N	H(N)	O(1^i)	2.79	3.582	143
O(3)	H(O3)	O(2^{ii})	1.67	2.518	176
O(5)	H(O5)	O(1^{iii})	1.61	2.512	160

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

Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974). All H atoms were located in difference Fourier maps and refined isotropically. Data collection and cell refinement: *CAD*-4 (Enraf-Nonius, 1977). Data reduction: *MolEN* (Fair, 1990); *SHELX*76 (Sheldrick, 1976). Program(s) used to solve structure: *SHELX*76. Program(s) used to refine structure: *SHELX*76. Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1987); *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF* in *MolEN* (Fair, 1990).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BK1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(5-acetylamino-1,3,4-thiadiazole-2-sulfon)amide Dihydrate

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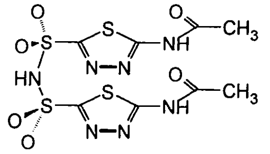
Abstract

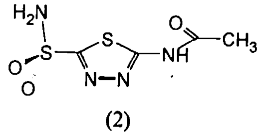
The molecules of the title compound [bis(5-acetylamino-1,3,4-thiadiazole-2-sulfonyl)amine dihydrate, $C_8H_9N_7O_6S_4.2H_2O$ (1)] consist of two nearly planar acetylaminothiadiazolesulfonyl units which are parallel to one another. Coplanarity of the acetylamino groups and the thiadiazole rings is achieved by π-electron delocalization over these groups and by non-bonded S···O interactions [S1···O1 2.667 (4), S1′···O1′ 2.661 (4) Å].

Comment

The title compound (1) was isolated as an impurity in the chemical synthesis of the commercial diuretic known by its generic name of acetazolamide (2) (Roblin & Clapp, 1950).



(1)



(2)

The title compound is a dimer containing two acetazolamide moieties (Fig. 1) and, as in acetazolamide itself (Mathew & Palenik, 1974), the acetylamino group is nearly coplanar with the thiadiazole ring [C2—N3—C3—O1 1.3 (8), C2′—N3′—C3′—O1′ −5.8 (7)°]. This coplanarity is most probably caused by π-electron delocalization over the acetylamino and thiadiazole groups as well as by non-bonded S···O interactions

[S1···O1 2.667 (4), S1′···O1′ 2.661 (4) Å, C1—S1···O1 158.8 (3), C1′—S1′···O1′ 158.6 (3)°] (Kucsman, Kapovits, Párkányi, Argay & Kálmán, 1984). The two acetylaminothiadiazolesulfonyl units are parallel to each other and are linked through the sp^2 NH group [O2—S2—N 115.8 (2), S2—N—S2′ 124.4 (2), N—S2′—O2′ 116.2 (2)°]. The segment O2—S2—NH—S2′—O2′ is nearly planar [O2—S2—N—S2′ −168.1 (3), S2—N—S2′—O2′ 168.8 (3), O2—S2—N—H 11.8 (2), H—N—S2′—O2′ −11.1 (2)°], probably as a result of the participation of the lone pair of the sp^2 N atom in a $d\pi$-$p\pi$ interaction with the S^{VI}—N—S^{VI} moiety. *MM*2 calculations (Allinger & Yuh, 1977) confirmed this to be the most stable molecular conformation.

The intramolecular distances between the S atoms [S1···S2 3.070 (2), S1′···S2′ 3.069 (2), S2···S2′ 2.807 (3) and S1···S1′ 3.552 (3) Å] are slightly shorter than the sum of the corresponding van der Waals radii (3.70 Å).

The molecules are linked together along the *b* axis by the hydrogen bonds N3(—H)···O2′ and N3′(—H)···O2 [2.863 (5) and 3.030 (5) Å, respectively] (Fig. 2). The water molecules participate in the hydrogen bonding but inspection of the maxima in the final difference Fourier map indicates that the H atoms of the two water molecules are disordered. The intermolecular contacts (O1*W*···O1, O1*W*···O3′, O1*W*···N1′, O1*W*···O2*W*, O1*W*···O2*W*′, O2*W*···O1′, O2*W*···O3, O2*W*···N1; Table 3) suggest that five interactions per water molecule can be considered as hydrogen bonds.

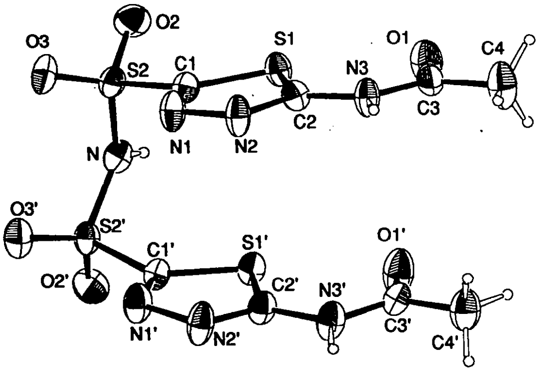


Fig. 1. *ORTEPII* (Johnson, 1976) drawing of the title compound showing the labelling scheme for the non-H atoms. Displacement ellipsoids are drawn at the 48% level.

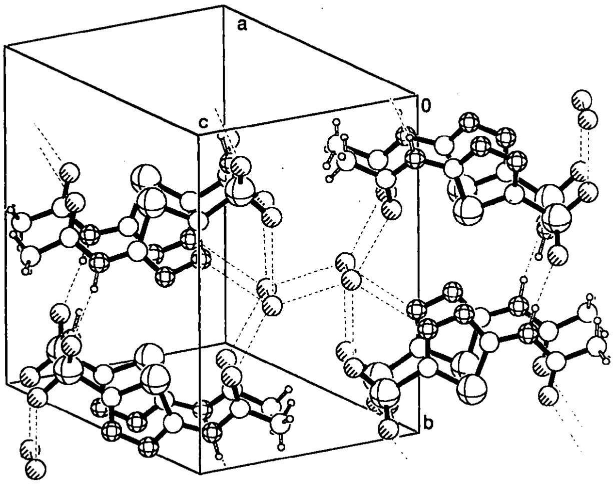


Fig. 2. *PLUTON* (Spek, 1982) drawing of the unit cell of (1). Hydrogen bonds are represented by dashed lines.

Experimental

Crystal data

$C_8H_9N_7O_6S_4.2H_2O$
M_r = 463.47
Monoclinic
$P2_1/a$
a = 16.045 (7) Å
b = 11.868 (4) Å
c = 9.828 (5) Å
β = 104.74 (2)°
V = 1810 (1) Å^3
Z = 4
D_x = 1.70 Mg m^{-3}
D_m = 1.71 Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 18 reflections
θ = 8-10°
μ = 0.556 mm^{-1}
T = 293 K
Irregular
0.46 × 0.23 × 0.11 mm
Colourless

Data collection

Philips PW1100/10 diffractometer
θ-2θ scans
Absorption correction: none
4379 measured reflections
2641 independent reflections
2251 observed reflections [$F > 3.0\sigma(F)$]

$R_{\rm int}$ = 0.034
$\theta_{\rm max}$ = 27.01°
$h = -20 \rightarrow 20$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity variation: 8.8%

Refinement

Refinement on F
R = 0.043
wR = 0.065
S = 0.796
2251 reflections
282 parameters
All H-atom parameters refined except for the disulfonylamine H atom for which only the coordinates were refined

Calculated weights
$w = 1/[\sigma^2(F)+0.00389F^2]$
$(\Delta/\sigma)_{\rm max}$ = 0.053
$\Delta\rho_{\rm max}$ = 0.545 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.387 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S2	0.2244 (1)	0.2209 (1)	0.6460 (1)	0.0290 (3)
S1	0.3751 (1)	0.2415 (1)	0.9094 (1)	0.0299 (3)
O3	0.1872 (2)	0.3002 (3)	0.5376 (3)	0.0385 (9)

O2	0.2782 (2)	0.1323 (3)	0.6136 (4)	0.0408 (12)
O1	0.5024 (2)	0.2281 (3)	1.1439 (4)	0.0439 (12)
N1	0.2786 (3)	0.4071 (3)	0.7943 (4)	0.0350 (13)
N2	0.3358 (2)	0.4523 (3)	0.9108 (4)	0.0351 (12)
N3	0.4500 (3)	0.4031 (3)	1.1001 (4)	0.0338 (12)
C1	0.2928 (3)	0.3000 (4)	0.7815 (4)	0.0301 (13)
C2	0.3882 (3)	0.3762 (3)	0.9786 (5)	0.0283 (14)
C3	0.5045 (3)	0.3265 (4)	1.1789 (5)	0.0344 (14)
C4	0.5666 (4)	0.3717 (5)	1.3081 (6)	0.0528 (21)
N	0.1590 (2)	0.1591 (3)	0.7169 (4)	0.0286 (12)
S2′	0.0754 (1)	0.2164 (1)	0.7460 (1)	0.0272 (3)
S1′	0.1902 (1)	0.2447 (1)	1.0456 (1)	0.0291 (3)
O3′	0.0301 (2)	0.2935 (3)	0.6415 (3)	0.0397 (11)
O2′	0.0262 (2)	0.1258 (3)	0.7845 (4)	0.0403 (12)
O1′	0.3091 (3)	0.2462 (3)	1.2901 (4)	0.0502 (14)
N1′	0.0923 (3)	0.4007 (3)	0.9096 (4)	0.0374 (12)
N2′	0.1342 (3)	0.4487 (3)	1.0364 (4)	0.0409 (13)
N3′	0.2382 (3)	0.4098 (3)	1.2443 (4)	0.0369 (13)
C1′	0.1153 (3)	0.2965 (3)	0.9014 (4)	0.0270 (13)
C2′	0.1873 (3)	0.3779 (4)	1.1151 (4)	0.0309 (14)
C3′	0.3001 (3)	0.3427 (4)	1.3245 (4)	0.0315 (13)
C4′	0.3559 (4)	0.3966 (5)	1.4531 (6)	0.0458 (18)
O1*W*	0.4700 (2)	−0.0319 (3)	0.6934 (4)	0.0469 (13)
O2*W*	0.3768 (3)	0.0355 (4)	0.4153 (4)	0.0617 (16)

Table 2. *Selected geometric parameters* (Å, °)

S2—N	1.580 (4)	N—S2′	1.593 (4)
S2—O3	1.433 (3)	S2′—O3′	1.429 (3)
S2—O2	1.446 (4)	S2′—O2′	1.440 (4)
S2—C1	1.765 (4)	S2′—C1′	1.774 (4)
S1—C1	1.721 (4)	S1′—C1′	1.722 (4)
S1—C2	1.729 (4)	S1′—C2′	1.727 (5)
O1—C3	1.216 (6)	O1′—C3′	1.213 (6)
N1—N2	1.381 (5)	N1′—N2′	1.380 (5)
N1—C1	1.303 (6)	N1′—C1′	1.299 (5)
N2—C2	1.297 (5)	N2′—C2′	1.302 (6)
N3—C2	1.382 (6)	N3′—C2′	1.377 (5)
N3—C3	1.359 (6)	N3′—C3′	1.358 (6)
C3—C4	1.500 (7)	C3′—C4′	1.494 (7)
C1—S2—N	105.6 (2)	N—S2′—C1′	104.3 (2)
O2—S2—N	115.8 (2)	N—S2′—O2′	116.2 (2)
O3—S2—N	105.1 (2)	N—S2′—O3′	105.7 (2)
O3—S2—C1	105.2 (2)	O3′—S2′—C1′	106.0 (2)
O2—S2—C1	105.8 (2)	O2′—S2′—C1′	106.4 (2)
O2—S2—O3	118.2 (2)	O2′—S2′—O3′	117.0 (2)
C1—S1—C2	84.7 (2)	C1′—S1′—C2′	85.1 (2)
N2—N1—C1	111.3 (4)	N2′—N1′—C1′	111.6 (4)
N1—N2—C2	111.3 (4)	N1′—N2′—C2′	111.4 (4)
C2—N3—C3	123.7 (4)	C2′—N3′—C3′	123.3 (4)
S1—C1—N1	116.3 (3)	S1′—C1′—N1′	116.0 (3)
S2—C1—N1	120.1 (4)	S2′—C1′—N1′	121.2 (3)
S2—C1—S1	123.4 (3)	S2′—C1′—S1′	122.7 (2)
N2—C2—N3	120.8 (4)	N2′—C2′—N3′	121.0 (4)
S1—C2—N3	122.8 (3)	S1′—C2′—N3′	123.2 (4)
S1—C2—N2	116.4 (3)	S1′—C2′—N2′	115.8 (3)
O1—C3—N3	120.9 (5)	O1′—C3′—N3′	120.9 (4)
N3—C3—C4	115.8 (4)	N3′—C3′—C4′	115.6 (4)
O1—C3—C4	123.2 (5)	O1′—C3′—C4′	123.5 (5)
S2—N—S2′	124.4 (2)		

Table 3. *Contact distances* (Å)

S1···O1	2.667 (4)	O1*W*···O1$^{\rm ii}$	2.795 (5)
S1′···O1′	2.661 (4)	O1*W*···O3′$^{\rm iii}$	3.073 (5)
S1···S2	3.070 (2)	O1*W*···N1′$^{\rm iii}$	2.944 (5)
S2···S2′	2.807 (3)	O1*W*···O2*W*	2.875 (5)
S1···S1′	3.552 (3)	O1*W*···O2*W*$^{\rm iv}$	2.919 (6)
S1′···S2′	3.069 (2)	O2*W*···O1′$^{\rm v}$	2.875 (6)
N3′···O2$^{\rm i}$	3.030 (5)	O2*W*···O3$^{\rm vi}$	3.051 (6)
N3···O2′$^{\rm i}$	2.863 (5)	O2*W*···N1$^{\rm vi}$	3.189 (6)

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

Data collection: Philips PW1100/10 software. Cell refinement: Philips PW1100/10 software. Data reduction: *STRUFA* (*ZOAK7*; Vicković, 1975). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTON* (Spek, 1982), *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Nitrobiphenyl and 2,2′-Dinitrobiphenyl

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Abstract

2-Nitrobiphenyl, $C_{12}H_9NO_2$, and 2,2′-dinitrobiphenyl, $C_{12}H_8N_2O_4$, are not planar. The molecule of 2,2′-dinitrobiphenyl is located on a crystallographic twofold