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Methyl 2-(4-methoxypyrimidin-2-yl-carbamoylsulfamoyl)benzoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.119
Data-to-parameter ratio = 14.9

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

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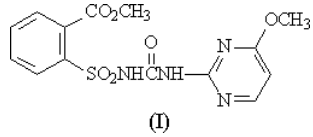
The title compound, $C_{14}H_{14}N_4O_6S$, has a basal plane which contains a urea group, a pyrimidine ring and the S atom. The overall conformation is V-shaped. There exists a pseudo-six-membered ring involving the pyrimidine ring and the urea moiety as a result of an intramolecular N—H···N hydrogen bond. The molecules form dimers *via* an intermolecular N—H···N hydrogen bond and π–π interactions stabilize the crystal structure.

Comment

Sulfonylureas are a successful class of herbicides used worldwide (Levitt, 1991). As part of a study devoted to understanding the reaction mechanisms of sulfonylureas, we have determined the crystal structures of a number of such compounds (Li *et al.*, 1992, 1993, 1994, 1997; Jiang *et al.*, 2000). We have also recently reported the structure of 1-(4-methoxypyrimidin-2-yl)-3-(2-nitrophenylsulfonyl)urea (Ma *et al.*, 2003).



(I)

The title compound, (I), has a basal plane involving the pyrimidine ring, the urea group and the S atom, with a mean deviation of 0.02 Å. The phenyl ring is twisted out of the basal plane, with a dihedral angle of 82.6 (4)°. The orientation of the phenyl ring is defined by the torsion angles N1—S1—C7—C8 −80.1 (2)° and C7—S1—N1—C1 −64.0 (3)° (Table 1); hence the overall conformation of (I) is V-shaped. This is similar to the so-called ε conformer of the sulfonylureas, thromboxane synthase inhibitor (TXSI) and thromboxane receptor antagonist (TXRA), proposed by Michaux *et al.* (2001, 2002). The plane containing atoms C13, O5, O6 and C14 is inclined at 65.1 (5)° to the aryl ring plane, and at 17.7 (3)° to the basal plane. The presence of the intramolecular hydrogen bond N1—H1···N3 means that atoms N3, C2, N2, C1, N1 and H1 form a pseudo-six-membered ring (Table 2), which resulting in the coplanarity of the pyrimidine ring and the urea moiety.

In the crystal structure, the molecules form dimers *via* N2—H2···N4^{i} intermolecular hydrogen bonds [Table 2, symmetry code: (i) $1-x, 1-y, -z$]. Each dimer has π–π interactions between aryl rings with a second dimer [distance 3.764 (3) Å], and π–π interactions between pyrimidine rings with a third dimer [distance 3.747 (4) Å], see Fig. 2. The crystal structure is stabilized by all of these intermolecular hydrogen bonds and the π–π-stacking interactions.

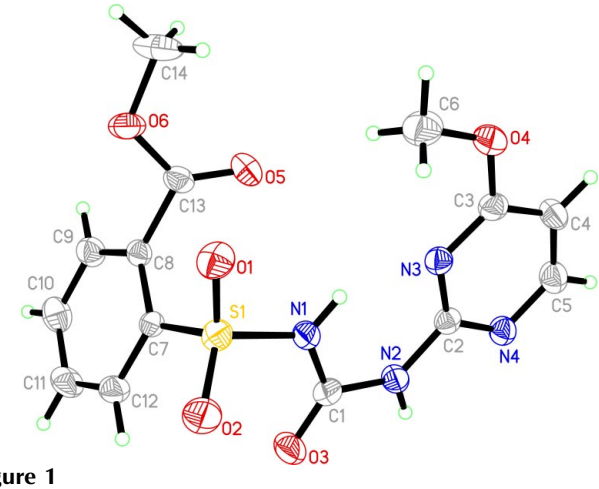


Figure 1
The molecular structure of (I), showing the labelling of all non-H atoms, with displacement ellipsoids at the 30% probability level.

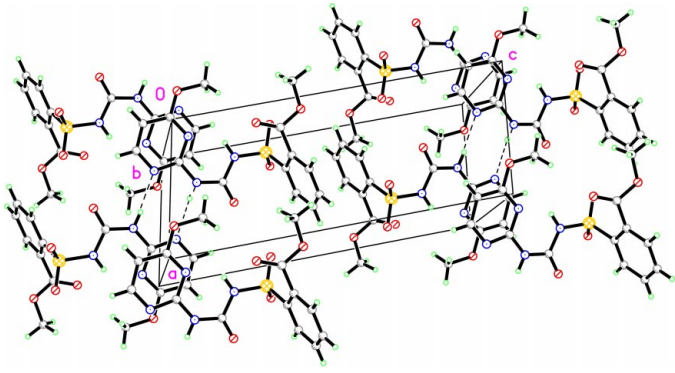


Figure 2
Molecular packing diagram, showing the intermolecular hydrogen bonds (dashed lines) and π–π-stacking interactions.

Experimental

The title compound was synthesized by condensation of 2-methoxycarbonylbenzenesulfonyl isocyanate and 2-amino-4-methoxypyrimidine (Li *et al.*, 1993). Single crystals suitable for crystallographic analysis were obtained by slow evaporation of a 1:1 methanol/acetone solution at room temperature over a period of 15 d.

Crystal data

$C_{14}H_{14}N_4O_6S$
M_r = 366.35
Triclinic, $P\overline{1}$
a = 7.129 (2) Å
b = 7.529 (2) Å
c = 16.371 (5) Å
α = 94.350 (5)°
β = 101.426 (5)°
γ = 100.753 (5)°
V = 840.3 (4) Å^3
Z = 2
D_x = 1.448 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 960 reflections
θ = 2.8–21.6°
μ = 0.23 mm^{-1}
T = 293 (2) K
Prism, colourless
0.36 × 0.32 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.918, T_{max} = 0.955
4849 measured reflections
3413 independent reflections
1931 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.4°
$h = -8 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -18 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.119
S = 1.01
3413 reflections
229 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 0.2733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.424 (2)	N3—C2	1.338 (4)
S1—N1	1.645 (2)	O3—C1	1.207 (4)
S1—C7	1.764 (3)	O4—C3	1.330 (4)
N1—C1	1.382 (4)	O4—C6	1.445 (4)
N2—C1	1.378 (4)	O5—C13	1.197 (4)
N2—C2	1.388 (4)	O6—C13	1.329 (4)
N3—C3	1.325 (4)	C3—C4	1.395 (4)
O2—S1—O1	119.87 (14)	C1—N2—C2	132.0 (3)
O2—S1—N1	109.02 (13)	C3—O4—C6	118.6 (3)
O2—S1—C7	108.33 (15)	O3—C1—N2	120.8 (3)
N1—S1—C7	104.77 (13)	N2—C1—N1	115.4 (3)
C1—N1—S1	122.9 (2)	O5—C13—C8	124.3 (3)
C7—S1—N1—C1	−64.0 (3)	C6—O4—C3—N3	0.7 (4)
S1—N1—C1—N2	175.0 (2)	N1—S1—C7—C8	−80.1 (2)
C1—N2—C2—N4	179.4 (3)	C7—C8—C13—O5	65.6 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3	0.86	2.01	2.690 (6)	136
N2—H2···N4^{i}	0.86	2.11	2.971 (7)	174

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

All H atoms were placed at calculated positions and treated as riding atoms (C—H = 0.93–0.96 Å and N—H = 0.86 Å), with U_{iso} equal to $1.2U_{eq}$ of the parent N or C atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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