Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: PA1192). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bandoli, G. & Nicolini, M. (1977). *J. Cryst. Mol. Struct.* **7**, 229–240.

Dupont, L., Pirotte, B., de Tullio, P., Diouf, O., Masereel, B. & Delarge, J. (1995). *Acta Cryst.* C**51**, 2412–2414.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Stoe & Cie (1987*a*). *DIF*4. *Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1987*b*). *EMPIR*. *Empirical Absorption Correction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1987*c*). *REDU*4. *Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Tullio, P. de, Pirotte, B., Dupont, L., Masereel, B., Laeckmann, D., Podona, T., Diouf, O., Lebrun, P. & Delarge, J. (1995). *Tetrahedron*, **51**, 3221–3234.

Acta Cryst. (1995). C**51**, 2416–2417

7-Methyl-3-methylthio-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazin-7-ium-4-ide 1,1-Dioxide Zwitterion

LÉON DUPONT

Unité de Cristallographie, Institut de Physique B5, Université de Liège au Sart Tilman, B-4000 Liège, Belgium

PASCAL DE TULLIO, BERNARD PIROTTE, BERNARD MASEREEL AND JACQUES DELARGE

Laboratoire de Chimie Pharmaceutique, Institut de Pharmacie F1, Université de Liège, Rue Fusch 5, B-4000 Liège, Belgium

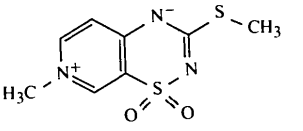
(*Received* 22 *May* 1995; *accepted* 19 *June* 1995)

Abstract

The title compound, $C_8H_9N_3O_2S_2$, is an original drug structurally related to diazoxide, an antihypertensive compound. At neutral pH, the molecule is a zwitterion which contains a pyridinium heterocycle. The negative charge is localized at the 4 position.

Comment

The title compound, 7-methyl-3-methylthio-4*H*-pyrido-[4,3-*e*]-1,2,4-thiadiazin-7-ium-4-ide 1,1-dioxide, (I), results from the reaction of 3-methylthio-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazine 1,1-dioxide with iodomethane in aqueous alkaline solution. At neutral pH, the compound is isolated as a zwitterion and is used for the synthesis of 3-aminoalkyl derivatives that have been tested as original pyridothiadiazine potassium channel openers. It is structurally related to diazoxide [7-chloro-3-methyl-2*H*(or 4*H*)-1,2,4-benzothiadiazine 1,1-dioxide], a well known antihypertensive drug (Bandoli & Nicolini, 1977).



(I)

The value of the C7—N8—C9 angle [119.2 (3)°] is typical of a pyridinium ring (Dupont, Pirotte, de Tullio, Masereel & Delarge, 1995). The values of the torsion angles show that the molecule is almost planar. The deviations of atoms N4, S1 and C12 from the mean plane of the pyridinium ring are 0.032 (4), −0.068 (4) and 0.059 (5) Å, respectively. These values are comparable with those found in the structure of the 3,7-dimethyl-4*H*-pyrido[4,3-*e*]-1,2,4-thiadiazine 1,1-dioxide zwitterion (Dupont *et al.*, 1995). The molecule is a zwitterion where the negative charge is localized on N4 rather than on N2. The N2—C3 bond length [1.322 (4) Å] is closer to the standard double-bond value than that of the 3,7-dimethyl derivative [1.335 (4) Å].

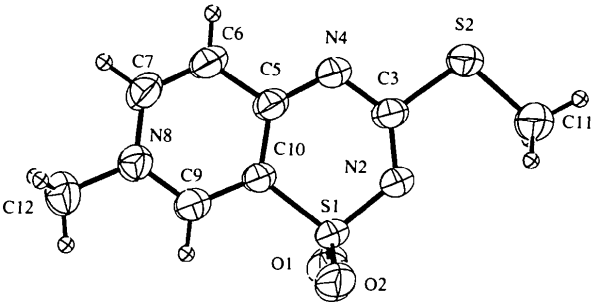


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

The title compound was synthesized at the Laboratory of Medicinal Chemistry of Liège. The method of preparation will be published elsewhere. Crystals were obtained by slow evaporation of a water–methanol solution (75/25% by volume) at room temperature.

Crystal data

$C_8H_9N_3O_2S_2$
M_r = 243.30
Triclinic
$P\bar{1}$
a = 7.3767 (7) Å
b = 8.1696 (7) Å
c = 8.5483 (7) Å
α = 82.557 (9)°
β = 83.195 (7)°
γ = 84.162 (5)°
V = 505.27 (8) Å^3
Z = 2
D_x = 1.599 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 41 reflections
θ = 27.85–37.31°
μ = 4.668 mm^{-1}
T = 293 (2) K
Prism
0.61 × 0.30 × 0.27 mm
Colourless

Data collection

Stoe Siemens AED four-circle diffractometer
ω scans
Absorption correction: ψ scan (Stoe & Cie, 1987*b*)
T_{min} = 0.244, T_{max} = 0.356
1378 measured reflections
1378 independent reflections

1276 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 57.44°
$h = 0 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -9 \rightarrow 9$
2 standard reflections
frequency: 60 min
intensity decay: 2.4%

Refinement

Refinement on F^2
$R(F)$ = 0.0420
$wR(F^2)$ = 0.1297
S = 1.051
1376 reflections
139 parameters
H atoms restrained (included as riding atoms)
$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.3378P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.259 e Å^{-3}
$\Delta\rho_{min}$ = −0.375 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.102 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
S1	0.26777 (11)	0.27588 (9)	0.75221 (8)	0.0488 (4)
N2	0.3043 (4)	0.4556 (3)	0.7859 (3)	0.0561 (7)
C3	0.2920 (4)	0.5866 (4)	0.6780 (3)	0.0464 (7)
N4	0.2559 (4)	0.6006 (3)	0.5271 (3)	0.0538 (7)
C5	0.2336 (4)	0.4615 (4)	0.4666 (3)	0.0476 (7)
C6	0.2012 (5)	0.4711 (4)	0.3049 (4)	0.0580 (9)
C7	0.1803 (5)	0.3355 (4)	0.2380 (4)	0.0561 (8)
N8	0.1898 (4)	0.1815 (3)	0.3217 (3)	0.0519 (7)
C9	0.2186 (4)	0.1656 (4)	0.4754 (4)	0.0511 (8)
C10	0.2378 (4)	0.3003 (4)	0.5500 (3)	0.0451 (7)
C11	0.3437 (6)	0.7443 (4)	0.9369 (4)	0.0666 (10)
C12	0.1744 (5)	0.0335 (5)	0.2434 (4)	0.0658 (10)
S2	0.32680 (12)	0.78010 (9)	0.72877 (9)	0.0541 (4)
O1	0.0998 (4)	0.2308 (3)	0.8423 (3)	0.0706 (7)
O2	0.4245 (4)	0.1621 (3)	0.7796 (3)	0.0708 (7)

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

S1—O2	1.430 (2)	C5—C10	1.413 (4)
S1—O1	1.434 (2)	C5—C6	1.421 (4)
S1—N2	1.587 (3)	C6—C7	1.341 (5)
S1—C10	1.752 (3)	C7—N8	1.365 (4)
N2—C3	1.322 (4)	N8—C9	1.343 (4)
C3—N4	1.336 (4)	N8—C12	1.476 (4)
C3—S2	1.743 (3)	C9—C10	1.369 (4)
N4—C5	1.340 (4)	C11—S2	1.782 (3)
O2—S1—O1	115.1 (2)	N4—C5—C6	119.3 (3)
O2—S1—N2	110.2 (2)	C10—C5—C6	114.9 (3)
O1—S1—N2	108.8 (2)	C7—C6—C5	121.7 (3)
O2—S1—C10	109.34 (14)	C6—C7—N8	121.6 (3)
O1—S1—C10	108.87 (14)	C9—N8—C7	119.2 (3)
N2—S1—C10	104.05 (14)	C9—N8—C12	120.3 (3)
C3—N2—S1	122.6 (2)	C7—N8—C12	120.5 (3)
N2—C3—N4	130.9 (3)	N8—C9—C10	121.6 (3)
N2—C3—S2	119.1 (2)	C9—C10—C5	121.1 (3)
N4—C3—S2	110.0 (2)	C9—C10—S1	120.4 (2)
C3—N4—C5	117.8 (2)	C5—C10—S1	118.6 (2)
N4—C5—C10	125.8 (3)	C3—S2—C11	104.2 (2)
O2—S1—N2—C3	123.0 (3)	C6—C7—N8—C12	−177.3 (3)
O1—S1—N2—C3	−110.0 (3)	N8—C9—C10—S1	177.9 (2)
C10—S1—N2—C3	5.9 (3)	N4—C5—C10—S1	2.0 (5)
S1—N2—C3—N4	−2.4 (6)	C6—C5—C10—S1	−177.5 (2)
S1—N2—C3—S2	177.8 (2)	N2—S1—C10—C9	174.4 (3)
N2—C3—N4—C5	−2.8 (5)	O2—S1—C10—C5	−123.4 (3)
S2—C3—N4—C5	177.1 (2)	O1—S1—C10—C5	110.1 (3)
C3—N4—C5—C10	2.6 (5)	N2—S1—C10—C5	−5.7 (3)
C3—N4—C5—C6	−177.9 (3)	N2—C3—S2—C11	−7.2 (3)

Data collection: *DIF*4 (Stoe & Cie, 1987*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1987*c*). 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 Belgian FNRS (Fonds National de la Recherche Scientifique) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: PA1194). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bandoli, G. & Nicolini, M. (1977). *J. Cryst. Mol. Struct.* **7**, 229–240.
Dupont, L., Pirotte, B., de Tullio, P., Masereel, B. & Delarge, J. (1995). *Acta Cryst.* C**51**, 2414–2416.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
Stoe & Cie (1987*a*). *DIF*4. *Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1987*b*). *EMPIR*. *Empirical Absorption Correction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1987*c*). *REDU*4. *Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.