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N^4-Acetylsulfamerazine

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.100
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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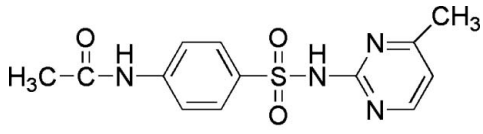
In the title compound [systematic name: 4-acetamido-*N*-(4-methylpyrimidin-2-yl)benzenesulfonamide], $C_{13}H_{14}N_4O_3S$ (NSMZ), which is the acetyl derivative of 4-amino-*N*-(4-methylpyrimidin-2-yl)benzenesulfonamide (sulfamerazine, SMZ), a network of N—H···N and N—H···O interactions results in sheets of molecules in the crystal structure.

Comment

4-Amino-*N*-(4-methylpyrimidin-2-yl)benzenesulfonamide (sulfamerazine, SMZ) is commonly used as an antibacterial agent in veterinary practice. The title compound, N^4-acetylsulfamerazine, NSMZ, (I), is the acetylation metabolite of SMZ (Nouws *et al.*, 1988, 1989). NSMZ is also the precursor of SMZ in the synthetic step before hydrolysis (Roblin & Winneck, 1940). Due to the similarity between the molecular structures of SMZ and NSMZ, NSMZ may substitute for some SMZ molecules in the crystal structure of SMZ and distrupt the hydrogen-bonding interactions with the incoming SMZ molecules. This may explain why a trace amount of NSMZ can stablize the metastable crystalline form of SMZ in an acetonitrile suspension (Gu *et al.*, 2002).



(I)

The fully ordered crystal structure of compound (I) is reported here. It crystallizes in space group *C*2/*c* with one molecule in the asymmetric unit (Fig. 1). In the crystal structure of (I), two NSMZ molecules related by a center of symmetry form a dimer *via* a pair of intermolecular N—H···N hydrogen bonds (Table 1), The dimers are further linked with each other by N—H···O intermolecular hydrogen bonds among the acetylamido groups, forming two dimensional infinite zigzag-shaped sheets parallel to the $(\bar{1}02)$ plane. Such sheets stack to form the whole complete crystal structure with no hydrogen-bonding interactions between them. One of the sheets in shown in Fig. 2.

Experimental

NSMZ was synthesized as suggested by Roblin & Winneck (1940). 1.06 g (9.8 mmol) of 2-amino-4-methylpyrimidine was suspended in 2.5 ml of dry pyridine. 2.38 g (10.2 mmol) of *N*-acetylsulfanilyl chloride was added gradually with stirring. The temperature of the

reaction system was maintained below 328 K. The mixture was then heated on a steam bath for two h. A solution containing 0.44 g (11 mmol) of sodium hydroxide in 2.2 ml of water was added slowly. The pyridine was removed by distillation under reduced pressure. Yellow–brown NSMZ solid was separated by filtration. Crystals of (I) were prepared from a tetrahydrofuran (THF) solution by slow evaporation at room temperature.

Crystal data

$C_{13}H_{14}N_4O_3S$
M_r = 306.34
Monoclinic, $C2/c$
a = 12.2826 (14) Å
b = 9.2479 (11) Å
c = 26.010 (3) Å
β = 95.610 (2)°
V = 2940.2 (6) Å^3
Z = 8
D_x = 1.384 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 173 (2) K
Block, yellow
0.25 × 0.15 × 0.10 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan *SADABS* (Bruker, 2000)
T_{min} = 0.921, T_{max} = 0.980
10158 measured reflections
2596 independent reflections
2046 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.100
S = 1.05
2596 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 2.453P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected torsion angles (°).

C3—N1—C2—C1	−175.4 (2)	S1—N2—C9—N4	4.6 (3)
N2—S1—C6—C5	−107.95 (18)	S1—N2—C9—N3	−175.67 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.8799 (10)	2.228 (11)	3.036 (2)	153 (2)
N2—H2B···N3^{ii}	0.8799 (11)	2.014 (5)	2.885 (3)	170 (2)

Symmetry codes: (i) $-x-\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

The N-bound H atoms were located in difference maps and their positions were refined with the distance restraint N—H = 0.88 (1) Å and U_{iso}(H) = 1.2U_{eq}(N). The C-bound H atoms were placed in calculated positions with C—H = 0.95–0.98 Å and and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: Mercury (Version 1.4.1; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

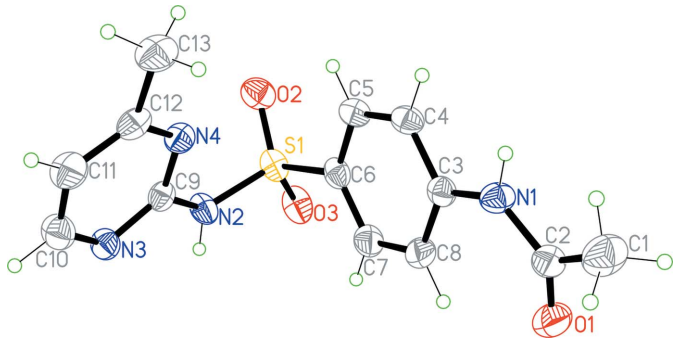


Figure 1
View of (I), showing 50% displacement ellipsoids.

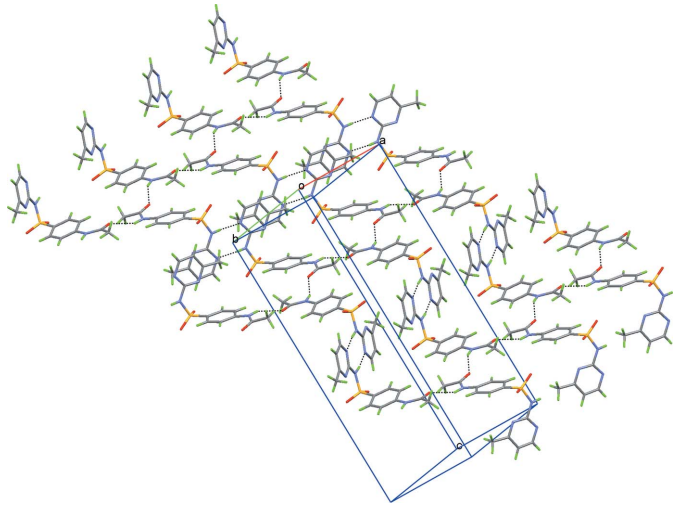


Figure 2
Part of an infinite sheet formed by hydrogen-bonding (dashed lines) interactions in the crystal structure of (I)

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