(Molecular Structure Corporation, 1985) system. Molecular graphics were obtained using *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1131). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Methyl-4*H*-pyrido[3,2-*e*][1,2,4]thiadiazine 1,1-Dioxide

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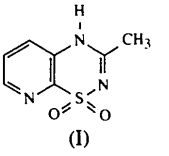
(*Received* 12 *September* 1994; *accepted* 23 *November* 1994)

Abstract

The title compound, $C_7H_7N_3O_2S$, was prepared for comparison with diazoxide, an antihypertensive agent, from a structural and pharmacological point of view. The crystal structure determination shows that the 4*H* tautomeric (rather than the 2*H*) form is preferentially adopted by this pyridothiadiazine derivative in the solid state.

Comment

The present structure, (I), is representative of a new class of heterocyclic compound, the 4*H*-pyrido[3,2-*e*][1,2,4]thiadiazine 1,1-dioxides, and was prepared for comparison with diazoxide from a structural and pharmacological point of view.



(I)

Diazoxide [7-chloro-3-methyl-2*H*(or 4*H*)-1,2,4-benzothiadiazine 1,1-dioxide] is a well known antihypertensive agent currently reported as the pharmacological reference compound for the benzothiadiazine class of ATP-sensitive potassium-channel openers (Edwards & Weston, 1990). The X-ray determination of the title compound may help in the identification of the preferential tautomeric form (the 2*H* or 4*H* tautomer) adopted by this pyridothiadiazine derivative in the solid state.

There are two independent molecules in the asymmetric unit. Molecules are linked by hydrogen bonds: N4—H4···N9^{i} [(i) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$] with N4···N9^{i} 3.089 (5), H4···N9^{i} 2.27 (5) Å, N4—H4···N9^{i} 159.0 (2)° and N24—H24···N29^{ii} [(ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$] with N24···N29^{ii} 3.068 (5), H24···N29^{ii} 2.23 (5) Å, N24—H24···N29^{ii} 166.3 (2)°.

According to the bond lengths of N2—C3 and N4—C3 (N22—C23, N24—C23 in molecule *B*) and the hydrogen-bonding network, the 4*H* tautomeric form seems to be predominant in the crystal.

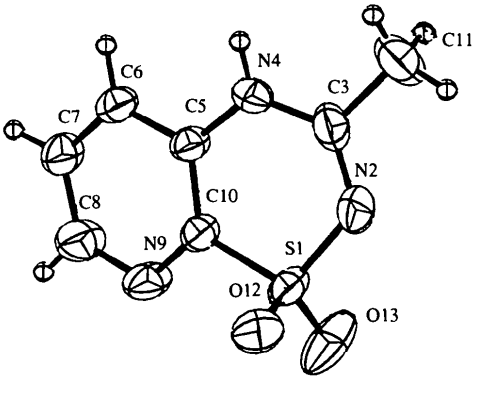


Fig. 1. Molecular structure with atom-labelling scheme of molecule *A*. (In molecule *B*, the atom numbering is incremented by 20.) Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Crystal data

$C_7H_7N_3O_2S$
$M_r = 197.22$
Monoclinic
Cc
$a = 20.709\ (3)$ Å
$b = 7.7770\ (14)$ Å
$c = 17.276\ (3)$ Å
$\beta = 143.279\ (5)°$
$V = 1663.6\ (5)$ Å^3
$Z = 8$
$D_x = 1.575$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54180$ Å
Cell parameters from 39 reflections
$\theta = 11.43–39.45°$
$\mu = 3.237$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.38 × 0.27 × 0.25 mm
Colourless
Crystal source: Laboratory of Medicinal Chemistry, Liège

Data collection

Stoe Siemens AED four-circle diffractometer
ω scans
Absorption correction: semi-empirical *via* ψ scans
$T_{min} = 0.3892$, $T_{max} = 0.5141$
1049 measured reflections
1049 independent reflections
1032 observed reflections $[I > 2\sigma(I)]$
$\theta_{max} = 54.99°$
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 8$
$l = -18 \rightarrow 10$
2 standard reflections
frequency: 60 min
intensity decay: 5.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0268$
$wR(F^2) = 0.0691$
$S = 1.117$
1049 reflections
240 parameters
H atoms included as riding atoms at calculated positions and constrained
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 1.2809P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.178$ e Å^{-3}
$\Delta\rho_{min} = -0.223$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + 0.001\chi F_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0056 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983), $\eta = 0.09\ (2)$

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

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

	x	y	z	U_{eq}
S1	0.26022 (8)	0.06330 (15)	0.82916 (9)	0.0441 (4)
N2	0.1610 (4)	−0.0669 (5)	0.7277 (5)	0.0543 (12)
C3	0.0618 (4)	−0.0189 (7)	0.6465 (6)	0.0481 (15)
N4	0.0312 (3)	0.1350 (5)	0.6422 (4)	0.0427 (9)
C5	0.1018 (3)	0.2573 (6)	0.7384 (4)	0.0365 (10)
C6	0.0659 (4)	0.4028 (6)	0.7434 (5)	0.0461 (12)
C7	0.1394 (5)	0.5136 (6)	0.8433 (6)	0.0527 (13)
C8	0.2488 (4)	0.4778 (8)	0.9400 (6)	0.062 (2)
N9	0.2860 (3)	0.3421 (6)	0.9374 (4)	0.0525 (11)
C10	0.2130 (3)	0.2377 (6)	0.8371 (4)	0.0379 (10)
C11	−0.0245 (5)	−0.1502 (8)	0.5519 (7)	0.074 (2)
O12	0.2861 (3)	0.1236 (5)	0.7772 (4)	0.0593 (10)
O13	0.3461 (4)	−0.0172 (6)	0.9522 (4)	0.0832 (15)
S21	0.03733 (7)	0.03070 (13)	0.88108 (8)	0.0383 (3)
N22	0.1458 (3)	−0.0644 (5)	0.9548 (4)	0.0465 (11)
C23	0.2431 (4)	−0.0119 (6)	1.0665 (5)	0.0360 (11)
N24	0.2678 (3)	0.1352 (5)	1.1237 (3)	0.0369 (9)
C25	0.1927 (3)	0.2591 (5)	1.0735 (4)	0.0334 (10)
C26	0.2240 (4)	0.4173 (6)	1.1345 (4)	0.0413 (11)
C27	0.1475 (4)	0.5357 (7)	1.0781 (5)	0.0464 (12)
C28	0.0390 (4)	0.4967 (6)	0.9609 (5)	0.0466 (12)
N29	0.0063 (3)	0.3481 (5)	0.9003 (4)	0.0413 (9)
C30	0.0828 (3)	0.2312 (6)	0.9575 (4)	0.0359 (11)
C31	0.3343 (4)	−0.1270 (7)	1.1316 (5)	0.0540 (14)
O32	−0.0054 (3)	−0.0625 (4)	0.9030 (4)	0.0539 (9)
O33	−0.0333 (3)	0.0571 (5)	0.7483 (3)	0.0604 (10)

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

S1—O13	1.419 (4)	S21—O32	1.410 (4)
S1—O12	1.444 (4)	S21—O33	1.437 (4)
S1—N2	1.595 (5)	S21—N22	1.621 (4)
S1—C10	1.741 (4)	S21—C30	1.752 (5)
N2—C3	1.306 (7)	N22—C23	1.310 (6)
C3—N4	1.328 (6)	C23—N24	1.317 (6)
C3—C11	1.491 (8)	C23—C31	1.493 (7)
N4—C5	1.382 (6)	N24—C25	1.392 (5)
C5—C6	1.393 (7)	C25—C26	1.396 (6)
C5—C10	1.392 (6)	C25—C30	1.390 (6)
C6—C7	1.351 (7)	C26—C27	1.354 (7)
C7—C8	1.390 (8)	C27—C28	1.395 (7)
C8—N9	1.328 (7)	C28—N29	1.324 (6)
N9—C10	1.322 (6)	N29—C30	1.342 (6)
O13—S1—O12	115.5 (3)	O32—S21—O33	116.5 (2)
O13—S1—N2	109.3 (3)	O32—S21—N22	109.1 (2)
O12—S1—N2	108.5 (2)	O33—S21—N22	108.4 (2)
O13—S1—C10	109.5 (2)	O32—S21—C30	109.2 (2)
O12—S1—C10	107.9 (2)	O33—S21—C30	108.7 (2)
N2—S1—C10	105.7 (2)	N22—S21—C30	104.2 (2)
C3—N2—S1	122.0 (4)	C23—N22—S21	122.7 (3)
N2—C3—N4	125.9 (5)	N22—C23—N24	126.1 (4)
N2—C3—C11	117.0 (5)	N22—C23—C31	116.5 (4)
N4—C3—C11	117.2 (5)	N24—C23—C31	117.3 (4)
C3—N4—C5	123.8 (4)	C23—N24—C25	124.4 (4)
N4—C5—C6	122.1 (4)	N24—C25—C26	121.9 (4)
N4—C5—C10	120.9 (4)	N24—C25—C30	120.7 (4)
C6—C5—C10	117.0 (4)	C26—C25—C30	117.3 (4)
C7—C6—C5	119.1 (4)	C27—C26—C25	119.3 (4)
C6—C7—C8	119.3 (5)	C26—C27—C28	119.4 (5)
N9—C8—C7	123.3 (5)	N29—C28—C27	123.0 (5)
C10—N9—C8	116.6 (4)	C28—N29—C30	117.1 (4)
N9—C10—C5	124.7 (4)	N29—C30—C25	123.9 (4)
N9—C10—S1	117.2 (3)	N29—C30—S21	116.4 (3)
C5—C10—S1	118.1 (3)	C25—C30—S21	119.7 (3)
O13—S1—N2—C3	134.8 (5)	O32—S21—N22—C23	−100.3 (4)
O12—S1—N2—C3	−98.5 (5)	O33—S21—N22—C23	131.9 (4)
C10—S1—N2—C3	17.0 (5)	C30—S21—N22—C23	16.3 (5)
S1—N2—C3—N4	−4.4 (8)	S21—N22—C23—N24	−11.8 (7)
N2—C3—N4—C5	−12.5 (9)	N22—C23—N24—C25	0.6 (8)
C3—N4—C5—C10	11.3 (7)	C23—N24—C25—C30	1.9 (7)
N4—C5—C10—S1	4.7 (6)	N24—C25—C30—S21	6.0 (5)
O13—S1—C10—C5	−134.4 (4)	O32—S21—C30—C25	103.0 (4)
O12—S1—C10—C5	99.1 (4)	O33—S21—C30—C25	−128.9 (4)
N2—S1—C10—C5	−16.8 (4)	N22—S21—C30—C25	−13.5 (4)

Data collection: *DIF*4 (Stoe & Cie, 1987*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1987*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

The authors thank M. M. Vermeire for his helpful assistance in the diffractometry measurements and the FNRS for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1150). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 946–948

3-Methyl-4*H*-pyrido[4,3-*e*][1,2,4]thiadiazine 1,1-Dioxide Monohydrate

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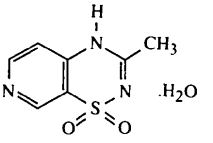
(*Received* 12 *October* 1994; *accepted* 15 *November* 1994)

Abstract

The title compound, $C_7H_7N_3O_2S.H_2O$, was prepared for structural and pharmacological comparison with diazoxide, an antihypertensive agent. The crystal structure determination shows that the 4*H* (rather than 2*H*) tautomeric form is preferentially adopted by this pyridothiadiazine derivative in the solid state.

Comment

Little has been reported on the pyrido[4,3-*e*][1,2,4]-thiadiazine 1,1-dioxide ring system. Only a few 4-aryl- and 3-aminoalkyl-4*H*-pyrido[4,3-*e*][1,2,4]thiadiazine 1,1-dioxides have been described (Delarge & Lapière, 1974; Pirotte, de Tullio, Lebrun, Antoine, Fontaine, Masereel, Schynts, Dupont, Herchuelz & Delarge, 1993). The title compound (I) also contains this ring system and is related structurally to diazoxide [7-chloro-3-methyl-2*H*(or 4*H*)-1,2,4-benzothiadiazine 1,1-dioxide], a well known antihypertensive agent currently reported as the pharmacological reference for the benzothiadiazine class of ATP-sensitive potassium-channel openers (Edwards & Weston, 1990).



(I)

The X-ray analysis of the title compound may help to determine whether the 2*H* or 4*H* tautomer is the preferred form in the solid state. The thiadiazine molecule is linked to the water molecule by the hydrogen bonds N4—H4···O14 [N4···O14 2.734 (3), H4···O14 1.89 (4) Å, N4—H4···O14 171 (3)°] and O14—H142···N8^{i} [O14···N8^{i} 2.775 (3), H142···N8^{i} 1.76 (2) Å, O14—H142···N8^{i} 179 (2)°; symmetry code: (i) $x-\frac{1}{2}, -\frac{1}{2}-y, \frac{1}{2}+z$]. The N2—C3 and N4—C3 bond lengths, the location of the H atom on atom N4 rather than N2 and the hydrogen-bonding scheme all indicate that the 4*H* tautomeric form is favoured in the crystal. The same conclusion has been made for diazoxide [N2—C3 1.300 (9) and N4—C3 1.335 (9) Å; Bandoli & Nicolini, 1977] and the [3,2-*e*] derivative [N2—C3 1.306 (7) and N4—C3 1.328 (6) Å; Dupont, Pirotte, de Tullio, Masereel & Delarge, 1995].

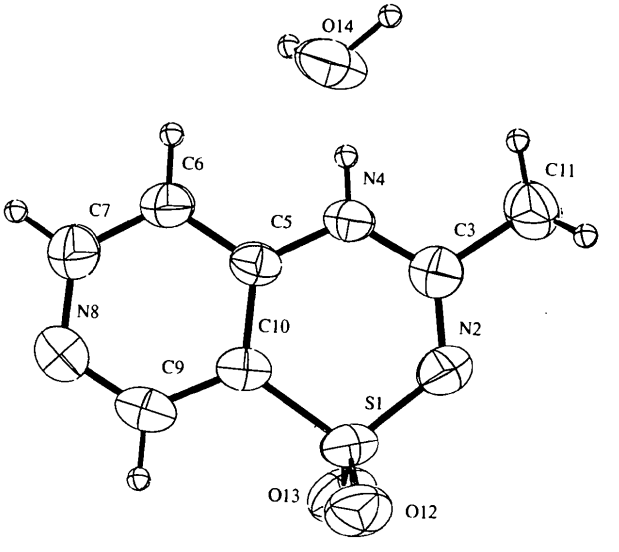


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.