

Butylammonium sulfadiazinate monohydrate

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Key indicators

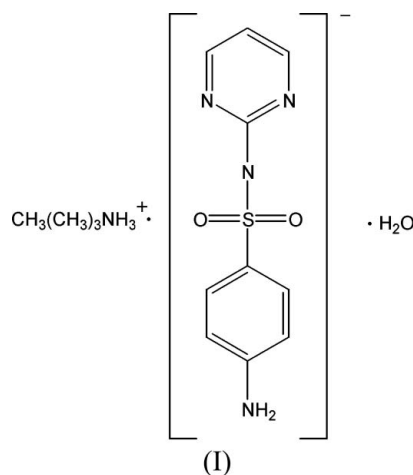
Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.039
 wR factor = 0.106
Data-to-parameter ratio = 13.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_4\text{H}_{12}\text{N}^+ \cdot \text{C}_{10}\text{H}_{11}\text{N}_4\text{O}_2\text{S}^- \cdot \text{H}_2\text{O}$, the bond distances and angles around the S atom correspond to a distorted tetrahedral geometry. The crystal packing is stabilized by $\text{N}-\text{H} \cdots \text{N}$, $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds and a $\pi-\pi$ interaction.

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Comment

The synthesis of metal sulfanilamide compounds has received much attention due to the fact that sulfanilamides were the first effective chemotherapeutic agents to be employed for the prevention and cure of bacterial infections in humans (Nogrady, 1988). Sulfadiazine (2-sulfanilamidopyrimidine, H-SD) compounds are widely used for the treatment of bacterial infections (Silverman, 1992). Sulfadiazine is an approved antibiotic for treating meningococcal meningitis. For people with HIV, it is most commonly used in combination with pyrimethamine for treating toxoplasmosis (Coopman *et al.*, 1993). Silver sulfadiazine is an antibiotic that is active against a wide range of bacteria and it is used to treat and prevent infections at the site of burns (Baenziger & Strauss, 1976; Cook & Turner, 1975). The zinc(II) complex of sulfadiazine is used to prevent bacterial infection in burned animals (Brown *et al.*, 1987). Interesting structural results have been described in the literature (Baenziger *et al.*, 1983; Garcia Raso *et al.*, 1997; Işık *et al.*, 2005). In this paper, we report the crystal structure of the title salt, (I).



The asymmetric unit of the title compound consists of butylammonium and sulfadiazonate ions and a water molecule (Fig. 1). Salt formation occurs by donation of the H atom from the sulfonamide group to the butylamine base. Selected bond distances and angles are given in Table 1. The N4—C8 distance is slightly longer than the previously reported value

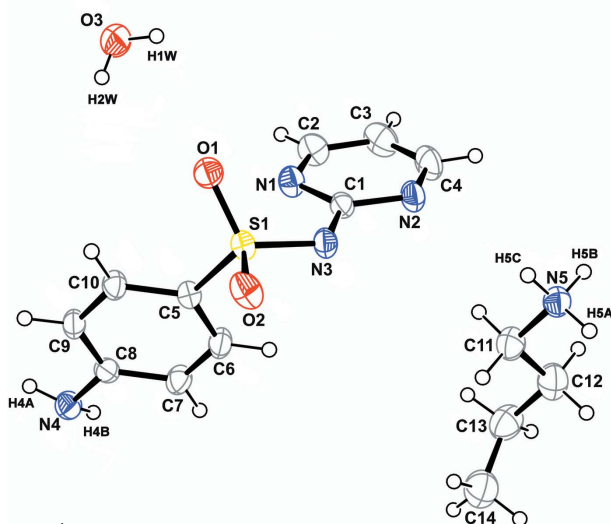


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme, and with displacement ellipsoids drawn at the 50% probability level.

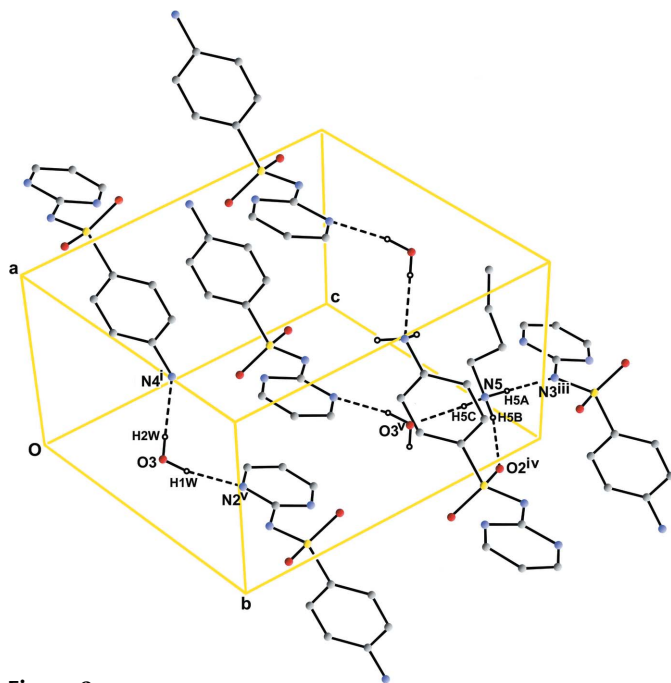


Figure 2
A packing view of (I), showing the hydrogen bonds as dashed lines. H atoms not involved in the hydrogen bonds have been omitted for clarity. Symmetry codes are as given in Table 2.

for sulfadimethoxine (Chung *et al.*, 1975). This is caused by N—H···N, N—H···O and O—H···N hydrogen bonds (Table 2). This value is in agreement with reported values for sulfadiazine and metal sulfadiazinate complexes (Shin *et al.*, 1974; Joshi *et al.*, 1983; Baenziger *et al.*, 1983). In addition, the bond distances and angles in the pyrimidine and benzene rings are consistent with the corresponding values reported for related structures (Shin *et al.*, 1974; Joshi *et al.*, 1983; Baenziger *et al.*, 1983; Marques *et al.*, 2003). The dihedral angle between the pyrimidine and benzene rings is 75.17 (6)°.

The bond distances and angles around the S atom correspond to a distorted tetrahedral geometry. The torsion angle

C5—S1—N3—C1 is slightly greater than that in sulfadimethoxine (Chung *et al.*, 1975). This may be caused by the N5—H5A···N3ⁱⁱⁱ hydrogen bond (symmetry code as in Table 2).

The packing of (I) (Fig. 2) is mainly stabilized by moderate hydrogen bonds (Table 2), and is also supported by an intermolecular π – π interaction, which occurs between pyrimidine rings of neighbouring anions related by an inversion centre. The interplanar and centroid-to-centroid distances are 3.462 (2) Å and 3.478 (2) Å, respectively.

Experimental

A solution of butylamine (2 mmol) in ethanol (20 ml) was added dropwise with stirring to an ethanol solution (25 ml) of sulfadiazine (2 mmol). The mixture was heated to 323 K and stirred for 2 h. The reaction mixture was then cooled to room temperature and the precipitated solid was separated by filtration (yield 70%). Recrystallization from methanol afforded crystals of the pure product, (I), after one week. Analysis, calculated: C 64.37, H 8.81, N 26.82%; found: C 63.98, H 8.76, N 27.06%.

Crystal data

C₄H₁₂N⁺·C₁₀H₉N₄O₂S[−]·H₂O
 $M_r = 341.43$
 Triclinic, $P\bar{1}$
 $a = 7.922$ (2) Å
 $b = 10.082$ (2) Å
 $c = 10.9514$ (18) Å
 $\alpha = 74.557$ (16)°
 $\beta = 82.902$ (17)°
 $\gamma = 85.34$ (2)°

$V = 835.6$ (3) Å³
 $Z = 2$
 $D_x = 1.357$ Mg m^{−3}
 Mo $K\alpha$ radiation
 $\mu = 0.22$ mm^{−1}
 $T = 296$ K
 Prism, colourless
 0.49 × 0.35 × 0.28 mm

Data collection

Stoe IPDS II diffractometer
 ω scans
 Absorption correction: integration
 (X-RED32; Stoe & Cie, 2002)
 $T_{\min} = 0.904$, $T_{\max} = 0.954$

9209 measured reflections
 3292 independent reflections
 2924 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$
 $\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.106$
 $S = 1.04$
 3292 reflections
 236 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.2803P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.37$ e Å^{−3}
 $\Delta\rho_{\text{min}} = -0.39$ e Å^{−3}

H atoms treated by a mixture of independent and constrained refinement

Table 1

Selected geometric parameters (Å, °).

S1—O1	1.4456 (12)	S1—C5	1.7630 (16)
S1—O2	1.4511 (13)	N4—C8	1.397 (2)
S1—N3	1.5791 (14)	C11—N5	1.485 (3)
O1—S1—O2	114.11 (8)	N2—C1—N3	114.32 (14)
O1—S1—N3	114.76 (7)	C7—C8—C9	118.61 (15)
O2—S1—N3	104.54 (8)	C7—C8—N4	120.45 (15)
N1—C1—N2	124.23 (15)	C9—C8—N4	120.94 (15)
N1—C1—N3	121.45 (14)		
C5—S1—N3—C1	68.95 (14)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N4-H4A\cdots N1^i$	0.86 (2)	2.28 (3)	3.127 (2)	169 (2)
$N4-H4B\cdots O1^{ii}$	0.85 (2)	2.34 (2)	3.175 (2)	165 (2)
$N5-H5A\cdots N3^{iii}$	0.95 (2)	1.93 (2)	2.872 (2)	172.8 (19)
$N5-H5B\cdots O2^{iv}$	0.87 (2)	1.99 (3)	2.847 (2)	168 (2)
$N5-H5C\cdots O3^v$	0.90 (3)	1.97 (3)	2.846 (2)	164 (2)
$O3-H1W\cdots N2^v$	0.88 (3)	2.01 (3)	2.877 (2)	168 (2)
$O3-H2W\cdots N4^i$	0.77 (3)	2.18 (3)	2.939 (2)	172 (3)

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x+1, y, z$; (iii) $-x, -y+1, -z+2$; (iv) $x, y+1, z$; (v) $-x, -y+1, -z+1$.

The H atoms attached to atoms O3, N4 and N5 were refined isotropically; refined O–H and N–H bond lengths are in the ranges 0.77 (3)–0.88 (3) and 0.85 (2)–0.95 (2) Å, respectively. The remaining H atoms were placed in geometrically idealized positions, with C–H = 0.93–0.97 Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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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