

Table 4. *Hydrogen bonds* (Å)Structure (I) (e.s.d.'s are *ca* 0.004 Å)

N(1')...O(12 ^b)	2.846 Å
N(1)...O(12 ^b)	2.925
O(13)...N(4 ⁱⁱ)	2.862
O(13')...O(S ^{iv})	2.724
O(S)...O(13')	2.872

Symmetry codes: (i) $1+x, y, z$; (ii) $x-1, y, z$; (iii) $1-x, 1-y, -1-z$; (iv) $2-x, 1-y, 1-z$; (v) $x, y, 1+z$.Structure (II) (e.s.d.'s are *ca* 0.005 Å)

N(1)...O(12 ^b)	2.931
O(13)...O(S ⁱⁱ)	2.823
O(S)...O(S ⁱⁱⁱ)	2.936
O(S)...O(12)	2.985

Symmetry codes: (i) $-x, -y, 1-z$; (ii) x, y, z .

(Bandoli & Clemente 1976), isopropyl alcohol solvate and isoamyl alcohol solvate, the solvent is efficient in the cohesion of the crystal. In (II) the number of hydrogen bonds between lorazepam and the solvent is greater than in the other two solvates. For all three solvates the intermolecular hydrogen bonding between N(1)—H of the reference molecule (x, y, z) and the C(2)—O(12) keto group of the molecule at ($-x, -y, -z$) for ethanol solvate, ($-x, -y, 1-z$) for (II) and ($1+x, y, z$) for (I) forms lorazepam dimers *via* a ring of eight atoms. It is interesting to note that this benzodiazepine exhibits, in addition to its capacity to form solvates, a

number of hydrogen bonds more important than those found in other analogues.

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The Structure of an Aziridine Derivative

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Abstract. 1,2-Bis(dimethylaminosulfonyl)-1,1a,1b,2-tetrahydroaziridino[1,2-*a*]cyclopropano[*c*]naphthyridene, $C_{14}H_{20}N_4O_4S_2$, $M_r = 372.5$, triclinic, $P\bar{1}$, $a = 9.171$ (2), $b = 9.918$ (2), $c = 10.699$ (2) Å, $\alpha = 84.86$ (2), $\beta = 79.57$ (1), $\gamma = 62.28$ (2)°, $V = 847.2$ Å³, $Z = 2$, $D_m = 1.45$, $D_x = 1.460$ g cm⁻³, Cu $K\alpha$, $\lambda = 1.54178$ Å, $\mu = 29.51$ cm⁻¹, $F(000) = 392$,

$T = 293$ K, $R = 0.0531$, $wR = 0.0561$ for 3100 observed reflections. The central part of the molecule is nearly planar with the cyclopropane and aziridine rings bent in opposite directions. The cyclopropane and aziridine rings make dihedral angles of 108.2 and 75.7° respectively with the naphthyridine plane.

Introduction. This investigation is the second in a series of structure determinations of bis(aziridine) and cyclopropaneaziridine derivatives obtained in the reaction of

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quinoxaline and naphthyridines with α -halocarbanions (Goliński, Makosza & Rykowski, 1983). The compound was prepared in the Institute of Organic Chemistry, Polish Academy of Science.

Experimental. Colourless crystal recrystallized from methanol by slow evaporation, D_m by flotation from aqueous KI solution. Crystal dimensions $0.39 \times 0.22 \times 0.10$ mm. Enraf-Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, no absorption correction, 3914 reflections scanned by ω - 2θ scans up to $\theta_{\max} = 75^\circ$, 3363 reflections considered observed $I > \sigma(I)$, index range $-10 \leq h \leq 10$, $-11 \leq k \leq 11$, $0 \leq l \leq 13$. Two standard reflections ($\bar{3}\bar{1}0$, 033) measured every 1 h; intensity variation $< 2\%$. Cell dimensions from measurements of 25 reflections in the range $9 < 2\theta < 22^\circ$, 3132 unique reflections ($R_{\text{int}} = 0.0303$), 3100 with $F > 3.92\sigma(F)$ used in refinement. Structure solved by direct methods with *MULTAN80* (Main *et al.*, 1980). Least-squares refinement with *SHELX76* (Sheldrick, 1976). All H atoms located from difference Fourier map and refined isotropically; non-H atoms refined anisotropically. Empirical extinction correction $F_{\text{corr}} = F(1 - gF^2/\sin\theta)$, $g = 2.16(14) \times 10^{-6}$. Final $R = 0.0531$, $wR = 0.0561$, $w = 1$, $S = 11.149$; in final cycle max. $\Delta/\sigma = 0.113$ (non-H atoms) and 0.378 (H atoms); residual electron density in final difference map within $+0.40$ and -0.46 e \AA^{-3} , atomic scattering factors as supplied with the program.

Discussion. Final atomic parameters are listed in Table 1,* bond lengths, angles and short intermolecular contacts in Table 2. The atomic numbering scheme and Newman projections are shown in Figs. 1 and 2, respectively.

The central part of the molecule is less distorted from planarity than unsubstituted 1,8-naphthyridine (Dapporto, Ghilardi, Mealli, Orlandini & Pacinotti, 1984). Except for C(9) [deviation $0.030(3)$ Å], all C atoms deviate from the least-squares plane less than $\pm 0.012(2)$ Å, while deviations for N(1) and N(2) atoms are $0.020(2)$ and $-0.024(2)$ Å, respectively. The flatness of the formally saturated part of the ring, an effect of the fusion of two three-membered rings, has previously been observed in a similar structure (Pniewska & Anulewicz, 1986).

The small rings are bent in opposite directions with respect to the naphthyridine plane making dihedral angles with it of 108.2° for the cyclopropane ring and 75.7° for the aziridine ring, with the angle between the

Table 1. Final fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$)

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
S(1)	3422.7 (8)	6824.0 (8)	3085.9 (6)	475 (2)
O(11)	1719 (2)	7882 (2)	3503 (2)	558 (7)
O(12)	3827 (3)	5291 (2)	2804 (2)	648 (7)
N(11)	4470 (3)	6740 (3)	4178 (2)	503 (7)
C(11)	6279 (4)	5764 (4)	3926 (3)	600 (10)
C(12)	3956 (5)	8102 (5)	4905 (4)	710 (10)
C(1)	3552 (3)	8383 (3)	-556 (2)	413 (7)
C(2)	4764 (3)	8800 (3)	-1159 (2)	375 (7)
N(1)	4513 (3)	9843 (2)	-2079 (2)	466 (7)
C(3)	2975 (4)	10542 (3)	-2406 (3)	514 (9)
C(4)	1709 (3)	10209 (3)	-1879 (3)	530 (10)
C(5)	2014 (3)	9091 (3)	-993 (3)	495 (9)
N(2)	6406 (2)	8160 (2)	-839 (2)	404 (6)
C(6)	3926 (3)	7189 (3)	442 (2)	448 (8)
C(7)	4177 (3)	7547 (3)	1715 (2)	421 (7)
C(8)	5630 (3)	6449 (3)	803 (2)	425 (8)
C(9)	6873 (3)	6927 (3)	118 (2)	414 (7)
C(10)	7468 (3)	6555 (3)	-1247 (2)	413 (7)
S(2)	9561.2 (8)	6018.8 (8)	-1937.3 (6)	455 (2)
O(21)	10347 (3)	6443 (3)	-1130 (2)	681 (8)
O(22)	10224 (3)	4453 (2)	-2264 (2)	587 (7)
N(22)	9532 (3)	6973 (3)	-3244 (2)	501 (7)
C(21)	9353 (7)	8503 (5)	-3201 (5)	830 (20)
C(22)	8654 (6)	6818 (6)	-4183 (4)	770 (10)

Table 2. Bond lengths (Å) and angles ($^\circ$) with e.s.d.'s in parentheses

C(1)–C(2)	1.394 (3)	N(2)–C(10)	1.484 (3)
C(1)–C(6)	1.479 (3)	N(11)–C(11)	1.468 (4)
C(1)–C(5)	1.380 (3)	N(11)–C(12)	1.455 (4)
C(3)–C(4)	1.364 (4)	N(22)–C(21)	1.452 (5)
C(4)–C(5)	1.381 (4)	N(22)–C(22)	1.455 (4)
C(6)–C(7)	1.520 (3)	S(1)–C(7)	1.746 (2)
C(6)–C(8)	1.494 (4)	S(1)–O(11)	1.431 (2)
C(7)–C(8)	1.511 (3)	S(1)–O(12)	1.435 (2)
C(8)–C(9)	1.483 (3)	S(1)–N(11)	1.616 (2)
C(9)–C(10)	1.479 (3)	S(2)–C(10)	1.767 (2)
N(1)–C(2)	1.331 (3)	S(2)–O(21)	1.420 (2)
N(1)–C(3)	1.347 (3)	S(2)–O(22)	1.430 (2)
N(2)–C(2)	1.431 (3)	S(2)–N(22)	1.614 (2)
N(2)–C(9)	1.475 (3)		
C(2)–C(1)–C(5)	117.5 (2)	C(8)–C(6)–C(7)	60.2 (2)
C(2)–C(1)–C(6)	121.0 (2)	C(2)–N(11)–C(3)	116.7 (2)
C(5)–C(1)–C(6)	121.5 (2)	C(2)–N(2)–C(9)	118.2 (2)
C(1)–C(2)–N(1)	123.7 (2)	C(2)–N(2)–C(10)	113.4 (2)
C(1)–C(2)–N(2)	123.3 (2)	C(9)–N(2)–C(10)	60.0 (2)
N(1)–C(2)–N(2)	112.9 (2)	C(11)–N(11)–C(12)	114.6 (3)
N(1)–C(3)–C(4)	124.0 (3)	C(11)–N(11)–S(1)	116.8 (2)
C(3)–C(4)–C(5)	118.2 (3)	C(12)–N(11)–S(1)	118.9 (2)
C(1)–C(5)–C(4)	119.7 (3)	C(21)–N(22)–C(22)	112.8 (3)
N(2)–C(10)–C(9)	59.7 (1)	C(21)–N(22)–S(2)	119.1 (3)
N(2)–C(10)–S(2)	117.7 (2)	C(22)–N(22)–S(2)	117.1 (2)
C(9)–C(10)–S(2)	121.6 (2)	C(7)–S(1)–O(12)	106.5 (1)
N(2)–C(9)–C(10)	60.3 (2)	C(7)–S(1)–O(11)	108.8 (1)
N(2)–C(9)–C(8)	120.8 (2)	C(7)–S(1)–N(11)	107.3 (1)
C(8)–C(9)–C(10)	118.0 (2)	O(12)–S(1)–N(11)	107.4 (3)
C(7)–C(8)–C(9)	118.9 (2)	O(11)–S(1)–N(11)	107.2 (1)
C(6)–C(8)–C(9)	118.1 (2)	O(11)–S(1)–O(12)	119.1 (1)
C(6)–C(8)–C(7)	60.8 (2)	C(10)–S(2)–O(22)	104.4 (1)
C(6)–C(7)–C(8)	59.0 (2)	C(10)–S(2)–O(21)	109.9 (1)
S(1)–C(7)–C(8)	118.8 (2)	C(10)–S(2)–N(22)	107.4 (1)
S(1)–C(7)–C(6)	117.9 (2)	O(22)–S(2)–N(22)	107.6 (1)
C(1)–C(6)–C(8)	118.5 (2)	O(21)–S(2)–N(22)	107.3 (1)
C(1)–C(6)–C(7)	118.2 (2)	O(21)–S(2)–O(22)	119.6 (1)

Short intermolecular contacts with symmetry codes

$A \cdots B$	$A \cdots B(\text{\AA})$	$A \cdots H(\text{\AA})$	$A \cdots H-B(^\circ)$	
O(12) \cdots H(10)–C(10)	3.290 (4)	2.31 (3)	175 (2)	– <i>x</i> , – <i>y</i> , – <i>z</i>
N(1) \cdots H(7)–C(7)	3.397 (4)	2.58 (3)	148 (2)	– <i>x</i> , 1– <i>y</i> , – <i>z</i>

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44156 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

three-membered rings being $123.1(4)^\circ$. The aziridine ring is an equilateral triangle within experimental error, while the cyclopropane ring shows only slight deviation from this. The bond length C(10)—S(2) between the sulfonyl group and aziridine [$1.767(2)\text{ \AA}$] is greater than the corresponding C(7)—S(1) bond for cyclopropane [$1.746(2)\text{ \AA}$]. All the exocyclic angles at the cyclopropane atoms are very close to the average value of $118.4(2)^\circ$.

The three-membered rings show very different orientations with respect to the sulfonyl groups (Figs. 2*c,d*) though the orientations of the remaining substituents of the sulfonamide groups are uniform (Figs. 2*a,b*). The sulfonamide groups are drawn in opposite directions. In the phenylsulfonylquinoxaline derivative with two aziridine fragments (Pniewska & Anulewicz, 1986) both substituents point in the same direction. Newman projections of respective enantiomers (Figs. 2*e,f*) show that the conformation of the aziridine part of the present molecule is similar to that in the previous structure while the substituent connected to the cyclopropane ring points in the opposite direction.

Short intermolecular aziridine C—H...O and cyclopropane C—H...N contacts link pairs of molecules related by centres of symmetry (Table 2).

The pyramidal conformation of the sulfonamide N atoms is appreciably flatter than that for the aziridine N(2) atom; the distances of N(11), N(22) and N(2) from the planes of their three substituents are $0.274(3)$, $0.291(2)$ and $0.672(2)\text{ \AA}$, respectively. Partial flattening of both amine groups is probably caused by π -electron interactions between these groups and the electron-accepting sulfone groups.

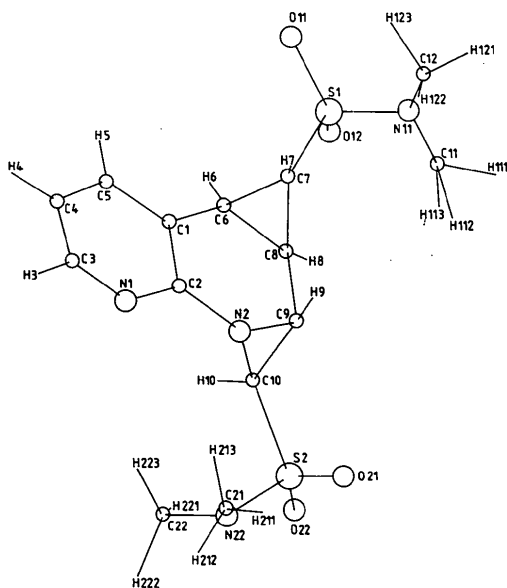


Fig. 1. A view of the molecule with the numbering of the atoms.

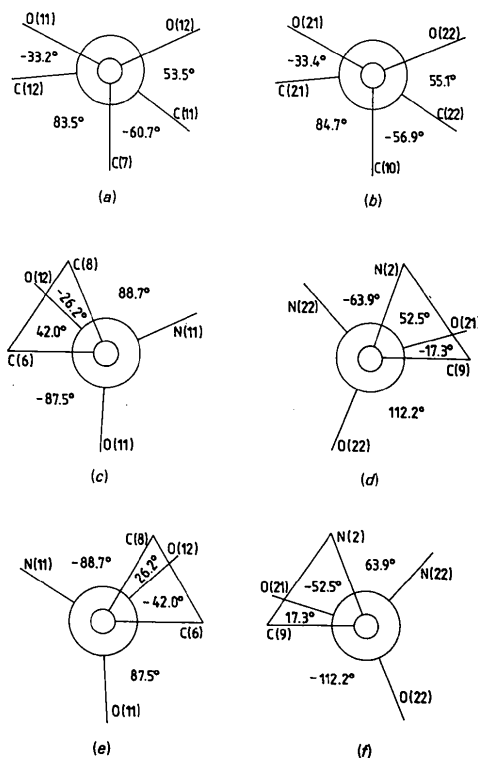


Fig. 2. Newman projection along the bonds: (a) S(1)—N(11), (b) S(2)—N(22), (c) C(7)—S(1), (d) C(10)—S(2), (e) C(7)—S(1), (f) C(10)—S(2). (e) and (f) are for enantiomers. E.s.d.'s are 0.3° .

The H atoms at C(6), C(7), C(8), C(9), and C(10) show a *trans* conformation; the compound forms racemic crystals.

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