Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

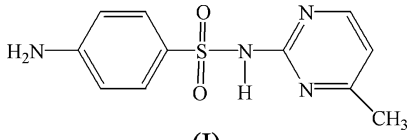
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 April 2006
Accepted 20 April 2006

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

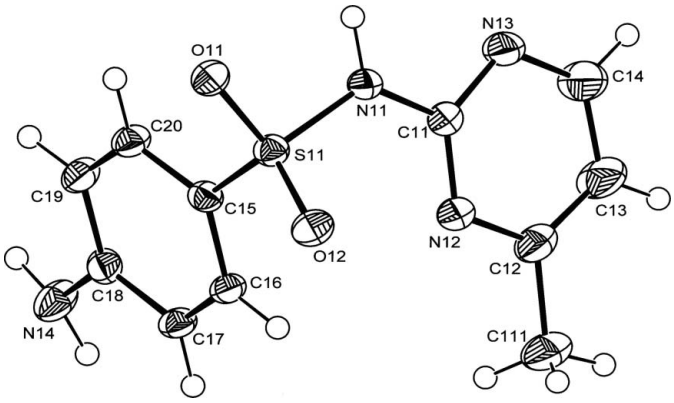


Figure 1
A drawing of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

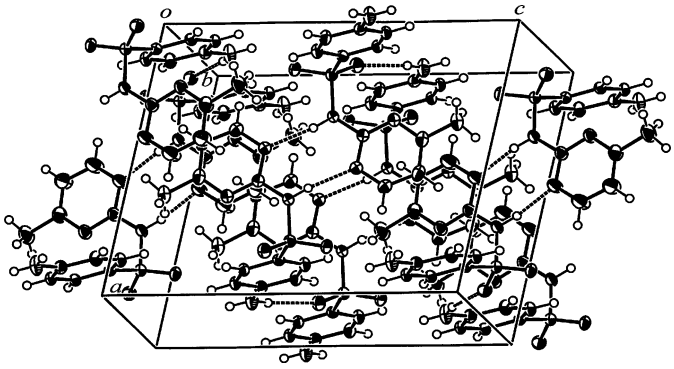


Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

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).

The author acknowledges the Ministry of Science and Technology, Bangladesh Secretariat, Dhaka, Bangladesh, for awarding a Bangabandhu Fellowship.

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supporting information

Acta Cryst. (2006). E**62**, o2166–o2167 [https://doi.org/10.1107/S1600536806014449]

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(I)

Crystal data

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$M_r = 264.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.0966$ (5) Å
$b = 8.3152$ (5) Å
$c = 13.9640$ (7) Å
$\beta = 99.327$ (4)°
$V = 1271.43$ (11) Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.381$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2147 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.26$ mm^{-1}
$T = 150$ K
Block, colourless
0.20 × 0.15 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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 $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 8$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.133$
$S = 1.05$
2872 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S11	0.19021 (5)	0.01361 (7)	0.50270 (4)	0.02192 (18)
O11	0.17220 (14)	−0.0052 (2)	0.39886 (11)	0.0260 (4)
O12	0.13096 (15)	0.1430 (2)	0.54387 (12)	0.0280 (4)
N11	0.33972 (17)	0.0379 (2)	0.52968 (14)	0.0255 (5)
H11	0.3811	0.0369	0.4810	0.031*
N12	0.34604 (18)	0.0793 (3)	0.69574 (14)	0.0287 (5)
N13	0.52733 (19)	0.0576 (3)	0.62466 (15)	0.0413 (6)
N14	0.0937 (2)	−0.6007 (3)	0.68583 (15)	0.0352 (5)
H14A	0.0727	−0.6013	0.7440	0.042*
H14B	0.1019	−0.6920	0.6557	0.042*
C11	0.4063 (2)	0.0597 (3)	0.62188 (17)	0.0265 (5)
C12	0.4146 (2)	0.1024 (3)	0.78327 (18)	0.0361 (6)
C13	0.5399 (3)	0.1057 (5)	0.7940 (2)	0.0559 (10)
H13	0.5885	0.1236	0.8556	0.067*
C14	0.5925 (3)	0.0823 (5)	0.7126 (2)	0.0608 (11)
H14	0.6791	0.0836	0.7191	0.073*
C15	0.15662 (19)	−0.1663 (3)	0.55534 (15)	0.0216 (5)
C16	0.1662 (2)	−0.3099 (3)	0.50627 (16)	0.0252 (5)
H16	0.1877	−0.3083	0.4432	0.030*
C17	0.1446 (2)	−0.4548 (3)	0.54875 (16)	0.0274 (5)
H17	0.1508	−0.5525	0.5146	0.033*
C18	0.1134 (2)	−0.4583 (3)	0.64244 (16)	0.0249 (5)
C19	0.1006 (2)	−0.3120 (3)	0.68991 (16)	0.0236 (5)
H19	0.0767	−0.3126	0.7522	0.028*
C20	0.1221 (2)	−0.1676 (3)	0.64734 (16)	0.0224 (5)
H20	0.1136	−0.0694	0.6803	0.027*
C111	0.3460 (3)	0.1215 (4)	0.8667 (2)	0.0491 (8)
H11A	0.2952	0.2184	0.8571	0.074*
H11B	0.4041	0.1313	0.9273	0.074*
H11C	0.2939	0.0273	0.8703	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S11	0.0216 (3)	0.0251 (3)	0.0192 (3)	0.0007 (2)	0.0036 (2)	0.0005 (2)
O11	0.0286 (9)	0.0323 (10)	0.0164 (8)	−0.0008 (7)	0.0019 (6)	0.0016 (7)
O12	0.0310 (9)	0.0253 (9)	0.0280 (9)	0.0056 (7)	0.0058 (7)	−0.0007 (7)
N11	0.0220 (10)	0.0365 (12)	0.0190 (10)	−0.0048 (9)	0.0066 (8)	−0.0030 (8)
N12	0.0285 (11)	0.0354 (12)	0.0230 (10)	−0.0037 (9)	0.0067 (8)	−0.0046 (9)
N13	0.0234 (11)	0.0737 (18)	0.0272 (11)	−0.0050 (11)	0.0055 (9)	−0.0150 (11)

N14	0.0586 (15)	0.0254 (12)	0.0240 (11)	−0.0072 (10)	0.0140 (10)	−0.0021 (9)
C11	0.0256 (12)	0.0319 (14)	0.0224 (12)	−0.0041 (10)	0.0053 (9)	−0.0041 (10)
C12	0.0384 (15)	0.0452 (17)	0.0249 (13)	−0.0003 (13)	0.0058 (11)	−0.0085 (11)
C13	0.0341 (16)	0.103 (3)	0.0291 (15)	0.0014 (17)	−0.0010 (12)	−0.0241 (16)
C14	0.0264 (15)	0.122 (3)	0.0332 (16)	−0.0059 (17)	0.0021 (12)	−0.0265 (18)
C15	0.0187 (11)	0.0265 (13)	0.0195 (11)	0.0000 (9)	0.0032 (9)	0.0012 (9)
C16	0.0299 (13)	0.0281 (13)	0.0186 (11)	0.0000 (10)	0.0068 (9)	−0.0008 (9)
C17	0.0344 (13)	0.0272 (13)	0.0213 (12)	−0.0008 (11)	0.0063 (10)	−0.0045 (9)
C18	0.0254 (12)	0.0270 (13)	0.0222 (12)	−0.0041 (10)	0.0035 (9)	0.0000 (9)
C19	0.0245 (11)	0.0309 (13)	0.0159 (11)	0.0014 (10)	0.0049 (9)	−0.0009 (9)
C20	0.0220 (11)	0.0276 (13)	0.0182 (11)	0.0023 (10)	0.0049 (9)	−0.0032 (9)
C111	0.0490 (18)	0.073 (2)	0.0278 (15)	−0.0030 (16)	0.0130 (13)	−0.0129 (14)

Geometric parameters (Å, º)

S11—O11	1.4398 (16)	C13—H13	0.9500
S11—O12	1.4293 (17)	C14—H14	0.9500
S11—N11	1.6530 (19)	C15—C16	1.389 (3)
S11—C15	1.734 (2)	C15—C20	1.399 (3)
N11—C11	1.388 (3)	C16—C17	1.381 (3)
N11—H11	0.8800	C16—H16	0.9500
N12—C11	1.327 (3)	C17—C18	1.407 (3)
N12—C12	1.345 (3)	C17—H17	0.9500
N13—C11	1.338 (3)	C18—C19	1.403 (3)
N13—C14	1.336 (3)	C19—C20	1.378 (3)
N14—C18	1.364 (3)	C19—H19	0.9500
N14—H14A	0.8800	C20—H20	0.9500
N14—H14B	0.8800	C111—H11A	0.9800
C12—C13	1.374 (4)	C111—H11B	0.9800
C12—C111	1.500 (4)	C111—H11C	0.9800
C13—C14	1.373 (4)		
O11—S11—O12	119.28 (10)	C13—C14—H14	118.5
O11—S11—N11	102.31 (9)	C16—C15—C20	120.0 (2)
O12—S11—N11	109.06 (10)	C16—C15—S11	119.56 (17)
O11—S11—C15	109.20 (10)	C20—C15—S11	120.38 (18)
O12—S11—C15	109.54 (10)	C15—C16—C17	120.4 (2)
N11—S11—C15	106.59 (10)	C17—C16—H16	119.8
C11—N11—S11	126.20 (16)	C15—C16—H16	119.8
C11—N11—H11	116.9	C16—C17—C18	120.2 (2)
S11—N11—H11	116.9	C16—C17—H17	119.9
C11—N12—C12	116.2 (2)	C18—C17—H17	119.9
C11—N13—C14	114.6 (2)	N14—C18—C17	120.8 (2)
C18—N14—H14A	120.0	N14—C18—C19	120.4 (2)
C18—N14—H14B	120.0	C17—C18—C19	118.7 (2)
H14A—N14—H14B	120.0	C18—C19—C20	120.9 (2)
N11—C11—N12	118.5 (2)	C20—C19—H19	119.6
N11—C11—N13	114.0 (2)	C18—C19—H19	119.6

N12—C11—N13	127.5 (2)	C15—C20—C19	119.7 (2)
N12—C12—C13	121.0 (2)	C19—C20—H20	120.1
N12—C12—C111	115.9 (2)	C15—C20—H20	120.1
C13—C12—C111	123.0 (2)	C12—C111—H11A	109.5
C12—C13—C14	117.7 (3)	C12—C111—H11B	109.5
C12—C13—H13	121.1	H11A—C111—H11B	109.5
C14—C13—H13	121.1	C12—C111—H11C	109.5
N13—C14—C13	122.9 (3)	H11A—C111—H11C	109.5
N13—C14—H14	118.5	H11B—C111—H11C	109.5

Hydrogen-bond geometry (Å, º)

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—H14*A*···O11^{ii}	0.88	2.43	3.089 (3)	132
N14—H14*B*···O12^{iii}	0.88	2.14	2.985 (3)	160

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) *x*, −*y*−1/2, *z*+1/2; (iii) *x*, *y*−1, *z*.