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The herbicide flumetsulam as the acetonitrile solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.134
Data-to-parameter ratio = 13.1

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

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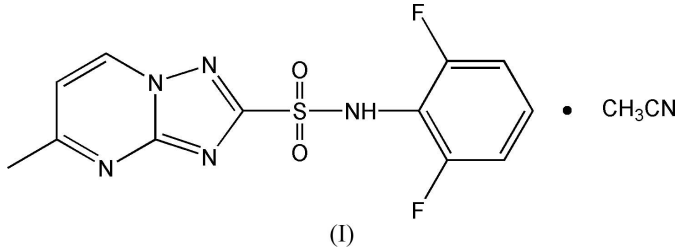
In the crystal structure of the title compound [systematic name: *N*-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo[1,5-*a*]-pyrimidine-2-sulfonamide acetonitrile solvate], $C_{12}H_9F_2N_5O_2S\cdot C_2H_3N$, the planar bicyclic triazolopyrimidine system is bound to the 2,6-difluorobenzene group *via* an SO_2–NH bridge, the C—S—N—C torsion angle being −77.85 (18)°; the dihedral angle formed by the bicyclic triazolopyrimidine system and the benzene ring is 128.0 (2)°. N—H···N bonds link the molecules into infinite chains running along the *a* axis of the crystal structure.

Comment

The title compound, (I) (Fig. 1.), is one of the most important herbicides targeting acetolactate synthase. It has a broad spectrum of activity against broadleaf weeds and exhibits good crop selectivity. In this paper, we report the results of an X-ray diffraction study of flumetsulam, as its acetonitrile solvate.



(I)

The molecular structure of (I) is shown in Fig. 1. The triazolopyrimidine ring system is planar within 0.02 Å. It is bound to the difluorophenyl group *via* a sulfonamide bridge, the C7—S1—N1—C6 torsion angle being −77.85 (18)°. The dihedral angle formed by the mean planes of the triazolopyrimidine and benzene ring systems (C7/N3/C12/N5/N2/N4/C10/C9/C8 and C1–C6) is 128.0 (2)°.

The N4—H4···N9*b* bond [symmetry code: (*b*) 1 + *x*, *y*, *z*] links the molecules of (I) into infinite chains running along the *a* axis of the crystal structure (Table 2 and Fig. 2). There are also a number of π–π interactions in the crystal structure (Fig. 3).

Experimental

Compound (I) was synthesized according to the method of Kleschik *et al.* (1990). Crystals suitable for single-crystal X-ray diffraction were obtained from acetonitrile, by slow evaporation at room temperature. ^{1}H NMR (400 MHz, *d*-DMSO): 2.70 (*s*, 3H), 7.11–7.43 (*m*, 5H), 7.47 (*d*, *J* = 6.8 Hz, 1H), 9.38 (*d*, *J* = 6.8 Hz, 1H), 10.85 (*s*, 1H); MS (EI, *m*/*z*) 325 (M^+). Analysis calculated for $C_{12}H_9F_2N_5O_2S$: C 44.31, H 2.79, N 21.53; found: C 44.73, H 2.60, N 21.14%.

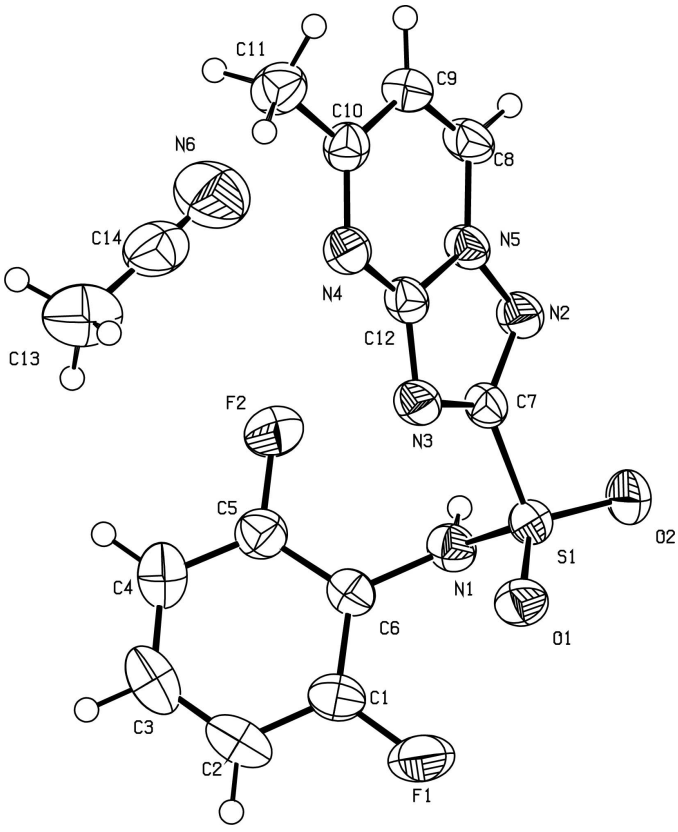


Figure 1
The asymmetric unit of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

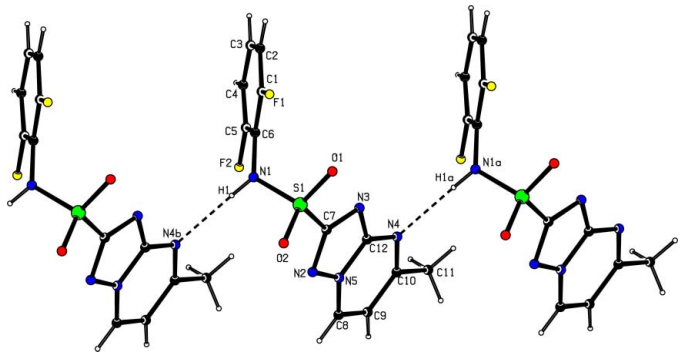


Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (*a*) $-1+x, y, z$; (*b*) $1+x, y, z$.]

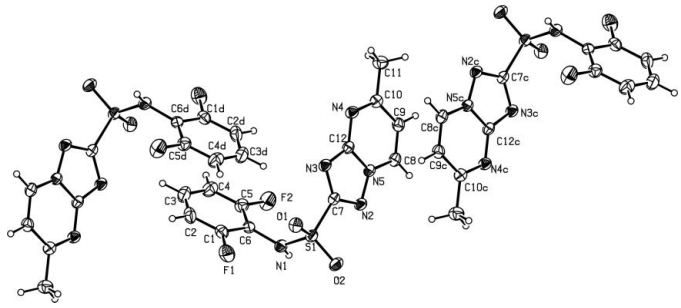


Figure 3
Fragment of the crystal packing of (I), showing the intermolecular π–π stacking interactions. [Symmetry codes: (*c*) $1-x, -y, 1-z$; (*d*) $-x, -y, -z$.]

Crystal data

$C_{12}H_9F_2N_5O_2S\cdot C_2H_3N$
$M_r = 366.36$
Triclinic, $P\overline{1}$
$a = 6.9505\ (7)$ Å
$b = 7.6096\ (7)$ Å
$c = 15.2250\ (15)$ Å
$\alpha = 95.985\ (2)^\circ$
$\beta = 96.095\ (2)^\circ$
$\gamma = 93.712\ (2)^\circ$
$V = 794.00\ (13)$ Å^3
$Z = 2$
$D_x = 1.532$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2570 reflections
$\theta = 2.7$–28.0°
$\mu = 0.25$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
0.40 × 0.40 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.907$, $T_{max} = 0.952$
4334 measured reflections
3042 independent reflections
2784 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 26.0^\circ$
$h = -8 \rightarrow 7$
$k = -9 \rightarrow 8$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.134$
$S = 1.10$
3042 reflections
232 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.2204P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Table 1
Selected torsion angles (°).

C7—S1—N1—C6	−77.85 (18)	N1—S1—C7—N2	−87.74 (17)
S1—N1—C6—C5	108.3 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N4^{i}	0.84 (3)	2.22 (3)	3.056 (2)	178 (2)

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

All H atoms bound to the C atoms were placed in idealized positions [C—H(methyl) = 0.96 Å and C—H(aromatic) = 0.93 Å], and included in the refinement using a riding model, with U_{iso}(methyl H) = $1.5U_{eq}$(C), U_{iso}(aromatic H) = $1.2U_{eq}$(C). Atom H1 bound to atom N1 was located in a difference map and refined isotropically.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); 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, 2001); software used to prepare material for publication: *SHELXTL*.

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