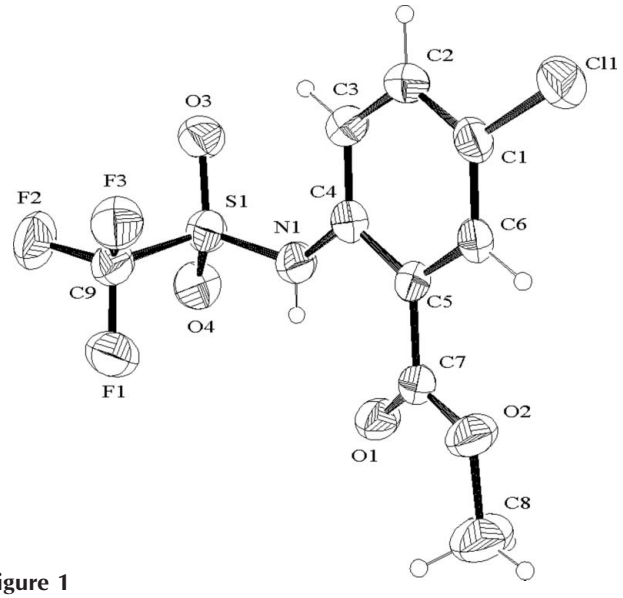


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Crystal data

$C_9H_7ClF_3NO_4S$
M_r = 317.67
Triclinic, $P\overline{1}$
a = 5.341 (2) Å
b = 9.887 (1) Å
c = 11.754 (3) Å
α = 85.212 (9)°
β = 85.47 (1)°
γ = 88.939 (6)°
V = 616.5 (3) Å^3
Z = 2
D_x = 1.711 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 3856 reflections
θ = 3.8–66.8°
μ = 4.84 mm^{-1}
T = 296.1 K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.178, T_{max} = 0.616
12598 measured reflections
2073 independent reflections
1398 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.127
θ_{max} = 68.2°
$h = -6 \rightarrow 6$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.105
$wR(F^2)$ = 0.284
S = 1.06
2073 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1063P)^2 + 1.5441P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.586 (8)	N1—C4	1.42 (1)
O4—S1—O3	122.6 (4)	S1—N1—C4	131.0 (6)
C9—S1—N1	104.1 (4)		
C9—S1—N1—C4	−79.3 (7)	C4—C5—C7—O1	9 (1)
S1—N1—C4—C5	158.8 (7)		

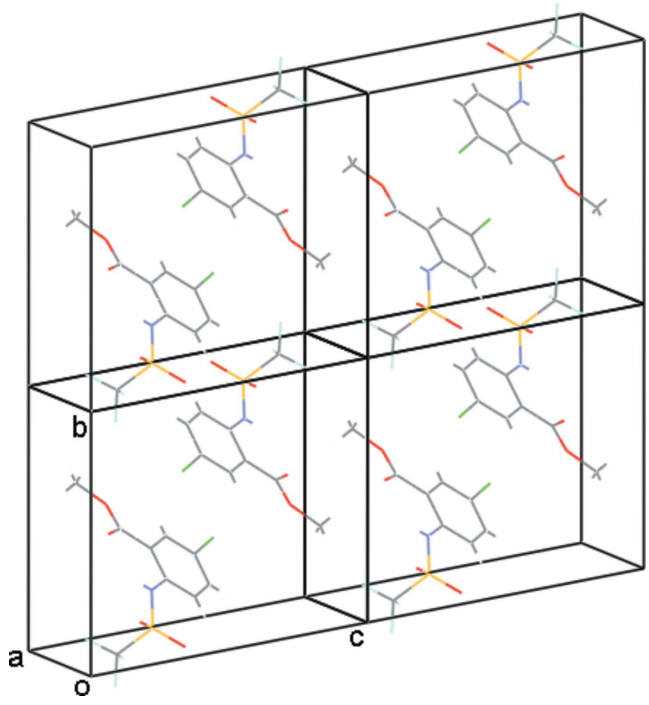


Figure 2
The packing of (I).

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H7···O1	0.78 (8)	2.01 (8)	2.606 (9)	132 (8)

The H atom bonded to the N atom was located in a difference map and refined isotropically. The H atoms bonded to C atoms were positioned geometrically (C—H = 0.95 Å) and refined with riding-model constraints and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

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