

Absorption correction: $\theta_{\max} = 25^\circ$
 refined from ΔF $h = 0 \rightarrow 9$
 (DIFABS; Walker & $k = -11 \rightarrow 11$
 Stuart, 1983) $l = -11 \rightarrow 11$
 $T_{\min} = 0.52$, $T_{\max} = 1.00$ 3 standard reflections
 2675 measured reflections frequency: 120 min
 2484 independent reflections intensity decay: < 3%

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F) + 0.0015F^2]$
 $R = 0.0278$ $(\Delta/\sigma)_{\max} = 0.06$
 $wR = 0.0382$ $\Delta\rho_{\max} = 0.66 \text{ e } \text{Å}^{-3}$
 $S = 0.67$ $\Delta\rho_{\min} = -1.07 \text{ e } \text{Å}^{-3}$
 2092 reflections Atomic scattering factors
 169 parameters from *International Tables*
 Only H-atom U 's refined for *X-ray Crystallography*
 (1974, Vol. IV)

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

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Te	0.04417 (4)	0.72426 (3)	0.48724 (4)	0.046
I	-0.21613 (5)	0.82044 (4)	0.72481 (4)	0.065
N(1)	0.2476 (5)	0.6122 (4)	0.3297 (4)	0.046
C(1)	0.1798 (7)	0.5157 (5)	0.6112 (6)	0.047
C(2)	0.1465 (8)	0.4648 (6)	0.7562 (6)	0.056
C(3)	0.2469 (9)	0.3222 (6)	0.8218 (7)	0.065
C(4)	0.3799 (8)	0.2307 (6)	0.7459 (7)	0.061
C(5)	0.4152 (7)	0.2807 (6)	0.6017 (6)	0.056
C(6)	0.3163 (7)	0.4236 (5)	0.5326 (6)	0.048
C(7)	0.2531 (7)	0.6814 (5)	0.1793 (5)	0.047
C(8)	0.2228 (7)	0.8329 (6)	0.1351 (6)	0.054
C(9)	0.2251 (8)	0.9026 (6)	-0.0069 (6)	0.056
C(10)	0.2511 (7)	0.8236 (6)	-0.1078 (6)	0.055
C(11)	0.2791 (8)	0.6714 (6)	-0.0598 (6)	0.058
C(12)	0.2795 (7)	0.6003 (6)	0.0823 (6)	0.053
C(13)	0.2507 (9)	0.9000 (8)	-0.2632 (7)	0.069
C(14)	0.3454 (7)	0.4793 (6)	0.3829 (6)	0.050

Table 2. Selected geometric parameters (Å , $^\circ$)

Te—I	2.936 (1)	C(5)—C(6)	1.397 (7)
Te—N(1)	2.230 (4)	C(6)—C(14)	1.424 (8)
Te—C(1)	2.102 (5)	C(7)—C(8)	1.377 (7)
N(1)—C(7)	1.447 (7)	C(7)—C(12)	1.376 (7)
N(1)—C(14)	1.285 (7)	C(8)—C(9)	1.375 (8)
C(1)—C(2)	1.379 (8)	C(9)—C(10)	1.395 (8)
C(1)—C(6)	1.406 (7)	C(10)—C(11)	1.387 (8)
C(2)—C(3)	1.389 (8)	C(10)—C(13)	1.505 (8)
C(3)—C(4)	1.376 (8)	C(11)—C(12)	1.379 (8)
C(4)—C(5)	1.372 (8)		
I—Te—N(1)	170.3 (1)	C(1)—C(6)—C(14)	118.0 (4)
I—Te—C(1)	94.8 (1)	C(5)—C(6)—C(14)	122.2 (5)
N(1)—Te—C(1)	76.3 (2)	N(1)—C(7)—C(8)	118.4 (4)
Te—N(1)—C(7)	121.2 (3)	N(1)—C(7)—C(12)	121.3 (4)
Te—N(1)—C(14)	114.5 (3)	C(8)—C(7)—C(12)	120.2 (5)
C(7)—N(1)—C(14)	124.0 (4)	C(7)—C(8)—C(9)	119.9 (5)
Te—C(1)—C(2)	126.5 (4)	C(8)—C(9)—C(10)	121.2 (5)
Te—C(1)—C(6)	113.9 (4)	C(9)—C(10)—C(11)	117.5 (5)
C(2)—C(1)—C(6)	119.6 (5)	C(9)—C(10)—C(13)	121.3 (5)
C(1)—C(2)—C(3)	119.2 (5)	C(11)—C(10)—C(13)	121.2 (5)
C(2)—C(3)—C(4)	121.7 (6)	C(10)—C(11)—C(12)	121.7 (5)
C(3)—C(4)—C(5)	119.6 (5)	C(7)—C(12)—C(11)	119.4 (5)
C(4)—C(5)—C(6)	120.1 (5)	N(1)—C(14)—C(6)	117.3 (4)
C(1)—C(6)—C(5)	119.8 (5)		

H atoms were placed in calculated positions (C—H 1.08 Å), riding on their respective bonded atoms. The methyl substituent was refined as a rigid rotating group.

Computer programs used include *SHELX76* (Sheldrick, 1976), *SHELX86* (Sheldrick, 1985) and *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1134). 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-4H-pyrido[2,3-e]-1,2,4-thiadiazine 1,1-Dioxide

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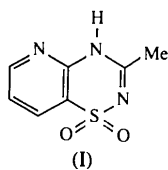
Abstract

The title compound, C₇H₇N₃O₂S, 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* (rather than 2*H*) tautomeric form is preferentially adopted by this pyridothiadiazine derivative in the solid state.

Comment

The title compound, (I), may be regarded as a pyridinic analogue of the antihypertensive agent diazoxide [7-chloro-3-methyl-2*H*(or 4*H*)-1,2,4-benzothiadiazine 1,1-dioxide] and was prepared in order to compare the two thiadiazine ring systems. The most interesting geometrical parameters in the title compound are the C—N bond lengths in the thiadiazine ring, as these indicate whether the C=N double bond is located in the 2,3 position (the 4*H* tautomer) or in the 3,4 position (the 2*H* tautomer). Diazoxide itself was originally reported to be a 2*H*-benzothiadiazine but was in fact shown to be the 4*H* tautomer using spectroscopic and X-ray data (Masereel, Ferrari, Pirotte, Schynts, Parenti & Delarge, 1992; Bandoli & Nicolini, 1977; Novello, Bell, Abrams, Ziegler & Sprague, 1960).



The molecules of (I) are linked by N4—H4···N6ⁱ hydrogen bonds [symmetry code: (i) $\frac{5}{2}-x, \frac{1}{2}-y, 1-z$] with N4···N6ⁱ 2.986 (3), H4···N6ⁱ 2.12 (3) Å and N4—H4···N6ⁱ 175 (2)°. According to the lengths of the N2—C3 and N4—C3 bonds and the hydrogen-bonding scheme, the 4*H* tautomeric form seems to be predominant in the crystal, as found for the [3,2-*e*] and [4,3-*e*] derivatives (Dupont, Pirotte, de Tullio, Masereel & Delarge, 1995; Dupont, de Tullio, Pirotte, Masereel & Delarge, 1995).

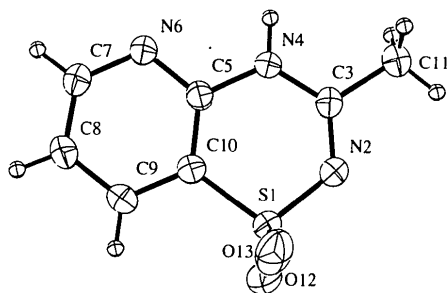


Fig. 1. Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level; H atoms are shown as small circles of arbitrary radii.

Experimental

Crystal data

C₇H₇N₃O₂S
M_r = 197.22
 Monoclinic
*C*2/*c*
a = 12.2439 (12) Å
b = 10.6348 (9) Å
c = 14.0329 (13) Å
 β = 117.964 (6)°
V = 1613.9 (3) Å³
Z = 8
D_x = 1.623 Mg m⁻³

Cu K α radiation
 λ = 1.5418 Å

Cell parameters from 33 reflections

θ = 18.40–36.59°

μ = 3.336 mm⁻¹

T = 293 (2) K

Prism

0.50 × 0.19 × 0.17 mm

Colourless

Crystal source: Laboratory of Medicinal Chemistry, Liège

Data collection

Stoe Siemens AED four-circle diffractometer

ω scans

Absorption correction:

ψ scans (EMPIR; Stoe & Cie, 1987b)

T_{min} = 0.493, *T_{max}* = 0.554

1017 measured reflections

1017 independent reflections

905 observed reflections

[*I* > 2 σ (*I*)]

θ_{\max} = 54.98°

h = 0 → 12

k = 0 → 11

l = -14 → 12

2 standard reflections

frequency: 60 min

intensity decay: 3.5%

Refinement

Refinement on *F*²

$R[F^2 > 2\sigma(F^2)] = 0.0284$

$wR(F^2) = 0.0769$

S = 1.084

1017 reflections

125 parameters

H atoms treated using a riding model

$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 2.02058P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} < 0.001

$\Delta\rho_{\max} = 0.238 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.236 \text{ e \AA}^{-3}$

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.0046 (3)

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 (Å²)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
S1	0.81558 (5)	0.25147 (5)	0.30271 (5)	0.0377 (3)
N2	0.8825 (2)	0.3858 (2)	0.3166 (2)	0.0461 (6)
C3	1.0007 (2)	0.4023 (2)	0.3765 (2)	0.0367 (6)
N4	1.0845 (2)	0.3109 (2)	0.4242 (2)	0.0369 (5)
C5	1.0570 (2)	0.1830 (2)	0.4093 (2)	0.0310 (6)
N6	1.1530 (2)	0.1040 (2)	0.4538 (2)	0.0352 (5)
C7	1.1268 (2)	-0.0182 (2)	0.4373 (2)	0.0396 (6)
C8	1.0098 (2)	-0.0667 (2)	0.3783 (2)	0.0443 (7)
C9	0.9116 (2)	0.0149 (2)	0.3335 (2)	0.0427 (7)
C10	0.9360 (2)	0.1426 (2)	0.3500 (2)	0.0332 (6)
C11	1.0492 (2)	0.5337 (2)	0.3934 (2)	0.0504 (8)
O12	0.7351 (2)	0.2313 (2)	0.1910 (2)	0.0579 (6)
O13	0.7603 (2)	0.2491 (2)	0.3718 (2)	0.0621 (6)

Table 2. Selected geometric parameters (Å, °)

S1—O13	1.419 (2)	N4—C5	1.393 (3)
S1—O12	1.423 (2)	C5—N6	1.338 (3)
S1—N2	1.612 (2)	C5—C10	1.385 (3)
S1—C10	1.742 (2)	N6—C7	1.333 (3)
N2—C3	1.300 (3)	C7—C8	1.375 (4)
C3—N4	1.342 (3)	C8—C9	1.373 (3)
C3—C11	1.494 (3)	C9—C10	1.386 (3)
O13—S1—O12	116.38 (13)	N6—C5—C10	123.0 (2)
O13—S1—N2	108.13 (12)	N6—C5—N4	116.5 (2)
O12—S1—N2	108.47 (12)	C10—C5—N4	120.6 (2)
O13—S1—C10	108.52 (11)	C7—N6—C5	116.4 (2)
O12—S1—C10	110.10 (11)	N6—C7—C8	124.6 (2)
N2—S1—C10	104.56 (10)	C9—C8—C7	118.7 (2)
C3—N2—S1	123.1 (2)	C8—C9—C10	118.0 (2)
N2—C3—N4	125.7 (2)	C5—C10—C9	119.3 (2)
N2—C3—C11	118.0 (2)	C5—C10—S1	119.9 (2)
N4—C3—C11	116.3 (2)	C9—C10—S1	120.7 (2)
C3—N4—C5	123.9 (2)		
O13—S1—N2—C3	99.7 (2)	C11—C3—N4—C5	-174.2 (2)
O12—S1—N2—C3	-133.3 (2)	C3—N4—C5—C10	-5.8 (3)
C10—S1—N2—C3	-15.8 (2)	N4—C5—C10—S1	-5.3 (3)
S1—N2—C3—N4	8.0 (4)	O13—S1—C10—C5	-100.9 (2)
S1—N2—C3—C11	-172.8 (2)	O12—S1—C10—C5	130.7 (2)
N2—C3—N4—C5	5.1 (4)	N2—S1—C10—C5	14.4 (2)

Data collection: *DIF4* (Stoe & Cie, 1987a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1987c). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1157). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Méthyl-7-phényl-3,5-dithioxo-3,4,5,6-tétrahydro-2H-1,2,4-triazépine

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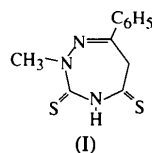
Abstract

The structure of the compound 2-methyl-7-phenyl-3,4,5,6-tetrahydro-2H-1,2,4-triazepine-3,5-dithione consists of (C₁₁H₁₁N₃S₂)₂ dimers. Asymmetric units are dimerized through two N—H···S hydrogen bonds [N···S 3,302 (2) Å]. The cell contains four dimers linked in chains along the *c* axis by van der Waals contacts.

Commentaire

Il a été démontré que les composés présentant un hétérocycle accolé à un cycle à sept chaînons, révélaient dans certains cas des propriétés pharmacologiques remarquables (Bellantuono, Reggi, Tognoni & Garattini, 1980; Bartsch & Erker, 1988). Les triazépines offrent la possibilité d'accéder à ce type de molécules actives (Hasnaoui, El Messaoudi & Lavergne, 1985; El Messaoudi, Hasnaoui, El Mouhadi & Lavergne, 1992).

Dans ce mémoire nous rapportons la structure cristallographique, déterminée par diffraction de rayons X sur monocristal d'une nouvelle triazépine: la 2-méthyl-7-phényl-3,5-dithioxo-3,4,5,6-tétrahydro-2H-1,2,4-triazépine, (I).



La première triazépine analogue, la 2,7-diméthyl-5-oxo-3-thioxo-3,4,5,6-tétrahydro-2H-1,2,4-triazépine, a été préparée par Losse, Hessler & Barth (1958) à partir du 2-méthylthiosemicarbazide et de l'acétylacétate d'éthyle. Cette même triazépine a été reprise par Hasnaoui, Lavergne & Viallefont (1978, 1980) comme