Data collection: *SMART* (Siemens, 1994*a*). Cell refinement: *SMART*. Data reduction: *SHELXTL* (Siemens, 1994*b*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1035). Services for accessing these data are described at the back of the journal.

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Two new compounds by reaction of taurolidine with methylene glycol

ALAN R. KENNEDY,[a] GRAHAM G. SKELLERN,[b] ROLF W. PFIRRMANN,[c] GORDON A. SMAIL,[b] NORMAN SHANKLAND[b] AND ALASTAIR J. FLORENCE[b]

[a]*Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow G1 1XL, Scotland,* [b]*Department of Pharmaceutical Sciences, University of Strathclyde, Glasgow G1 1XW, Scotland, and* [c]*Geistlich-Pharma, Wolhusen, Switzerland. E-mail: norman@pharmsci.strath.ac.uk*

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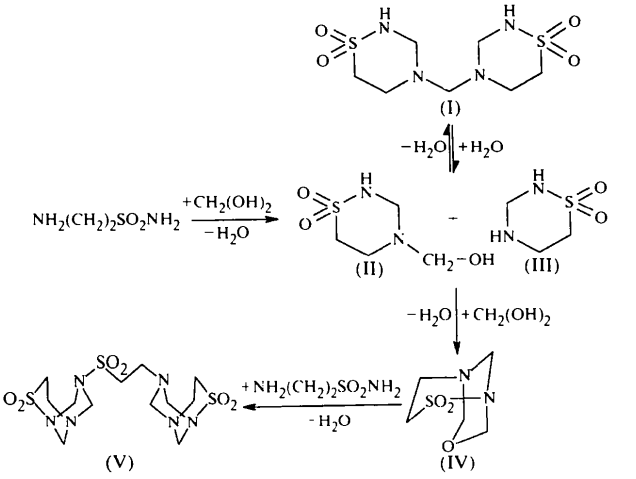
Abstract

The compounds 7-oxa-2[λ]^6-thia-1,5-diazabicyclo-[3.3.1]nonane-2,2-dione ($C_5H_{10}N_2O_3S$) and 7-{[2-(2,2-dioxo-2[λ]^6-thia-1,5,7-triazabicyclo[3.3.1]non-7-yl)ethyl]sulfonyl}-2[λ]^6-thia-1,5,7-triazabicyclo[3.3.1]-nonane-2,2-dione ($C_{12}H_{24}N_6O_6S_3$) are produced when taurolidine is reacted with an excess of methylene glycol. The saturated six-membered heterocyclic rings in both compounds adopt distorted chair conformations.

Comment

Taurolidine is a broad-spectrum bactericide and anti-endotoxin (Browne *et al.*, 1976). The scheme below shows the reaction sequence for the synthesis of taurolidine, (I), *via* compounds (II) and (III) which have been identified by NMR (Myers *et al.*, 1980; Erb *et al.*, 1982; Knight *et al.*, 1983; Hood *et al.*, 1994). We present here



the crystal structures of two new compounds, 7-oxa-2[λ]^6-thia-1,5-diazabicyclo[3.3.1]nonane-2,2-dione [(IV); Fig. 1] and 7-{[2-(2,2-dioxo-2[λ]^6-thia-1,5,7-triazabicyclo[3.3.1]non-7-yl)ethyl]sulfonyl}-2[λ]^6-thia-1,5,7-triazabicyclo[3.3.1]nonane-2,2-dione [(V); Fig. 2], which are formed when the reaction mixture contains an excess of methylene glycol.

Compound (IV) contains two six-membered rings sharing atoms N1, C5 and N2. Both the thiadiaza- and oxadiazacyclohexane rings adopt distorted chair conformations, with puckering parameters Q = 0.586 (3) Å, θ = 12.1 (3)°, φ = 8.8 (10)°, and Q = 0.550 (3) Å, θ = 5.4 (3)°, φ = 342 (3)°, respectively (Cremer & Pople, 1975). Compound (V) contains four six-membered rings, *i.e.* a thiadiaza- and a triazacyclohexane ring fused together across N1—C3—N2, and a thiadiaza- and a triazacyclohexane ring fused together across N5—C10—N6. As in compound (IV), each ring adopts a distorted chair conformation [puckering parameters: ring C3—N2—C4—N3—C5—N1 Q = 0.547 (3) Å, θ = 7.4 (3)° and φ = 359 (2)°; ring C3—N1—S1—C1—C2—N2 Q = 0.583 (2) Å, θ = 13.5 (3)° and φ = 13.6 (9)°; ring C10—N5—C11—C12—S3—N6 Q = 0.589 (3) Å, θ = 13.1 (3)° and φ = 354 (1)°; ring C10—N6—C9—N4—C8—N5 Q = 0.561 (3) Å, θ = 4.0 (3)° and φ = 29 (3)°].

Intermolecular C—H···O and C—H···N close contacts are listed in Tables 2 [compound (IV)] and 4 [compound (V)]. The observed distances are consistent with those commonly observed for weak hydrogen bonds in organic molecular crystals (Jeffrey & Saenger, 1991).

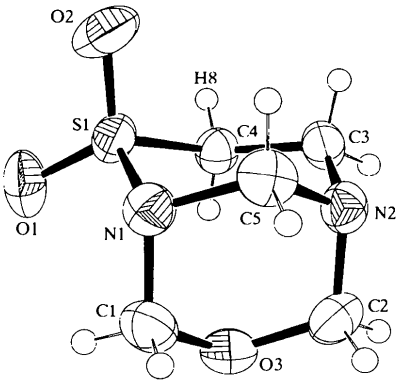


Fig. 1. *ORTEP*III (Burnett & Johnson, 1996) view of (IV). Displacement ellipsoids are set at the 50% probability level and H atoms are drawn as small spheres of arbitrary size.

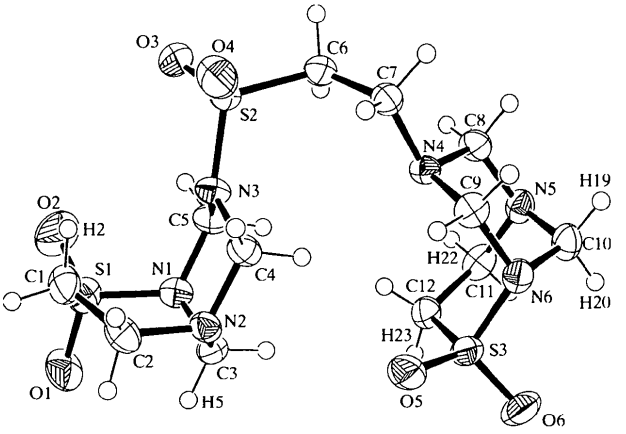


Fig. 2. *ORTEP*III (Burnett & Johnson, 1996) view of (V). Displacement ellipsoids are set at the 50% probability level and H atoms are drawn as small spheres of arbitrary size.

Experimental

Single crystals of (IV) and (V) were obtained from an aqueous reaction mixture in which (I) was treated with an excess of methylene glycol.

Compound (IV)

Crystal data

$C_5H_{10}N_2O_3S$
M_r = 178.21
Monoclinic
$P2_1/c$
a = 9.088 (2) Å
b = 8.108 (2) Å
c = 10.505 (2) Å
β = 102.14 (1)°
V = 756.8 (2) Å^3
Z = 4
D_x = 1.564 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 11.1–18.2°
μ = 0.387 mm^{-1}
T = 295 K
Plate
0.55 × 0.30 × 0.05 mm
Colourless

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.910, T_{max} = 0.981
2496 measured reflections
2213 independent reflections

1396 reflections with $I > 1.2\sigma(I)$
R_{int} = 0.019
θ_{max} = 30.01°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 11$
$l = -14 \rightarrow 14$
3 standard reflections
every 150 reflections
intensity decay: 4.60%

Refinement

Refinement on F
R = 0.048
wR = 0.051
S = 1.578
1396 reflections
141 parameters
All H atoms refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: type 2, Gaussian isotropic (Zachariasen, 1968)
Extinction coefficient: 1.6 (4) × 10^{-6}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °) for (IV)*

S1—O1	1.430 (2)	S1—N1	1.655 (2)
S1—O2	1.429 (2)	S1—C4	1.766 (3)
O1—S1—O2	118.8 (1)	O2—S1—N1	106.2 (1)
O1—S1—N1	107.7 (1)	O2—S1—C4	109.3 (1)
O1—S1—C4	109.6 (1)	N1—S1—C4	104.3 (1)

Table 2. *Hydrogen-bonding geometry (Å, °) for (IV)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H8···O3^{i}	0.96 (3)	2.59 (3)	3.318 (3)	133 (2)
C4—H8···O1^{ii}	0.96 (3)	2.51 (3)	3.265 (3)	136 (2)

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

Compound (V)

Crystal data

$C_{12}H_{24}N_6O_6S_3$
M_r = 444.54
Monoclinic
$P2_1/n$
a = 14.530 (1) Å
b = 8.0567 (8) Å
c = 15.928 (2) Å
β = 101.642 (8)°
V = 1826.3 (3) Å^3
Z = 4
D_x = 1.617 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 21 reflections
θ = 10.6–16.5°
μ = 0.451 mm^{-1}
T = 295 K
Block
0.40 × 0.40 × 0.20 mm
Colourless

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5895 measured reflections
5325 independent reflections
3238 reflections with $I > 2\sigma(I)$

R_{int} = 0.019
θ_{max} = 28.0°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 10$
$l = -21 \rightarrow 21$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.038$
$wR = 0.054$
$S = 1.972$
3238 reflections
268 parameters
Only H-atom U's refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 3. *Selected geometric parameters (Å, °) for (V)*

S1—O1	1.431 (2)	S2—N3	1.629 (2)
S1—O2	1.427 (2)	S2—C6	1.782 (3)
S1—N1	1.657 (2)	S3—O5	1.435 (2)
S1—C1	1.769 (3)	S3—O6	1.432 (2)
S2—O3	1.433 (2)	S3—N6	1.654 (2)
S2—O4	1.431 (2)	S3—C12	1.770 (3)
O1—S1—O2	118.3 (1)	O4—S2—N3	106.7 (1)
O1—S1—N1	105.4 (1)	O4—S2—C6	107.4 (1)
O1—S1—C1	109.0 (1)	N3—S2—C6	109.5 (1)
O2—S1—N1	107.8 (1)	O5—S3—O6	119.2 (1)
O2—S1—C1	110.9 (1)	O5—S3—N6	107.4 (1)
N1—S1—C1	104.4 (1)	O5—S3—C12	109.9 (1)
O3—S2—O4	120.0 (1)	O6—S3—N6	106.7 (1)
O3—S2—N3	105.9 (1)	O6—S3—C12	108.0 (1)
O3—S2—C6	107.1 (1)	N6—S3—C12	104.7 (1)
C4—N3—S2—C6	77.9 (2)	S2—C6—C7—N4	102.0 (2)
N3—S2—C6—C7	−69.7 (2)	C6—C7—N4—C8	79.4 (4)

Table 4. *Hydrogen-bonding geometry (Å, °) for (V)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H2···N5^{i}	0.941	2.585	3.305 (5)	133.7
C3—H5···O3^{ii}	0.987	2.349	3.227 (4)	147.7
C10—H19···O6^{iii}	0.986	2.573	3.440 (5)	146.7
C10—H20···O4^{iv}	0.941	2.493	3.154 (5)	127.3
C11—H22···O1^{v}	0.942	2.518	3.342 (5)	146.1
C12—H23···O2^{vi}	0.903	2.551	3.427 (5)	163.5

Symmetry codes: (i) $x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$; (ii) $x, y-1, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iv) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (vi) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

The structures were solved by direct methods and Fourier techniques, with all atoms (including H atoms) observed after a series of difference syntheses. All non-H atoms were treated anisotropically. For (IV), all H atoms were treated isotropically, while for (V), H atoms were placed as found and only their U_{iso} parameters were refined. Final refinement to convergence was by full-matrix least squares. All calculations were performed on a Silicon Graphics Indy R4600 workstation.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1985); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structures: *SIR* (Burla *et al.*, 1989); program(s) used to refine structures: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

The IUPAC names of compounds (IV) and (V) were obtained using the ACD/ILAB Web service version 2.6 at http://www.acdlabs.com/ilab

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1255). Services for accessing these data are described at the back of the journal.

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para-Acetoxyacetanilide†

STUART FINNIE, ALAN R. KENNEDY, KORLAKUNTE V. R. PRASAD, RADOLJUB I. RISTIC, DAVID B. SHEEN AND JOHN N. SHERWOOD

Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow, G1 1XL, Scotland. E-mail: a.r.kennedy@ccsun.strath.ac.uk

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Abstract

para-Acetoxyacetanilide, $C_{10}H_{11}NO_3$, is a habit modifier of the analgesic *para*-hydroxyacetanilide. Its structure is compared to that of *para*-hydroxyacetanilide and other simple biologically active acetanilides. The main difference is found to be its non-planar nature; the dihedral angle between the planes of the aryl ring and the acetoxy group is 83.5 (6)°

Comment

Interest in the crystal growth properties of pharmaceutical compounds led us to investigate the use of

† Alternative name: methyl 4-acetamidobenzoate.