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A new polymorph of sulfamerazine

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.133
Data-to-parameter ratio = 17.5

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

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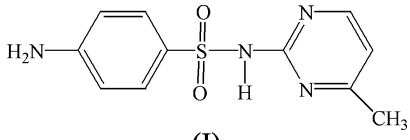
In the title compound, $C_{11}H_{12}N_4O_2S$, molecules are linked by intermolecular N—H···N and O—H···O hydrogen bonds, forming a hydrogen-bonded network.

Comment

Two polymorphs of sulfamerazine were previously determined in the space groups *Pbca* (Acharya *et al.*, 1982) and $Pna2_1$ (Caria & Mohamed, 1992). We have now obtained a new polymorph of sulfamerazine, (I), which crystallizes in the space group $P2_1/c$ and its crystal structure is reported here.



(I)

In the molecule of compound (I) (Fig. 1), the bond lengths and angles (Table 1) are in normal ranges (Allen *et al.*, 1987). The shortening of the C18—N14 [1.364 (3) Å], C15—S11 [1.734 (2) Å] and S11—N11 [1.6530 (19) Å] bonds with respect to the expected single-bond distances are attributed to dπ–pπ interactions, and are comparable with the corresponding values of 1.363 (12), 1.735 (7) and 1.654 (2) Å obtained by Acharya *et al.* (1982), and of 1.357 (7), 1.354 (7), 1.736 (4) and 1.654 (2) Å obtained by Caria & Mohamed (1992). The endocyclic N12—C11—N13 angle of 127.5 (2)° is also comparable with the corresponding values in the other two polymorphs of sulfamerazine; these angles are considerably larger than the value usually observed for a pyrimidine ring.

The planes of the benzene and pyrimidine rings are inclined to each other at 64.39 (2)°, which is comparable with the corresponding values of 71 (1)° (Acharya *et al.*, 1982) and 61.5 (5) and 58.5 (5)° (Caria & Mohamed, 1992) in the other sulfamerazine polymorphs. These indicate that the molecules adopt a *gauche* conformation when viewed along the S—N vector. The tetrahedral geometry around atom S11 is distorted, as evidenced by the deviations of the bond angles around atom S11 atom from 109°.

The crystal structure of (I) is stabilized by intermolecular N—H···N and O—H···O hydrogen bonds (Table 2), which result in the formation of a hydrogen-bonded network (Fig. 2).

Experimental

Solid sulfamerazine was dissolved in dimethylformamide, filtered and left for crystallization by slow evaporation of the solvent at room

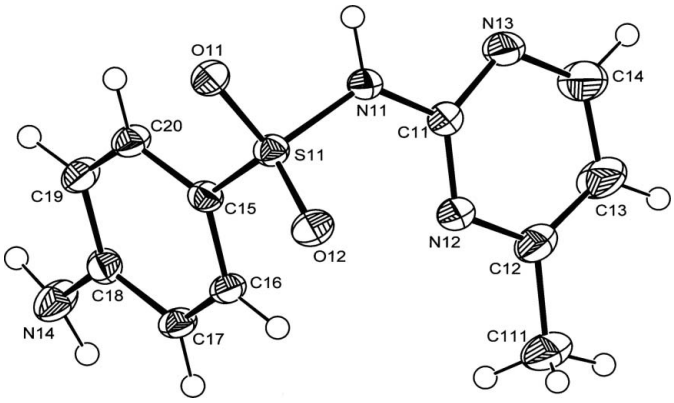


Figure 1
A drawing of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

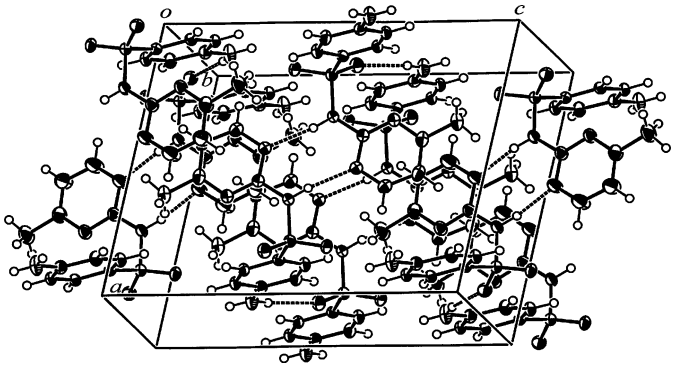


Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

temperature. Colourless block crystals were obtained after two weeks.

Crystal data

$C_{11}H_{12}N_4O_2S$
M_r = 264.31
Monoclinic, $P2_1/c$
a = 11.0966 (5) Å
b = 8.3152 (5) Å
c = 13.9640 (7) Å
β = 99.327 (4)°
V = 1271.43 (11) Å^3
Z = 4
D_x = 1.381 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 150 (2) K
Block, colourless
0.20 × 0.15 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.951, T_{max} = 0.970
11168 measured reflections
2872 independent reflections
2147 reflections with $I > 2\sigma(I)$
R_{int} = 0.091
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.133
S = 1.05
2872 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.8661P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S11—O11	1.4398 (16)	N12—C11	1.327 (3)
S11—O12	1.4293 (17)	N12—C12	1.345 (3)
S11—N11	1.6530 (19)	N13—C11	1.338 (3)
S11—C15	1.734 (2)	N13—C14	1.336 (3)
N11—C11	1.388 (3)	N14—C18	1.364 (3)
O11—S11—O12	119.28 (10)	N11—C11—N12	118.5 (2)
O11—S11—N11	102.31 (9)	N11—C11—N13	114.0 (2)
O12—S11—N11	109.06 (10)	N12—C11—N13	127.5 (2)
O11—S11—C15	109.20 (10)	N12—C12—C13	121.0 (2)
O12—S11—C15	109.54 (10)	N13—C14—C13	122.9 (3)
N11—S11—C15	106.59 (10)	C16—C15—S11	119.56 (17)
C11—N11—S11	126.20 (16)	C20—C15—S11	120.38 (18)
C11—N12—C12	116.2 (2)	N14—C18—C17	120.8 (2)
C11—N13—C14	114.6 (2)	N14—C18—C19	120.4 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11···N13^{i}	0.88	2.08	2.912 (3)	158
N14—H14A···O11^{ii}	0.88	2.43	3.089 (3)	132
N14—H14B···O12^{iii}	0.88	2.14	2.985 (3)	160

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

H atoms were positioned geometrically, with N—H = 0.88 Å (for NH and NH_2) and C—H = 0.95 and 0.98 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,N), where x = 1.5 for methyl H and 1.2 for all other H.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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