Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Sulfathiazolium nitrate monohydrate

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Received 16 March 2006
Accepted 21 March 2006.

The title compound, $C_9H_{10}N_3O_2S_2^+\cdot NO_3^-\cdot H_2O$, was obtained from a solution of sulfathiazole in dilute nitric acid at room temperature. The crystal structure is stabilized by a network of hydrogen bonds and van der Waals interactions.

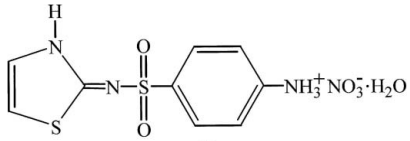
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 14.6

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

Comment

Sulfathiazole has a remarkable solvate-forming ability with interesting structural and conformational properties. Many solvent-containing sulfathiazoles are known and a lot of them have been studied crystallographically (Bingham *et al.*, 2001).



(I)

Shirotani *et al.* (1983) described three solvates of sulfathiazole and Caira *et al.* (1994) reported the crystal structure of the 1:1 complex of sulfathiazole and cyclodextrin, in which the molecules are hydrogen bonded with each other, forming a layer and these layers are linked by hydrogen bonds with water molecules.

The sulfathiazole molecule in the title complex, (I), is hydrogen bonded with a nitrate ion which is also hydrogen bonded with the water molecule. The sulfathiazole molecule is protonated on its terminal amino group.

The planes of the benzene and thiazole rings are inclined in a *gauche* conformation about the S12—N11 bond with a dihedral angle of 87.63 (6)°. The crystal structure is stabilized by a network of hydrogen bonds and van der Waals interactions.

Experimental

Solid sulfathiazole (0.255 g; 1 mmol) was dissolved in 1*M* HNO_3 acid (50 ml) and stirred for 30 minutes, filtered off and the clear solution was left at room temperature for crystallization. Pale-yellow platelike crystals of sulfathiazole nitrate were obtained by slow evaporation of the solution.

Crystal data

$C_9H_{10}N_3O_2S_2^+\cdot NO_3^-\cdot H_2O$
M_r = 336.35
Monoclinic, $P2_1/c$
a = 12.1917 (2) Å
b = 7.6348 (2) Å
c = 15.3895 (2) Å
β = 107.4664 (14)°
V = 1366.43 (5) Å^3
Z = 4
D_x = 1.635 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8182 reflections
θ = 2.9–27.5°
μ = 0.42 mm^{-1}
T = 120 (2) K
Plate, pale yellow
0.34 × 0.32 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (Blessing; 1995)
T_{min} = 0.869, T_{max} = 0.979
17799 measured reflections
3117 independent reflections
2669 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 27.5°
$h = -15 \rightarrow 15$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.05
3117 reflections
214 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.5659P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S11—C11	1.7362 (17)	N13—C17	1.462 (2)
S11—C12	1.732 (2)	C12—C13	1.335 (3)
S12—O11	1.4566 (13)	C14—C15	1.381 (2)
S12—O12	1.4436 (13)	C14—C19	1.398 (2)
S12—N11	1.5824 (15)	C15—C16	1.392 (2)
S12—C14	1.7740 (17)	C16—C17	1.382 (2)
N11—C11	1.344 (2)	C17—C18	1.382 (2)
N12—C11	1.335 (2)	C18—C19	1.384 (3)
N12—C13	1.384 (2)		
C11—S11—C12	90.84 (8)	C13—C12—S11	111.26 (14)
O11—S12—O12	117.06 (8)	C12—C13—N12	112.90 (17)
O11—S12—N11	104.67 (8)	C15—C14—C19	121.33 (15)
O12—S12—N11	114.68 (8)	C15—C14—S12	120.22 (13)
O11—S12—C14	106.67 (8)	C19—C14—S12	118.45 (13)
O12—S12—C14	106.87 (8)	C14—C15—C16	119.52 (15)
N11—S12—C14	106.17 (8)	C15—C16—C17	118.86 (15)
C11—N11—S12	120.76 (13)	C16—C17—C18	121.95 (16)
C11—N12—C13	115.14 (15)	C16—C17—N13	119.15 (15)
N12—C11—N11	119.41 (15)	C18—C17—N13	118.90 (15)
N12—C11—S11	109.85 (12)	C17—C18—C19	119.43 (16)
N11—C11—S11	130.74 (14)	C14—C19—C18	118.90 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13A···O1	0.95 (1)	1.82 (1)	2.765 (2)	177 (2)
O4—H4A···O2	0.95 (1)	1.87 (1)	2.8027 (19)	170 (2)
N12—H12A···O4^{i}	0.95 (1)	2.00 (1)	2.9050 (19)	159 (2)
O4—H4B···O11^{i}	0.95 (1)	1.97 (1)	2.8807 (18)	162 (2)
N13—H13C···O11^{ii}	0.88 (3)	2.03 (3)	2.907 (2)	176 (2)
N13—H13B···O4^{iii}	0.95 (1)	1.91 (1)	2.857 (2)	175 (2)

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

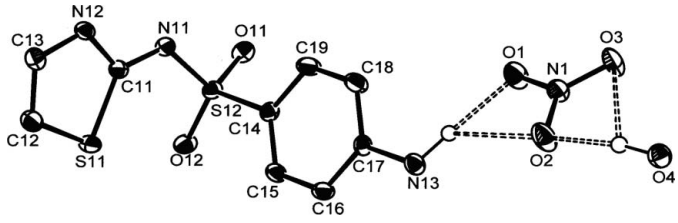


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms not involved in hydrogen bonding (dashed lines) have been omitted.

C-bound H atoms were included in the riding model approximation with C—H = 0.95 Å, and with U_{iso}(H) = 1.2U_{eq}(C). H atoms attached to N and O were located in an electron density map and refined isotropically with the N—H and O—H bond lengths restrained to 0.95 (5) Å.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); 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* publication routines (Farrugia, 1999).

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

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supporting information

Acta Cryst. (2006). E**62**, o1573–o1574 [https://doi.org/10.1107/S1600536806010312]

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Hall symbol: -P 2ybc
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$c = 15.3895\ (2)$ Å
$\beta = 107.466\ (1)°$
$V = 1366.43\ (5)$ Å^3
$Z = 4$
$F(000) = 696$
$D_x = 1.635$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8182 reflections
$\theta = 2.9–27.5°$
$\mu = 0.42$ mm^{-1}
$T = 120$ K
Platelike, pale yellow
0.34 × 0.32 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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Absorption correction: multi-scan Blessing (1995)
$T_{min} = 0.869$, $T_{max} = 0.979$
17799 measured reflections
3117 independent reflections
2669 reflections with $I > 2\sigma(I)$
$R_{int} = 0.081$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -15\rightarrow15$
$k = -9\rightarrow9$
$l = -19\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.106$
$S = 1.05$
3117 reflections
214 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.5659P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ 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	1.03911 (4)	0.33588 (6)	0.62604 (3)	0.02120 (14)
S12	0.80044 (4)	0.12718 (6)	0.62170 (3)	0.01730 (13)
O11	0.70722 (11)	0.07178 (17)	0.65647 (8)	0.0214 (3)
O12	0.90388 (11)	0.02267 (17)	0.64704 (8)	0.0233 (3)
N11	0.81921 (13)	0.32750 (19)	0.64842 (10)	0.0181 (3)
N12	0.91621 (12)	0.5878 (2)	0.64833 (9)	0.0172 (3)
H12A	0.8512 (12)	0.648 (2)	0.6541 (15)	0.028 (6)*
N13	0.61384 (13)	0.1154 (2)	0.21568 (10)	0.0190 (3)
H13A	0.5326 (5)	0.121 (3)	0.2005 (17)	0.039 (7)*
H13B	0.6352 (18)	0.0138 (17)	0.1891 (13)	0.025 (5)*
H13C	0.641 (2)	0.208 (4)	0.1947 (17)	0.041 (7)*
C11	0.91245 (14)	0.4134 (2)	0.64160 (11)	0.0168 (3)
C12	1.09071 (15)	0.5476 (3)	0.62748 (12)	0.0218 (4)
H12	1.1631	0.5768	0.6201	0.026*
C13	1.01555 (15)	0.6644 (2)	0.64025 (12)	0.0203 (4)
H13	1.0289	0.7871	0.6435	0.024*
C14	0.74583 (14)	0.1207 (2)	0.50113 (11)	0.0165 (3)
C15	0.80804 (14)	0.0404 (2)	0.45070 (12)	0.0192 (4)
H15	0.8802	−0.0122	0.4803	0.023*
C16	0.76426 (14)	0.0371 (2)	0.35615 (12)	0.0199 (4)
H16	0.8058	−0.0182	0.3205	0.024*
C17	0.65936 (14)	0.1156 (2)	0.31503 (11)	0.0158 (3)
C18	0.59642 (15)	0.1957 (3)	0.36506 (12)	0.0229 (4)
H18	0.5243	0.2481	0.3351	0.027*
C19	0.63940 (16)	0.1989 (3)	0.45914 (12)	0.0240 (4)
H19	0.5973	0.2534	0.4946	0.029*
O1	0.37657 (11)	0.1342 (2)	0.16636 (9)	0.0291 (3)
O2	0.42291 (11)	0.1643 (2)	0.04127 (9)	0.0319 (4)
O3	0.24360 (11)	0.15191 (19)	0.03616 (9)	0.0283 (3)
N1	0.34639 (13)	0.1506 (2)	0.08093 (10)	0.0206 (3)
O4	0.31305 (11)	0.19590 (17)	−0.14618 (9)	0.0202 (3)
H4A	0.346 (2)	0.198 (3)	−0.0823 (4)	0.047 (7)*
H4B	0.308 (2)	0.3159 (10)	−0.1620 (16)	0.039 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S11	0.0173 (2)	0.0231 (2)	0.0236 (3)	0.00481 (17)	0.00666 (17)	−0.00234 (17)
S12	0.0199 (2)	0.0182 (2)	0.0132 (2)	0.00319 (16)	0.00403 (16)	−0.00013 (15)
O11	0.0271 (7)	0.0204 (6)	0.0189 (6)	−0.0015 (5)	0.0104 (5)	0.0007 (5)

O12	0.0258 (7)	0.0230 (7)	0.0178 (6)	0.0100 (5)	0.0018 (5)	0.0002 (5)
N11	0.0179 (7)	0.0190 (7)	0.0171 (7)	0.0012 (6)	0.0048 (5)	−0.0019 (6)
N12	0.0143 (7)	0.0202 (8)	0.0161 (7)	0.0039 (6)	0.0029 (5)	−0.0001 (6)
N13	0.0157 (7)	0.0260 (8)	0.0149 (7)	0.0007 (6)	0.0038 (6)	0.0015 (6)
C11	0.0152 (8)	0.0218 (9)	0.0115 (8)	0.0040 (7)	0.0013 (6)	−0.0008 (6)
C12	0.0177 (8)	0.0266 (10)	0.0216 (9)	0.0000 (7)	0.0064 (7)	−0.0014 (7)
C13	0.0164 (8)	0.0233 (9)	0.0201 (9)	−0.0010 (7)	0.0036 (7)	0.0000 (7)
C14	0.0185 (8)	0.0176 (8)	0.0128 (8)	0.0008 (6)	0.0037 (6)	0.0000 (6)
C15	0.0150 (8)	0.0236 (9)	0.0176 (8)	0.0046 (7)	0.0027 (6)	0.0006 (7)
C16	0.0181 (8)	0.0246 (9)	0.0183 (8)	0.0044 (7)	0.0077 (6)	−0.0017 (7)
C17	0.0152 (8)	0.0181 (8)	0.0133 (8)	−0.0014 (6)	0.0030 (6)	0.0009 (6)
C18	0.0167 (8)	0.0307 (10)	0.0207 (9)	0.0065 (7)	0.0049 (7)	0.0011 (7)
C19	0.0205 (9)	0.0326 (11)	0.0198 (9)	0.0090 (8)	0.0073 (7)	−0.0011 (7)
O1	0.0212 (7)	0.0483 (9)	0.0166 (7)	0.0021 (6)	0.0041 (5)	0.0039 (6)
O2	0.0167 (6)	0.0584 (10)	0.0217 (7)	−0.0026 (6)	0.0075 (5)	0.0020 (6)
O3	0.0135 (6)	0.0431 (9)	0.0252 (7)	−0.0013 (6)	0.0010 (5)	0.0031 (6)
N1	0.0174 (7)	0.0242 (8)	0.0201 (8)	−0.0004 (6)	0.0053 (6)	0.0008 (6)
O4	0.0189 (6)	0.0212 (7)	0.0205 (6)	−0.0012 (5)	0.0061 (5)	0.0010 (5)

Geometric parameters (Å, º)

S11—C11	1.7362 (17)	C13—H13	0.9500
S11—C12	1.732 (2)	C14—C15	1.381 (2)
S12—O11	1.4566 (13)	C14—C19	1.398 (2)
S12—O12	1.4436 (13)	C15—C16	1.392 (2)
S12—N11	1.5824 (15)	C15—H15	0.9500
S12—C14	1.7740 (17)	C16—C17	1.382 (2)
N11—C11	1.344 (2)	C16—H16	0.9500
N12—C11	1.335 (2)	C17—C18	1.382 (2)
N12—C13	1.384 (2)	C18—C19	1.384 (3)
N12—H12A	0.945 (5)	C18—H18	0.9500
N13—C17	1.462 (2)	C19—H19	0.9500
N13—H13A	0.948 (5)	N1—O1	1.261 (2)
N13—H13B	0.949 (5)	N1—O2	1.2632 (19)
N13—H13C	0.88 (3)	N1—O3	1.236 (2)
C12—C13	1.335 (3)	O4—H4A	0.945 (5)
C12—H12	0.9500	O4—H4B	0.945 (5)
C11—S11—C12	90.84 (8)	C12—C13—H13	123.6
O11—S12—O12	117.06 (8)	N12—C13—H13	123.6
O11—S12—N11	104.67 (8)	C15—C14—C19	121.33 (15)
O12—S12—N11	114.68 (8)	C15—C14—S12	120.22 (13)
O11—S12—C14	106.67 (8)	C19—C14—S12	118.45 (13)
O12—S12—C14	106.87 (8)	C14—C15—C16	119.52 (15)
N11—S12—C14	106.17 (8)	C14—C15—H15	120.2
C11—N11—S12	120.76 (13)	C16—C15—H15	120.2
C11—N12—C13	115.14 (15)	C15—C16—C17	118.86 (15)
C11—N12—H12A	119.0 (13)	C17—C16—H16	120.6

C13—N12—H12A	125.7 (13)	C15—C16—H16	120.6
C17—N13—H13A	107.3 (15)	C16—C17—C18	121.95 (16)
C17—N13—H13B	112.6 (13)	C16—C17—N13	119.15 (15)
H13A—N13—H13B	109 (2)	C18—C17—N13	118.90 (15)
C17—N13—H13C	108.4 (17)	C17—C18—C19	119.43 (16)
H13A—N13—H13C	110 (2)	C17—C18—H18	120.3
H13B—N13—H13C	109 (2)	C19—C18—H18	120.3
N12—C11—N11	119.41 (15)	C14—C19—C18	118.90 (16)
N12—C11—S11	109.85 (12)	C18—C19—H19	120.5
N11—C11—S11	130.74 (14)	C14—C19—H19	120.5
C13—C12—S11	111.26 (14)	O1—N1—O2	119.04 (15)
C13—C12—H12	124.4	O1—N1—O3	120.80 (15)
S11—C12—H12	124.4	O2—N1—O3	120.16 (15)
C12—C13—N12	112.90 (17)	H4A—O4—H4B	103 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N13—H13*A*···O1	0.95 (1)	1.82 (1)	2.765 (2)	177 (2)
O4—H4*A*···O2	0.95 (1)	1.87 (1)	2.8027 (19)	170 (2)
N12—H12*A*···O4^{i}	0.95 (1)	2.00 (1)	2.9050 (19)	159 (2)
O4—H4*B*···O11^{i}	0.95 (1)	1.97 (1)	2.8807 (18)	162 (2)
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Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) −*x*+1, −*y*, −*z*.