Tabelle 3. *Bindungslängen (Å) und -winkel (°) am Stickstoff in einigen Methansulfonsäure- und Sulfonsäurederivaten des Ammoniaks und Hydroxylamins*

	N–S[a]	N–O	Winkelsumme am Stickstoff
$CH_3SO_2N(H)OH$[b]	1,664 (3)	1,437 (3)	320 (2)
$(CH_3SO_2)_2NOSO_2CH_3$[c]	1,734 (2)	1,430 (2)	339,0 (1)
$K[SO_3N(H)OH]$[d]	1,69 (1)	1,48 (2)	[e]
$K_2[SO_3N(H)OSO_3]$[f]	1,704 (3)	1,454 (4)	304 (2)
$K_3[(SO_3)_2NOSO_3].1,5H_2O$[g]	1,750 (2)	1,434 (1)	330,6 (1)
$CH_3SO_2NH_2$[h]	1,61 (2)		[e]
$(CH_3SO_2)_2NH.H_2O$[i]	1,645 (1)		360
$K[SO_3NH_2]$[j]	1,666 (6)		330,5 (5)
$K_2[(SO_3)_2NH]$[k]	1,669 (2)		346 (3)
$K_3[(SO_3)_3N].2H_2O$[l]	1,71 (2)		360 (1)

Anmerkungen: (*a*) Mittelwerte; (*b*) Brink & Mattes (1986); (*c*) diese Arbeit; (*d*) Pant & Cruickshank (1966); (*e*) H-Atom(e) nicht lokalisiert; (*f*) Hall, Johnson, Kennard, Smith, Skelton & White (1980); (*g*) Brown & Strydom (1977); (*h*) Vorontsova (1966); (*i*) Attig & Mootz (1975); (*j*) Cox, Sabine, Padmanabhan, Ban, Chung & Surjadi (1967); (*k*) Barbier, Parent & Mairesse (1979); (*l*) Tillack & Kennard (1970).

Mattes, 1985) und Aminen (Vorontsova, 1966; Klug, 1968; Naumov, Garaeva & Butenko, 1979; Attig & Mootz, 1975) keine Besonderheiten.

Vergleicht man die Strukturen der Mesylhydroxylamine mit denen der Mesylamine und denen der Sulfonate des Ammoniaks und Hydroxylamins, so lassen sich folgende Gesetzmäßigkeiten erkennen (vgl. Tabelle 3): Wie bei den Sulfonaten des Ammoniaks und Hydroxylamins mit steigender Anzahl an Sulfonatgruppen führt auch bei den Mesylaminen und Mesylhydroxylaminen die zunehmende Substitution durch Mesylgruppen zu einer Verlängerung der N–S-Bindung und zu einer Verringerung der Pyramidalität am Stickstoffatom. Bei den Hydroxylaminderivaten geht die Verlängerung der N–S-Bindung mit einer Verkürzung der N–O-Bindung konform. Sulfonate des Ammoniaks und Hydroxylamins besitzen in der Regel längere N–S-Bindungen und eine größere Pyramidalität am Stickstoffatom als analoge Mesylamine bzw. Mesylhydroxylamine.

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Acta Cryst. (1986). C42, 1627–1630

The Structure of 1,2-Bis(phenylsulfonyl)-1,1a,1b,2-tetrahydrodiaziridino[1,2-*a*:2,1-*c*]quinoxaline, an Aziridine Derivative

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Abstract. $C_{22}H_{18}N_2O_4S_2$, $M_r = 438\cdot6$, monoclinic, $P2_1/c$, $a = 13\cdot380$ (2), $b = 17\cdot319$ (2), $c = 9\cdot173$ (2) Å, $\beta = 101\cdot14$ (2)°, $V = 2085\cdot59$ Å^3, $Z = 4$, $D_m = 1\cdot32$ (3), $D_x = 1\cdot396$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 2\cdot39$ cm^{-1}, $F(000) = 912$, $T = 293$ K, $R = 0\cdot0513$, $wR = 0\cdot0415$ for 1893 observed reflections. The molecule exhibits approximate C_2 symmetry with non-equivalent deformations at sulfur and C-aziridinic atoms.

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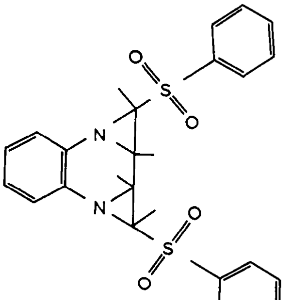
Introduction. The synthesis and properties of the title compound have been published previously (Goliński, Makosza & Rykowski, 1983). This investigation was undertaken to determine the geometry of the aziridine ring fusion and the conformation of the H atoms. Structural studies of other compounds with aziridine fragments have been reported (Allen, 1982; Brückner, Malpezz, Prosyanik & Bondarenko, 1985). The structural formula is indicated below.



Experimental. Colourless rectangular prisms were recrystallized from acetic acid by slow evaporation. D_m determined by flotation from KI solution. The space group uniquely defined from Weissenberg photographs by systematic absences $h0l$: $l = \text{odd}$, $0k0$: $k = \text{odd}$. Crystal $0{\cdot}13 \times 0{\cdot}13 \times 0{\cdot}26$ mm used for diffractometer measurement. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$, graphite monochromator. Cell dimensions from refinement of 25 reflections in the range $6{\cdot}3 \leq \theta \leq 13{\cdot}3°$, 4353 measured reflections, $\theta_{\max} = 26°$, 2273 reflections were considered as observed $[|F_o| \geq 3\sigma|F_o|]$, ω–2θ scan with $\omega = 90° + 0{\cdot}40° \tan\theta$, one standard reflection ($4\bar{1}0$) monitored every 1 h, no absorption correction. Index range $-17 \leq h \leq 17$, $0 \leq k \leq 22$, $0 \leq l \leq 12$. 2150 unique reflections, 1983 with $I_o > 3\sigma(I_o)$ used in refinement ($R_{\text{int}} = 0{\cdot}0538$). Structure was solved by direct methods using *SHELX76* (Sheldrick, 1976). All non-H atoms located in the best E map and subsequent difference map revealed positions of H atoms. Least-squares anisotropic refinement of positions of non-H atoms, isotropic refinement of all H atoms gave final $R(F) = 0{\cdot}0513$, $wR = 0{\cdot}0415$, $w = 2{\cdot}2543[\sigma^2(F_o) + 0{\cdot}000124(F_o)^2]^{-1}$; $\Delta/\sigma_{\max} = 0{\cdot}075$, residual density within max + 0·28, min −0·34 e Å^{-3}; atomic scattering factors as supplied by the program.

Table 1. *Atomic positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

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

	x	y	z	U_{eq}
C(1)	4565 (3)	1237 (3)	−3167 (5)	41 (3)
C(2)	5458 (3)	952 (2)	−2354 (5)	39 (3)
C(3)	6224 (4)	705 (3)	−3051 (6)	55 (4)
C(4)	6097 (5)	756 (4)	−4564 (7)	69 (4)
C(5)	5200 (5)	1030 (4)	−5396 (7)	64 (4)
C(6)	4440 (4)	1279 (3)	−4694 (6)	54 (4)
C(7)	3182 (3)	947 (3)	−1838 (5)	42 (3)
C(8)	3879 (3)	1483 (3)	−886 (5)	41 (3)
C(9)	4852 (3)	1182 (3)	−2 (5)	37 (3)
C(10)	5783 (3)	1639 (3)	22 (5)	35 (3)
N(1)	3751 (2)	1533 (2)	−2506 (4)	42 (2)
N(2)	5647 (2)	898 (2)	−759 (4)	40 (2)
S(1)	1852 (1)	1108 (1)	−2235 (1)	48 (1)
O(11)	1384 (2)	558 (2)	−1402 (3)	62 (2)
O(12)	1676 (2)	1913 (2)	−2033 (4)	63 (2)
C(11)	1544 (3)	883 (3)	−4140 (5)	43 (3)
C(12)	1356 (4)	127 (3)	−4584 (7)	56 (4)
C(13)	1156 (4)	−54 (4)	−6062 (8)	65 (4)
C(14)	1155 (4)	515 (4)	−7097 (8)	70 (5)
C(15)	1353 (5)	1267 (4)	−6672 (8)	76 (5)
C(16)	1549 (4)	1451 (3)	−5184 (8)	58 (4)
S(2)	6768 (1)	1686 (1)	1598 (1)	39 (1)
O(21)	6862 (2)	937 (2)	2286 (3)	50 (2)
O(22)	6555 (2)	2344 (2)	2435 (3)	51 (2)
C(21)	7855 (3)	1867 (3)	843 (4)	35 (3)
C(22)	8149 (3)	2615 (3)	646 (6)	53 (4)
C(23)	8984 (4)	2737 (4)	1 (7)	68 (4)
C(24)	9505 (4)	2130 (4)	−441 (6)	61 (4)
C(25)	9203 (4)	1397 (4)	−224 (6)	59 (4)
C(26)	8372 (4)	1252 (3)	399 (5)	52 (4)

Final atomic parameters are given in Table 1,* bond lengths and angles in Table 2. Fig. 1 shows the atomic numbering, and Fig. 2 a projection of the structure on the *ab* plane.

All calculations performed with *SHELX76* system of programs (Sheldrick, 1976) on the RIAD-40 computer. The equations of the least-squares plane, the torsion angles and the other geometrical data were calculated using the *PLANE* (Jaskólski, 1981*a*) and *GEOME* (Jaskólski, 1981*b*) programs. The thermal-motion analysis used the *THMB*-6 program (Trueblood, 1982).

Discussion. The title compound forms racemic crystals with two pairs of enantiomers in the unit cell. The six-membered ring (*P*-ring) formed by C(1), C(2), N(1), N(2), C(8), C(9) is nearly flat and coplanar with the benzene ring fused to it. Deviations of C(8), C(9), N(1), and N(2) from the benzene ring plane are less than 0·061 (5) Å; sp^3-hybridized carbon atoms C(7) and C(10) deviate from this plane by −1·166 (5) and 1·265 (5) Å respectively. Except for these two atoms and the attached thio groups the remainder of the molecule exhibits C_2 symmetry within a 3σ range of bond lengths and angles.

The aziridine rings are regular triangles with all internal angles of 60·0 (3)°, analogous to those in other aziridine rings (Allen, 1982). In these triangles the average C—C 1·473 (6) and N—C 1·466 (6) Å bond distances are not very different from those found in other aziridine compounds [mean values C—C 1·484 (3), N—C 1·474 (4) Å (Allen, 1982)]. The dihedral angles between the best mean plane of the

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* 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 43094 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

P-ring and aziridine rings are 106·6 (5)° for the N(1) and 74·9 (5)° for the N(2) ring, and between both aziridine rings the dihedral angle is 122·5 (4)°. The N(1) and N(2) atoms are displaced 0·667 (3) and 0·665 (3) Å out of the planes of their three substituents. The H atoms at C(7), C(8), C(9), C(10) show a *trans* conformation and with the pyramidal conformation of both nitrogen atoms also conform to C_2 symmetry.

The phenyl rings are each planar to within ±0·006 (7) Å and have normal bonds and angles. The dihedral angle between the mean planes of the phenyl rings amounts to 117·5 (8)° and the dihedral angles between the best *P*-ring plane and phenyl rings linked through S(1) and S(2) are 102·2 (8) and 74·9 (8)° respectively.

The O atoms of the sulfonyl groups are almost symmetrically oriented with respect to the phenyl rings as indicated by the torsion angles in Fig. 3(*a,b*). However, orientations of the phenylsulfonyl groups with respect to the neighboring aziridine rings are markedly different. The bonds around S(2) are more distorted from tetrahedral than are those around S(1). The torsion angles around corresponding bonds C(7)—S(1) and C(10)—S(2) differ by 14·4 (4)° (Fig. 3*c,d*). The exocyclic angles N(1)—C(7)—S(1) and N(2)—C(10)—S(2) differ by 13σ. These deviations may be explained by the three short intermolecular contacts for the O(21), O(22) oxygen atoms of the S(2) sulfonyl group compared to the two short intermolecular contacts only for O(11), O(12) oxygen atoms of the S(1) sulfonyl group (Table 2). Some of these contacts are expressed by dotted lines in Fig. 2.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Bond lengths corrected for rigid-body motion are given alongside their uncorrected values calculated by use of *THMB*-6 (Trueblood, 1982).

		Corrected			Corrected
S(1)–C(7)	1·768 (5)	1·770	S(2)–C(10)	1·760 (4)	1·762
S(1)–O(11)	1·439 (3)	1·440	S(2)–O(21)	1·437 (3)	1·439
S(1)–O(12)	1·433 (3)	1·434	S(2)–O(22)	1·433 (3)	1·435
S(1)–C(11)	1·760 (5)	1·763	S(2)–C(21)	1·756 (4)	1·758
N(1)–C(1)	1·439 (6)	1·442	N(2)–C(2)	1·439 (5)	1·441
N(1)–C(7)	1·473 (6)	1·474	N(2)–C(10)	1·464 (6)	1·466
N(1)–C(8)	1·465 (6)	1·468	N(2)–C(9)	1·463 (6)	1·465
C(1)–C(2)	1·372 (6)	1·373	C(1)–C(6)	1·380 (7)	1·382
C(2)–C(3)	1·377 (8)	1·379	C(3)–C(4)	1·376 (8)	1·377
C(4)–C(5)	1·376 (8)	1·377	C(5)–C(6)	1·374 (9)	1·374
C(7)–C(8)	1·475 (6)	1·478	C(8)–C(9)	1·490 (6)	1·490
C(9)–C(10)	1·472 (6)	1·472	C(11)–C(12)	1·380 (7)	1·381
C(11)–C(16)	1·373 (8)	1·376	C(12)–C(13)	1·367 (10)	1·369
C(13)–C(14)	1·369 (10)	1·370	C(14)–C(15)	1·371 (9)	1·372
C(15)–C(16)	1·376 (10)	1·379	C(21)–C(22)	1·376 (8)	1·378
C(21)–C(26)	1·375 (7)	1·377	C(22)–C(23)	1·377 (8)	1·380
C(23)–C(24)	1·366 (9)	1·367	C(24)–C(25)	1·356 (9)	1·358
C(25)–C(26)	1·374 (7)	1·375			

C(1)–C(2)–C(3)	120·5 (4)	C(2)–C(3)–C(4)	119·6 (5)
C(3)–C(4)–C(5)	120·6 (6)	C(4)–C(5)–C(6)	119·5 (6)
C(5)–C(6)–C(1)	120·2 (5)	C(6)–C(1)–C(2)	119·5 (5)
C(11)–C(12)–C(13)	120·0 (6)	C(12)–C(13)–C(14)	119·8 (6)
C(13)–C(14)–C(15)	120·9 (7)	C(14)–C(15)–C(16)	119·4 (6)
C(15)–C(16)–C(11)	120·0 (5)	C(16)–C(11)–C(12)	119·9 (5)
C(21)–C(22)–C(23)	118·5 (5)	C(22)–C(23)–C(24)	120·8 (6)
C(23)–C(24)–C(25)	119·8 (6)	C(24)–C(25)–C(26)	121·2 (6)
C(25)–C(26)–C(21)	118·5 (5)	C(26)–C(21)–C(22)	121·2 (4)
S(1)–C(7)–C(8)	121·2 (4)	S(2)–C(10)–C(9)	122·3 (3)
S(1)–C(7)–N(1)	112·7 (3)	S(2)–C(10)–N(2)	116·5 (3)
S(1)–C(11)–C(12)	119·8 (4)	S(2)–C(21)–C(22)	120·0 (4)
S(1)–C(11)–C(16)	120·1 (4)	S(2)–C(21)–C(26)	118·7 (4)
N(1)–C(1)–C(2)	123·3 (4)	N(2)–C(2)–C(1)	122·7 (4)
N(1)–C(1)–C(6)	117·1 (4)	N(2)–C(2)–C(3)	116·8 (4)
N(1)–C(7)–C(8)	59·6 (3)	N(2)–C(10)–C(9)	59·8 (3)
N(1)–C(8)–C(7)	60·1 (3)	N(2)–C(9)–C(10)	59·8 (3)
N(1)–C(8)–C(9)	119·4 (4)	N(2)–C(9)–C(8)	119·9 (4)
C(7)–N(1)–C(1)	115·0 (3)	C(10)–N(2)–C(2)	114·9 (3)
C(8)–N(1)–C(1)	117·2 (3)	C(9)–N(2)–C(2)	117·3 (3)
C(8)–N(1)–C(7)	60·3 (3)	C(9)–N(2)–C(10)	60·4 (3)
O(11)–S(1)–C(7)	108·1 (2)	O(21)–S(2)–C(10)	107·8 (2)
O(12)–S(1)–C(7)	108·2 (2)	O(22)–S(2)–C(10)	106·4 (2)
O(12)–S(1)–O(11)	118·4 (2)	O(22)–S(2)–O(21)	119·5 (2)
C(11)–S(1)–C(7)	101·7 (2)	C(21)–S(2)–C(10)	103·3 (2)
C(11)–S(1)–O(11)	109·8 (2)	C(21)–S(2)–O(21)	109·0 (2)
C(11)–S(1)–O(12)	109·3 (2)	C(21)–S(2)–O(22)	109·6 (2)

Short intermolecular contacts with symmetry codes.

A···H–*B*	*A*···*B* (Å)	*A*···H (Å)	
O(11)···H(26)–C(26)	3·265 (6)	2·49 (4)	$x, 1-y, -z$
O(12)···H(15)–C(15)	3·206 (8)	2·51 (7)	$x, \frac{1}{2}-y, \frac{1}{2}+z$
O(21)···H(4)–C(4)	3·262 (7)	2·53 (5)	$x, y, 1-z$
O(21)···H(7)–C(7)	3·288 (6)	2·47 (4)	$x, 1-y, -z$
O(22)···H(10)–C(10)	3·279 (6)	2·50 (4)	$x, \frac{1}{2}-y, \frac{1}{2}+z$

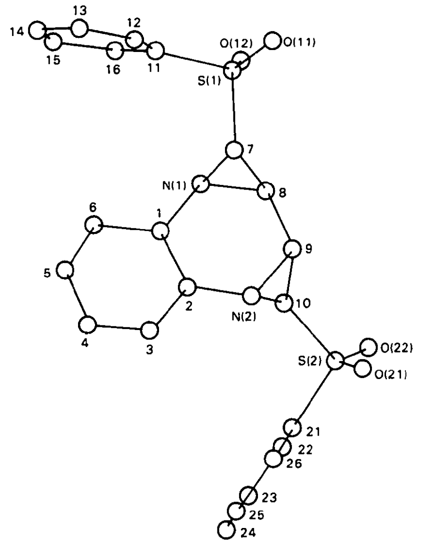

Fig. 1. A view of the molecule with the numbering of the atoms.

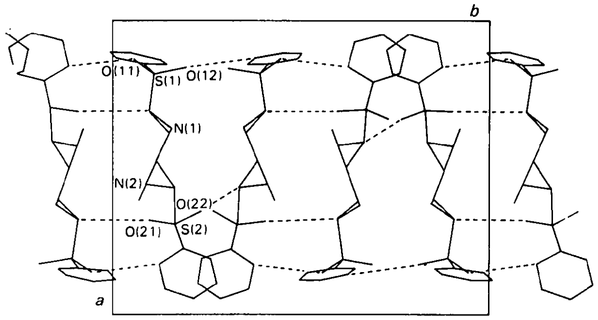

Fig. 2. Projection of the structure on the *ab* plane.

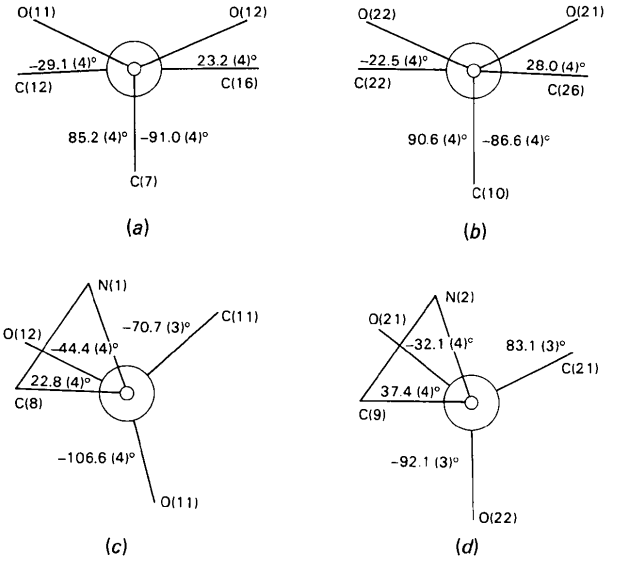

Fig. 3. Newman projections along the bonds: (*a*) S(1)—C(11), (*b*) S(2)—C(21), (*c*) C(7)—S(1), (*d*) C(10)—S(2).

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Acta Cryst. (1986). C**42**, 1630–1632

Epoxy-8,16 Dihydro-8,16 Dinaphto[2,1-*b*:2′,1′-*f*][dioxocinne-1,5]

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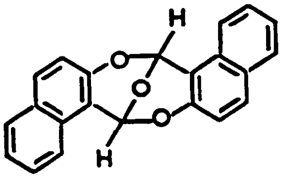
Abstract. $C_{22}H_{14}O_3$, synthetic compound, $M_r = 326\cdot4$, tetragonal, $P4_12_12$, $a = 11\cdot069$ (5), $c = 12\cdot418$ (5) Å, $V = 1521$ (2) Å^3, $Z = 4$, $D_x = 1\cdot425$ (2) g cm^{-3}, $\lambda(\text{Ag } K\alpha) = 0\cdot56083$ Å, $\mu = 0\cdot7$ cm^{-1}, $F(000) = 680$, $T = 293$ K, $R = 0\cdot058$ for 1158 unweighted reflections. Molecules have a twofold axis through the O(1) atoms. The geometries of the dioxocin and epoxy moieties in this compound and in 3,4,7,8-tetramethyl-*trans*-3,8;4,7-diepoxy-1,5-dioxocane are discussed. Bond lengths are shorter and bond angles greater in the title compound, owing to the presence of the naphthyl groups.

Introduction. Ce composé a été préparé en portant à ébullition une solution acétique d'*O*-hydroxy α-naphtalène en présence d'acide perchlorique anhydre (Andrieux, Bodo & Mohlo, 1973). C'est un produit de condensation, incolore, dont l'obtention a été aussi signalée au cours de synthèses de spirochromènes à partir d'une cétone R—CH_2—CO—CH_2—R_1 et d'hydroxy-2 α-naphtaldéhyde-1 (Dzhaparidze, Chubabriya & Mzhavanadze, 1976). L'étude structurale a été entreprise pour connaître la conformation non encore élucidée à notre connaissance d'un cycle dioxocinne avec un seul pont époxyde. Ce produit est d'autre part voisin d'un autre composé de condensation (dibromo-2,8 époxy-6,12 dihydro-6,12 diméthoxy-4,10 dibenzo[*b*,*f*][dioxocinne-1,5]) qui a montré des propriétés antidermatophytes et antibactériennes (Fiedler, 1964).



Partie expérimentale. Cristaux incolores, $F = 515$ K, 0,2 × 0,2 × 0,4 mm. Paramètres cristallins obtenus sur diffractomètre automatique Philips PW 1100 avec 25

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