O4	0.3374 (6)	0.0512 (3)	0.2918 (3)	0.070 (2)
O5	0.0079 (7)	0.0046 (3)	0.2663 (4)	0.094 (3)
O6	0.2643 (7)	0.2723 (2)	0.5531 (4)	0.066 (2)
O7	0.4048 (6)	0.3020 (2)	0.6986 (3)	0.073 (2)
O8	−0.0378 (8)	0.2979 (3)	0.4428 (3)	0.074 (2)
O9	−0.2950 (7)	0.3666 (3)	0.4370 (4)	0.084 (2)
N1	−0.3029 (8)	0.2154 (3)	0.6552 (4)	0.053 (2)
C1	−0.133 (1)	0.2028 (3)	0.7180 (5)	0.050 (2)
C2	0.0191 (9)	0.1639 (3)	0.6861 (5)	0.047 (3)
C3	0.0022 (8)	0.1413 (3)	0.5902 (4)	0.042 (3)
C4	−0.1787 (9)	0.1550 (3)	0.5284 (4)	0.039 (2)
C5	−0.3289 (9)	0.1929 (3)	0.5639 (5)	0.048 (3)
C6	−0.123 (1)	0.2348 (4)	0.8157 (6)	0.078 (4)
C7	0.1854 (9)	0.1050 (3)	0.5572 (5)	0.054 (3)
C8	0.085 (1)	0.0667 (6)	0.3999 (7)	0.126 (5)
C9	−0.2115 (9)	0.1305 (4)	0.4256 (5)	0.052 (3)
C10	0.130 (1)	0.0540 (7)	0.3136 (8)	0.144 (6)
C11	0.431 (1)	−0.0103 (5)	0.2883 (7)	0.077 (4)
C12	0.3729 (1)	−0.0424 (4)	0.1932 (7)	0.083 (4)
C13	0.194 (1)	−0.0353 (4)	0.1373 (6)	0.078 (4)
C14	0.018 (1)	0.0019 (4)	0.1659 (7)	0.076 (4)
C15	0.2673 (9)	0.3069 (3)	0.6289 (5)	0.051 (3)
C16	0.099 (1)	0.3543 (4)	0.6394 (5)	0.063 (3)
C17	−0.068 (1)	0.3688 (4)	0.5793 (5)	0.064 (3)
C18	−0.138 (1)	0.3432 (4)	0.4811 (5)	0.055 (3)

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.339 (8)	C1—C2	1.374 (9)
O2—C7	1.418 (8)	C1—C6	1.491 (11)
O2—C8	1.396 (11)	C2—C3	1.391 (9)
O3—C8	1.410 (9)	C3—C4	1.405 (8)
O3—C9	1.429 (9)	C3—C7	1.509 (9)
O4—C10	1.401 (9)	C4—C5	1.375 (9)
O4—C11	1.413 (12)	C4—C9	1.494 (9)
O5—C10	1.413 (13)	C8—C10	1.275 (15)
O5—C14	1.386 (11)	C11—C12	1.481 (13)
O6—C15	1.262 (8)	C12—C13	1.327 (11)
O7—C15	1.244 (7)	C13—C14	1.461 (12)
O8—C18	1.281 (10)	C15—C16	1.483 (10)
O9—C18	1.231 (9)	C16—C17	1.327 (9)
N1—C1	1.355 (8)	C17—C18	1.477 (10)
N1—C5	1.332 (9)		
C7—O2—C8	111.9 (5)	O2—C8—O3	112.8 (8)
C8—O3—C9	114.1 (7)	O3—C8—C10	118.62 (71)
C10—O4—C11	117.7 (8)	O2—C8—C10	118.96 (77)
C10—O5—C14	114.1 (7)	O3—C9—C4	111.2 (6)
C1—N1—C5	123.4 (6)	O5—C10—C8	114.0 (8)
N1—C1—C6	116.5 (6)	O4—C10—C8	121.2 (7)
N1—C1—C2	118.1 (6)	O4—C10—O5	112.1 (6)
C2—C1—C6	125.4 (6)	O4—C11—C12	111.2 (7)
O1—C2—C1	122.6 (6)	C11—C12—C13	126.4 (8)
C1—C2—C3	120.4 (6)	C12—C13—C14	124.0 (8)
O1—C2—C3	116.9 (5)	O5—C14—C13	113.6 (7)
C2—C3—C7	117.4 (5)	O6—C15—O7	122.6 (6)
C2—C3—C4	119.1 (5)	O7—C15—C16	117.1 (6)
C4—C3—C7	123.5 (5)	O6—C15—C16	120.3 (6)
C3—C4—C9	122.3 (5)	C15—C16—C17	130.5 (7)
C3—C4—C5	118.5 (5)	C16—C17—C18	130.0 (7)
C5—C4—C9	119.2 (6)	O9—C18—C17	118.1 (7)
N1—C5—C4	120.3 (6)	O8—C18—C17	121.1 (7)
O2—C7—C3	112.6 (5)	O8—C18—O9	120.8 (7)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	*D*—H	*D*···*A*	*D*—H···*A*
O1	H1O1	$O9^{i}$	0.91 (7)	2.675 (8)	153 (6)
N1	H1*N*	O^{ii}	1.07 (7)	2.714 (7)	169 (6)
O8	H1*M*	O6	1.08 (8)	2.410 (7)	168 (8)

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

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71176 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1031]

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1-Methanesulfonyl-1a,2,6,6a-tetrahydro-1*H*,4*H*-[1,3]dioxepino[5,6-*b*]azirine†

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Abstract

The dioxepine part of the title heterocycle adopts a chair conformation. The position of O1 opposite to the lone pair at the N atom and the inequality of the bond angles O1—S1—N1 ≫ O2—S1—N1 suggest an n-σ^* interaction of

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† Chemistry of 1,3-dioxepins. Part 8. Part 7: Vinković, Dumić & Kamenar (1993).

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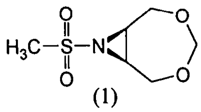
the lone-electron pair with the S^{VI}[O,O′,N,C] tetrahedral moiety.

Comment

As a part of our research on the synthesis of hypoglycemics (Dumić, Filić, Vinković, Jamnicky & Kamenar, 1993; Dumić, Butula, Vinković & Kamenar, 1992; Vinković, Dumić & Kamenar, 1992) the structure determination of the title compound (1) was undertaken in order to confirm the expected molecular structure and to study its structure-hypoglycemic activity relationships.



(1)

The dioxepine ring adopts a chair conformation having the dioxepine O atoms close to the azirine N atom (Fig. 1). The sum of the bond angles around N1 of 293° [C2—N1—S1 117.5 (2), C6—N1—S1 116.0 (2), C2—N1—C6 59.5 (2)°] indicates sp^3 hybridization of this atom. The values of the torsion angles O1—S1—N1—C2 [−27.6 (3)°] and O1—S1—N1—C6 [39.9 (2)°] show that atom O1 is positioned opposite to the lone pair at the azirine N atom (−167.2°). The position of O1 as well as the difference between the bond angles O1—S1—N1 [112.6 (1)°] and O2—S1—N1 [106.4 (2)°; difference 6.2°] suggest *n*-σ^* interaction of the lone-electron pair at the azirine N atom with the S^{VI}[O,O′,N,C] tetrahedral moiety (Kálmán, Czugler & Argay, 1981).

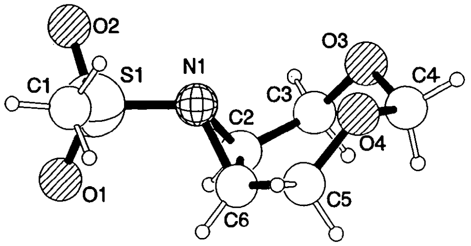


Fig. 1. *PLUTON* drawing of $C_6H_{11}NO_4S$ showing the labelling scheme.

Experimental

Crystal data

$C_6H_{11}NO_4S$
M_r = 193.22
Monoclinic
$P2_1/n$
a = 9.074 (5) Å
b = 5.452 (4) Å
c = 17.310 (9) Å
β = 91.17 (4)°
V = 856.2 (9) Å^3
Z = 4
D_x = 1.50 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 34 reflections
θ = 4–18°
μ = 0.150 mm^{-1}
T = 293 K
Needle
0.45 × 0.15 × 0.15 mm
Colourless

Data collection

Philips PW1100 diffractometer (modified by Stoe)
ω scans
Absorption correction: none
5401 measured reflections
2466 independent reflections
1239 observed reflections [$F > 3.0\sigma(F)$]

R_{int} = 0.0165
$\theta_{\max}$ = 30.0°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 24$
3 standard reflections
frequency: 60 min
intensity variation: 7.6%

Refinement

Refinement on F
Final R = 0.042
wR = 0.062
S = 0.5189
1239 reflections
120 parameters
All H-atom parameters refined
Calculated weights
$w = 1/[\sigma^2(F) + 0.01330F^2]$

$(\Delta/\sigma)_{\max}$ = 0.040
$\Delta\rho_{\max}$ = 0.276 e Å^{-3}
$\Delta\rho_{\min}$ = −0.388 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: Stoe & Cie (1992) software. Cell refinement: Stoe & Cie (1992) software. Data reduction: *REDU4S* (Stoe & Cie, 1992). Program(s) used to solve structure: *SIR*88 (Burla *et al.*, 1989). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTON* (Spek, 1982).

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

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

	x	y	z	U_{eq}
S1	0.2210 (1)	0.1872 (1)	0.5447	0.0385 (2)
O1	0.2594 (2)	0.3304 (4)	0.4780 (1)	0.0541 (8)
O2	0.2064 (3)	−0.0705 (4)	0.5335 (1)	0.0621 (7)
O3	0.5938 (2)	0.1123 (4)	0.7207 (1)	0.0505 (7)
O4	0.4336 (2)	0.3994 (4)	0.7710 (1)	0.0501 (7)
N1	0.3390 (2)	0.2243 (4)	0.6181 (1)	0.0327 (6)
C1	0.0603 (3)	0.3086 (6)	0.5840 (2)	0.0490 (9)
C2	0.4928 (3)	0.2909 (6)	0.5997 (2)	0.0431 (8)
C3	0.6166 (3)	0.1649 (7)	0.6417 (2)	0.056 (1)
C4	0.5781 (3)	0.3166 (6)	0.7686 (2)	0.052 (1)
C5	0.3925 (4)	0.5710 (6)	0.7129 (2)	0.058 (1)
C6	0.3938 (3)	0.4790 (5)	0.6312 (2)	0.0424 (8)

Table 2. *Geometric parameters* (Å, °)

S1—O1	1.442 (2)	O4—C5	1.418 (4)
S1—O2	1.424 (3)	N1—C2	1.483 (4)
S1—N1	1.657 (2)	N1—C6	1.491 (4)
S1—C1	1.752 (3)	C2—C3	1.493 (4)
O3—C3	1.417 (4)	C2—C6	1.475 (4)
O3—C4	1.398 (4)	C5—C6	1.501 (5)
O4—C4	1.388 (4)		
N1—S1—C1	100.7 (1)	C2—N1—C6	59.5 (2)
O2—S1—C1	110.5 (2)	N1—C2—C6	60.5 (2)
O2—S1—N1	106.4 (1)	N1—C2—C3	119.0 (3)
O1—S1—C1	108.8 (1)	C3—C2—C6	126.7 (3)
O1—S1—N1	112.6 (1)	O3—C3—C2	116.1 (3)
O1—S1—O2	116.6 (1)	O3—C4—O4	112.6 (3)
C3—O3—C4	115.5 (3)	O4—C5—C6	116.2 (3)
C4—O4—C5	115.4 (3)	C2—C6—C5	126.7 (3)
S1—N1—C6	116.0 (2)	N1—C6—C5	116.5 (2)
S1—N1—C2	117.5 (2)	N1—C6—C2	60.0 (2)
O1—S1—N1—C2	−27.6 (3)	O1—S1—N1—C6	39.9 (2)

This work was suported by the Ministry of Science, Technology and Informatics of the Republic of Croatia.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71175 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1030]

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Benzyl 2-[(2*R*,S*S*)-2-(Benzylaminosulfinyl)-4-oxoazetidin-1-yl]-3-methylbut-2-enoate

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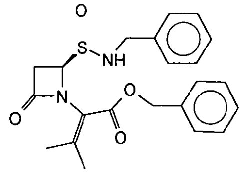
Abstract

The absolute configuration of the S atom was established as *S* using the known absolute configuration of the adjacent C atom (*R*). The 3-methylbut-2-enoate fragment is planar and it makes an angle (C3—N2—C4—C5) of −55.1 (5)° with the β-lactam ring.

Comment

The title compound (1) was synthesized and structurally characterized as part of our broader investigation of new β-lactams (Herak, Kovačević & Gašpert, 1989).



(1)

The main goal of this structure determination was to confirm the absolute configuration of the S atom (established as *S*) on the basis of the absolute configuration of C1 which was known to be *R* (Pant, Steele & Stoodley, 1982; Steele & Stoodley, 1983) (Fig. 1). The 3-methylbut-2-enoate part of the molecule is nearly planar [C5—C4—C8—O3 1.0 (6), O3—C8—O4—C9 −2.3 (5)°] while the values of the bond distances C4—C8 and C4—C5 [1.492 (4) and 1.350 (4) Å] indicate single- and double-bond character, respectively. This planar moiety lies at 58.4 (1)° to the best plane through the β-lactam ring and the torsion angle C3—N2—C4—C5 is −55.1 (5)°. The sum of the bond angles around the β-lactam nitrogen N2 is exactly 360° [C1—N2—C3 94.2 (3), C1—N2—C4 133.6 (3), C3—N2—C4 132.2 (3)° (Yang, Seiler & Dunitz, 1987; Kobal, 1991)]. The β-lactam ring is folded about a diagonal. The N2 atom is 0.07 Å out of the plane

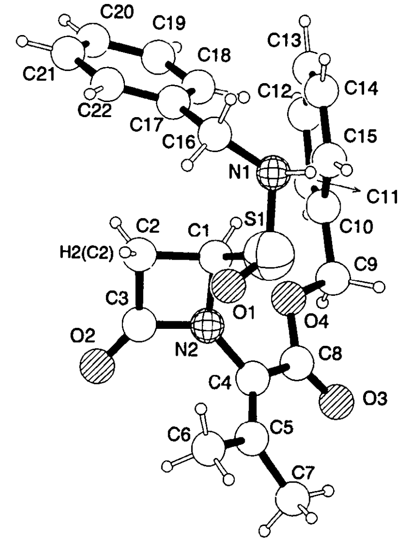


Fig. 1. *PLUTON* drawing of $C_{22}H_{24}N_2O_4S$ showing the atomic labelling scheme. H-atom labelling [except H2(C2)] omitted for clarity.

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