Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1316). Services for accessing these data are described at the back of the journal.

References

Bellucci, G., Chiappe, C., Bianchini, R., Lenoir, D. & Herges, R. (1995). *J. Am. Chem. Soc.* **117**, 12001–12002.

Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389–393.

Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.

Ermer, O. (1977). *Z. Naturforsch. Teil B*, **32**, 837–839.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Gano, A. J. & Gano, J. E. (1994). *BASE*, **122**, 30–36.

Gano, J. E., Jacob, E. J. & Roesner, R. (1991). *J. Comput. Chem.* **12**, 127–134.

Gano, J. E., Jacob, E. J., Sekher, P., Subramaniam, G., Eriksson, L. A. & Lenoir, D. (1996). *J. Org. Chem.* **61**, 6739–6743.

Gano, J. E., Kirschbaum, K. & Sekher, P. (1996). *Acta Cryst.* C**52**, 2334–2337.

Gano, J. E., Park, B.-S., Pinkerton, A. A. & Lenoir, D. (1990). *J. Org. Chem.* **55**, 2688–2693.

Gano, J. E., Park, B.-S., Pinkerton, A. A. & Lenoir, D. (1991). *Acta Cryst.* C**47**, 162–164.

Gano, J. E., Park, B.-S., Subramaniam, G., Lenoir, D. & Gleiter, R. (1991). *J. Org. Chem.* **56**, 4806–4808.

Gano, J. E., Sekher, P., Weber, L. & Lenoir, D. (1996). *J. Mass Spectrosc.* **31**, 363–366.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kahr, B., Mitchell, C. A., Chance, J. M., Vernonclark, R., Gantzel, P., Baldridge, K. K. & Siegel, J. S. (1995). *J. Am. Chem. Soc.* **117**, 4479–4482.

Laali, K. K., Gano, J. E., Lenoir, D. & Gundlach, C. W. I. (1994). *J. Chem. Soc. Perkin Trans.* 2, pp. 2169–2173.

Lenoir, D. & Burghard, H. (1980). *J. Chem. Res. (S)*, pp. 396–397; *J. Chem. Res. (M)*, pp. 4715–4725.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Ogawa, K., Harada, J. & Tomoda, S. (1995). *Acta Cryst.* B**51**, 240–248.

Ogawa, K., Sano, T., Yoshimura, S., Takeuchi, Y. & Toriumi, K. (1992). *J. Am. Chem. Soc.* **114**, 1041–1051.

Pilati, T. & Simonetta, M. (1982). *Acta Cryst.* B**38**, 1649–1653.

Saito, K. & Ikemoto, I. (1996). *Bull. Chem. Soc. Jpn*, **69**, 909–913.

Waldeck, D. H. (1991). *Chem. Rev.* **91**, 415–436.

Acta Cryst. (1997). C**53**, 1725–1726

(+)-Camphorsulfonylimine

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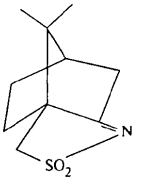
Abstract

The norbornane ring system in the title molecule, 8,8-dimethyl-3,3a,4,5,6,7-hexahydro-3a,6-methanobenz[*c*]isothiazole *S*,*S*-dioxide, $C_{10}H_{15}NO_2S$, is regular with normal bond lengths and angles. The bridgehead bond angle is 92.5 (2)°. The five-membered ring of the sulfonylimine moiety adopts a flattened envelope conformation. The crystal structure is stabilized by weak C—H···O hydrogen bonds.

Comment

Camphor derivatives are important chiral auxiliaries or catalysts for stereoselective synthesis (Oppolzer, 1987). The crystal structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation.



(I)

The bond lengths and angles of the camphor ring system are comparable to those in related molecules (Bear & Trotter, 1975; Garcia *et al.*, 1988). The six-membered ring of the norbornane ring system has a fairly symmetrical boat conformation, with atoms C3 and C7 displaced by −0.852 (3) and −0.845 (2) Å, respectively, from the best plane through atoms C1, C2, C5 and C6. The bridgehead bond angle (C3—C4—C7) of 92.5 (2)° is typical of norbornane derivatives. The two five-membered rings formed by the bridging atom C4 (C1—C2—C3—C4—C7 and C3—C5—C6—C7—C4) adopt envelope conformations with ΔC_s(C4) asymmetry parameters (Nardelli, 1983*a*) equal to 0.007 (2) and 0.015 (2), respectively. The angles between the

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plane of the three-atom bridge C3—C4—C7 and each of the four-atom planes of the boat-shaped six-membered rings (C1, C2, C3, C7 and C3, C5, C6, C7) are 56.0 (2) and 125.8 (2)°, respectively. The five-membered ring of the sulfonylimine moiety adopts a flattened envelope conformation [ΔC_s(C8) = 0.007 (2)], with C8 deviating from the mean plane by −0.290 (5) Å.

In the crystal, the screw-related molecules are linked by C—H···O bifurcated hydrogen bonds involving O1. This hydrogen bond and other short C—H···O contacts are listed in Table 2.

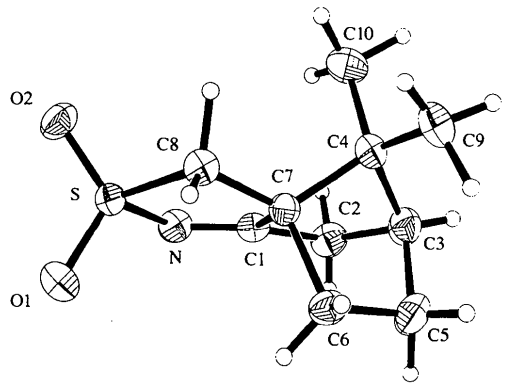

Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Formaldehyde bibenzylmercaptal (Ray & Chakraborty, 1997) (63 mg, 0.24 mmol) in 1 ml of chloroform was added to a stirred solution of (1*R*)-(−)-(10-camphorsulfonyl)oxaziridine (50 mg, 0.22 mmol; Aldrich) in 2 ml of chloroform under argon. After stirring for 1 h, (*R*)-camphorsulfonylimine and (*R*)-benzyl benzylthiomethyl sulfoxide were isolated by preparative TLC (silica gel, benzene/ethyl acetate, 9:1 then 4:1). Single crystals of (I) were obtained from dichloromethane.

Crystal data

$C_{10}H_{15}NO_2S$
M_r = 213.29
Monoclinic
$P2_1$
a = 7.7208 (9) Å
b = 7.7501 (9) Å
c = 9.0531 (10) Å
β = 94.818 (10)°
V = 539.79 (11) Å^3
Z = 2
D_x = 1.312 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 36 reflections
θ = 4.96–11.05°
μ = 0.275 mm^{-1}
T = 293 (2) K
Rectangular slab
0.64 × 0.26 × 0.06 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3014 measured reflections
2468 independent reflections
1852 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.49°
$h = -1 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.095
S = 0.905
2468 reflections
187 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.158 e Å^{-3}
$\Delta\rho_{min}$ = −0.252 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = −0.12 (10)

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

S—N	1.676 (2)	S—C8	1.802 (2)
O1—S—O2	117.22 (11)	N—C1—C7	121.5 (2)
N—S—C8	98.29 (10)	C3—C4—C7	92.5 (2)
N—C1—C2	130.8 (2)	C8—C7—C4	122.3 (2)

Table 2. *Hydrogen-bond geometry and short contacts (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···O1^{i}	1.12 (6)	2.33 (6)	3.411 (6)	160 (4)
C2—H2*A*···O2^{ii}	0.83 (4)	2.61 (4)	3.435 (6)	175 (3)
C8—H8*A*···O1^{iii}	1.06 (5)	2.53 (5)	3.534 (5)	158 (4)
C8—H8*B*···O2^{iv}	0.82 (3)	2.65 (3)	3.421 (5)	157 (3)

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

Data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994). Structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Structure refinement: *SHELXL*93 (Sheldrick, 1993). Geometric calculations: *PARST* (Nardelli, 1983*b*).

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References

Bear, A. C. & Trotter, J. (1975). *Acta Cryst.* B**31**, 903–904.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Garcia, S. M., Salmon, M., Toscano, R. A., Shomar, L. R. & Angles, E. (1988). *Acta Cryst.* C**44**, 1641–1644.
Nardelli, M. (1983*a*). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1983*b*). *Comput. Chem.* **7**, 95–98.
Oppolzer, W. (1987). *Tetrahedron*, **43**, 1969–2004.
Ray, J. K. & Chakraborty, A. (1997). Unpublished results.
Sheldrick, G. M. (1990). *SHELXTL/PC Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994). *XSCANS Users Manual.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.