

which with $F_o^2 > 3.0\sigma(F_o^2)$ were considered observed. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1990) and least-squares refined on F with the *SHELX76* (Sheldrick, 1976) program. The H atoms were located and included in Fourier summations. 104 parameters were refined to $R = 0.047$, $wR = 0.048$ and $S = 0.34$ with $w = [\sigma^2(F) + 0.045F^2]^{-1}$; max. and min. heights in final $\Delta\rho$ map are $0.54(3)$ and $-0.84(3)$ e Å⁻³, respectively, $(\Delta/\rho)_{\max} = 0.008$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV), except for H atoms which were taken from Stewart, Davidson & Simpson (1965).

Final atomic coordinates and isotropic or equivalent isotropic displacement parameters are listed in Table 1.* Bond distances and angles are given in Table 2. All geometric calculations were calculated with program *PLATON* (Spek, 1990). A perspective drawing of one of the molecules is shown in Fig. 1 giving the atomic numbering scheme.

Each chloride ion accepts two hydrogen bonds: N(3)—H(31)⋯Cl(1) with angle $159(2)^\circ$ and N(3)—

H(32)⋯Cl(1) with angle $162(2)^\circ$. The N(3)⋯Cl(1) distances are $3.090(2)$ and $3.072(2)$ Å, respectively. The effects of hydrogen bonding on the puckering angle seems to cancel out in this system owing to the symmetric forces on the N atom.

Related literature. This structure determination is part of an investigation of the conformational properties of the azetidinol system (Gajhede *et al.*, 1989; Hagen, Volden, Gajhede, Anthoni, Christophersen & Nielsen, 1991).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55670 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1008]

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Structure and Configuration of the Michael Adduct of 2-(Methylthio)thiolane S-Oxide and Butenone at 173 K*

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Abstract. (*E*)-2-Methylthio-2-(3-oxobutyl)thiolane 1-oxide, C₉H₁₆O₂S₂, $M_r = 220.36$, orthorhombic, $P2_1ab$ (No. 29, non-standard), $a = 7.737(3)$, $b =$

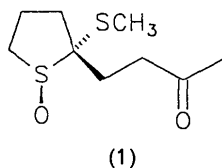
$9.533(2)$, $c = 14.725(5)$ Å, $V = 1086.1(6)$ Å³, $Z = 4$, $D_x = 1.348$ g cm⁻³, $\lambda(\text{Cu } K\alpha_1) = 1.54051$ Å, $\mu = 41.3$ cm⁻¹, $F(000) = 472$, $T = 173$ K, $R = 0.063$ for 990 observed reflections [$|F_o| \geq 4\sigma(F_o)$]. The thiolane ring in (1) exhibits a half-chair conformation with quasi-axial positions of the sulfoxide O atom and the methylthio group. The *E* (*trans*) isomer (1) is the

* C,C-Coupling with Sulfur-Stabilized Carbanions. 3. Part 2: Böge, Brunck, Schwär & Voss (1992).

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main product from the Michael addition of 2-(methylthio)thiolane *S*-oxide and 2-butenone. Its geometric structure could not be determined by spectroscopic methods; therefore, an X-ray structure analysis was indispensable and afforded the required unambiguous information despite its rather moderate precision. The predominant formation of (1) can be rationalized in terms of steric repulsion and the anomeric effect, *i.e.* dipole compensation of the ring substituents.

Experimental. The ketone (1) was formed by deprotonation of 2-(methylthio)thiolane *S*-oxide with butyllithium and subsequent reaction with butenone.



A crystal suitable for X-ray analysis (colourless block, $0.5 \times 0.5 \times 0.4$ mm) was obtained by slow crystallization from ethyl acetate/trichloromethane/hexane. The data were collected at 173 K using graphite-monochromated Cu $K\alpha_1$ radiation on an Enraf-Nonius CAD-4 diffractometer. Lattice parameters were obtained from 22 reflections with $30 < \theta < 47^\circ$. Data were collected using the $\theta/2\theta$ scan mode [1042 symmetry-independent reflections, 990 observed reflections with $|F_o| > 4\sigma(F_o)$] with a 2θ range of $4.5\text{--}153^\circ$ ($h\ 0 \rightarrow 9$, $k\ 0 \rightarrow 12$, $l\ 0 \rightarrow 18$). Three reflections (600, 180 and $2,3,12$) were remeasured every 500 reflections to monitor instrument and crystal stability. The structure was solved by direct methods and refined by full-matrix least-squares methods. The non-H atoms were refined with anisotropic parameters. All H atoms were placed in theoretical positions (sp^3 hybridization) with a C—H distance of 0.96 Å.

In all, 120 parameters were refined; the ratio reflections/parameters was 8.25. Final $R = 0.063$ for 990 reflections, $wR = 0.082$ $\{w = [\sigma^2(F) + 0.0002F^2]^{-1}\}$. Scattering factors were taken from the program system *SHELXTL-Plus* (Sheldrick, 1990). An empirical absorption correction was performed. Atomic parameters and equivalent isotropic displacement coefficients are listed in Table 1. Selected bond lengths, bond angles and torsion angles for the non-H atoms are compiled in Table 2. A drawing of

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters, and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55864 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1014]

Table 1. Atomic coordinates ($\times 10^5$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^4$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
S(1)	66750 (41)	112461 (15)	10129 (9)	414 (5)
S(2)	75000	88818 (14)	22092 (11)	443 (5)
O(1)	61659 (117)	127490 (47)	10430 (25)	486 (17)
O(2)	68341 (143)	132518 (53)	43776 (33)	728 (25)
C(2)	61641 (132)	104273 (74)	21423 (38)	404 (20)
C(3)	42869 (144)	99592 (79)	20544 (41)	477 (25)
C(4)	40550 (146)	93114 (78)	11171 (41)	460 (22)
C(5)	49141 (146)	103234 (82)	4529 (39)	517 (26)
C(21)	64894 (145)	115184 (67)	28765 (36)	415 (21)
C(22)	60249 (164)	110107 (69)	38278 (42)	490 (24)
C(23)	62805 (166)	120970 (78)	45498 (42)	548 (27)
C(24)	58564 (199)	116651 (90)	55132 (44)	718 (35)
C(26)	96582 (142)	96190 (83)	23098 (48)	473 (24)

Table 2. Selected bond lengths (Å), bond angles ($^\circ$) and torsion angles ($^\circ$)

S(1)—O(1)	1.487 (5)	S(1)—C(2)	1.879 (6)
S(1)—C(5)	1.819 (10)	S(2)—C(2)	1.802 (8)
S(2)—C(26)	1.818 (11)	O(2)—C(23)	1.208 (10)
C(2)—C(3)	1.525 (15)	C(2)—C(21)	1.521 (9)
C(3)—C(4)	1.523 (9)	C(4)—C(5)	1.526 (11)
C(21)—C(22)	1.525 (9)	C(22)—C(23)	1.497 (10)
C(23)—C(24)	1.513 (10)		
O(1)—S(1)—C(2)	108.6 (3)	O(1)—S(1)—C(5)	106.3 (4)
C(2)—S(1)—C(5)	92.5 (4)	C(2)—S(2)—C(26)	102.4 (4)
S(1)—C(2)—S(2)	105.5 (4)	S(1)—C(2)—C(3)	104.3 (5)
S(2)—C(2)—C(3)	108.2 (5)	S(1)—C(2)—C(21)	108.1 (5)
S(2)—C(2)—C(21)	115.2 (6)	C(3)—C(2)—C(21)	114.7 (7)
C(2)—C(3)—C(4)	107.9 (7)	C(3)—C(4)—C(5)	105.9 (6)
S(1)—C(5)—C(4)	110.0 (5)	C(2)—C(21)—C(22)	113.4 (6)
C(2)—C(22)—C(23)	113.7 (6)	C(22)—C(23)—C(24)	116.7 (7)
O(2)—C(23)—C(22)	121.9 (6)	O(2)—C(23)—C(24)	121.4 (7)
S(1)—C(2)—C(3)—C(4)	42.1 (7)	C(5)—S(1)—C(2)—C(3)	-21.0 (5)
C(2)—C(3)—C(4)—C(5)	-46.6 (9)	O(1)—S(1)—C(2)—S(2)	-159.0 (5)
C(3)—C(4)—C(5)—S(1)	29.3 (9)	O(1)—S(1)—C(2)—C(21)	-35.4 (8)
C(4)—C(5)—S(1)—C(2)	-4.7 (7)	O(1)—S(1)—C(2)—C(3)	87.1 (6)

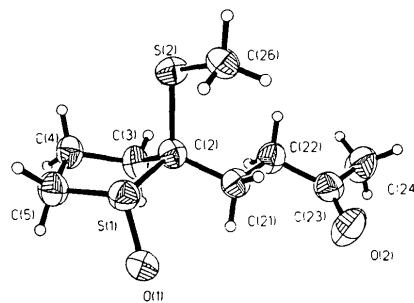


Fig. 1. Drawing of the molecular structure with displacement ellipsoids (50% probability) and atomic numbering scheme.

the molecule is shown in Fig. 1. Programs used were *SHELXTL-Plus* (Sheldrick, 1990), *DIFABS* (Walker & Stuart, 1983), and *PLATON88* (Spek, 1982) on MicroVAX II and VAX 3200 computers.

Related literature. The molecular structures of thiolane *S*-oxide and 2-methylthiolane *S*-oxide (Forgacs, Schultz, Hargittai, Jalsovszky & Kucsman, 1989) and thiane *S*-oxide (Forgacs, Hargittai, Jalsovszky & Kucsman, 1991) have been reported.

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Structure at 198 K of a Chiral Tricyclic Aminochloride, C₁₀H₁₇N·0.5HCl

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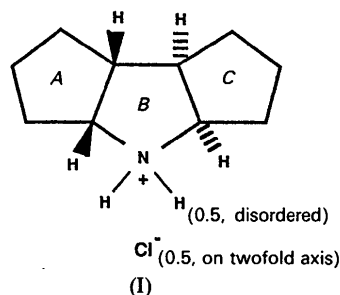
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Abstract. [3*aR*-(3*aα*,4*aβ*,7*aβ*,7*bα*)]-Decahydro-1*H*-dicyclopenta[*b,d*]pyrrole 0.5-hydrochloride, C₁₀H₁₇N·0.5HCl, *M_r* = 169.5, trigonal, *R*32, *a* = 17.320 (2), *c* = 16.963 (2) Å, *V* = 4406.8 (8) Å³, *Z* = 18, *D_x* = 1.149 g cm⁻³, λ(Mo *Kα*) = 0.71069 Å, μ = 1.96 cm⁻¹, *F*(000) = 1674, *T* = 198 K, *R* = 0.0462 for 1589 reflections [*I* ≥ 3σ(*I*)]. Crystal chirality was assigned to correspond to the known chirality of the parent amine and confirmed by refining the η value [chirality/polarity, 1.2 (2)]. The three five-membered rings (*A*, *B* and *C*) exhibit envelope conformations which appear to be preferred over possible twist conformations. Several closely related compounds exhibit similar envelope conformations. Atoms C(3), C(10) and C(7) (IUPAC numbering: C2, C4*a* and C7, respectively) occupy the flap position in rings *A*, *B* and *C*, respectively. All bond lengths and angles appear to be normal except for a slight elongation of bonds around the trisubstituted ring-junction atoms C(1), C(5), C(6) and C(10). The lengthening of these bonds, relative to those around the disubstituted ring atoms, has been seen in related compounds. Two kinds of intermolecular interactions are observed in the crystal structure. H(1*b*) (with 0.5 occupancy at the N atom) links cations related by a twofold axis in the *ab* planes to form dimers [N(1)—H(1*b*)···N(1) (0.333 + *y*, -0.333 + *x*, 0.667 - *z*): N—H = 0.93 (7), H···N = 1.80 (8), N···N = 2.727 (3) Å, N—H···N = 175 (3)°]. The Cl⁻ anion interacts with two H(1*a*) atoms related by a twofold axis (passing through the Cl⁻ anion) in the *ab* planes [N(1)—H(1*a*)···Cl (x - *y*, -*y*, 1 - *z*): N—H =

0.89 (3), H···Cl = 2.28 (4), N···Cl = 3.154 (3) Å, N—H···Cl = 167 (2)°]. The combination of these two kinds of intermolecular interactions produces infinite spirals of ions along the *c* axis.

Experimental. The mandelic acid salt of this tricyclic amine was synthesized according to a previous procedure (Whitesell, Minto & Chen, 1988). The amine was freed from its mandelic acid salt in 95% yield using 4*N* sodium hydroxide and dichloromethane. Colorless crystals of the half-hydrochloride salt (I) were obtained after drying and concentrating the organic layer. The data crystal had dimensions 0.31



× 0.35 × 0.38 mm. A Nicolet *R3m/V* diffractometer equipped with a graphite monochromator and a Nicolet LT-2 low-temperature delivery system (198 K) was used to collect the data. The lattice parameters were obtained from the least-squares refinement of 50 reflections with 22.25 < 2θ < 24.81°. The data were collected using the ω-scan technique with a 2θ range 3.0–55.0° and a 1.0° ω scan at 3–6° min⁻¹ (*h* = -22→22, *k* = -22→22, *l* = 0→22).

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