Références

Averbuch-Pouchot, M. T. & Durif, A. (1991*a*). *Acta Cryst.* C**47**, 1579–1583.
Averbuch-Pouchot, M. T. & Durif, A. (1991*b*). *Eur. J. Solid State Inorg. Chem.* **28**, 9–22.
Averbuch-Pouchot, M. T. & Durif, A. (1993). *Eur. J. Solid State Inorg. Chem.* **30**, 943–951.
Blessing, R. H. (1986). *Acta Cryst.* B**42**, 613–621.
Boullé, A. (1938). *C. R. Acad. Sci.* **206**, 517–519.
Brown, I. D. (1976). *Acta Cryst.* A**32**, 24–31.
Durif, A. & Averbuch-Pouchot, M. T. (1989). *Acta Cryst.* C**45**, 1884–1887.
Durif, A. & Averbuch-Pouchot, M. T. (1990). *Acta Cryst.* C**46**, 2026–2028.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, Les Pays-Bas.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, Les Pays-Bas.
Gharbi, A., Jouini, A. & Durif, A. (1995). *J. Solid State Chem.* **114**, 42–51.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, EU.
Schülke, U. & Kayser, R. (1985). *Z. Anorg. Chem.* **4**, 549.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Université de Göttingen, Allemagne.
Soumhi, E. H. & Jouini, T. (1995*a*) *Acta Cryst.* C**51**, 1883–1885.
Soumhi, E. H. & Jouini, T. (1995*b*) *Acta Cryst.* C**51**, 1457–1459.
Soumhi, E. H. & Jouini, T. (1995*c*) *Acta Cryst.* C**51**, 432–433.

Acta Cryst. (1996). C**52**, 2805–2806

(1*S*,5*R*,6*R*,8*R*,10*R*,12*R*)-5,8,15,15-Tetramethyl-6-phenyl-7,9-dioxa-3-thia-4-azatetracyclo[10.2.1.0^{1,10}.0^{4,8}]pentadecane 3,3-Dioxide

Michael Bolte and Cordula Strahringer

Institut für Organische Chemie der Universität Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/Main, Germany. E-mail: bolte@chemie.uni-frankfurt.de

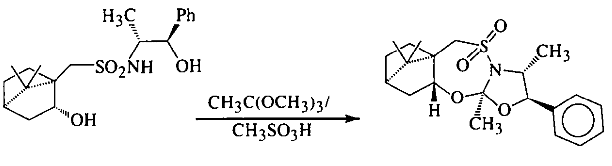
(*Received 21 May 1996; accepted 2 July 1996*)

Abstract

In the title compound, $C_{21}H_{29}NO_4S$, the seven-membered ring has a chair conformation and the oxazolidine ring a twist conformation.

Comment

2-Alkyl-2-alkoxy-3-arenesulfonyl-1,3-oxazolidines exhibit high diastereofacial selectivity in their Lewis-acid-catalysed C—C-coupling reactions (Harder, Löhl, Bolte, Wagner & Hoppe, 1994). In an attempt at further improvement, the title compound, (I), a tetracyclic oxazolidine, was prepared.



(I)

The structure determination was carried out in order to determine the absolute configuration of the stereogenic centres. The seven-membered ring has a chair conformation [q_2 = 0.6 Å, φ_2 = 157.2°, q_3 = 0.6 Å, φ_3 = 349.3° (Cremer & Pople, 1975)] with C1 forming the apex. The oxazolidine ring exhibits a twist conformation [q_2 = 0.3 Å, φ_2 = 266.4°] with C6 and O7 deviating by 0.291 (5) and −0.199 (4) Å, respectively, from the plane of the remaining three atoms. The phenyl ring occupies an equatorial position forming an angle of 77.58 (7)° with the oxazolidine ring. The observation that the geometry about the N atom is nearly planar (the sum of the bond angles is 354.1°) is in agreement with the statistical investigation of Herbst-Irmer (1990) on arylsulfonyl-oxazolidines.

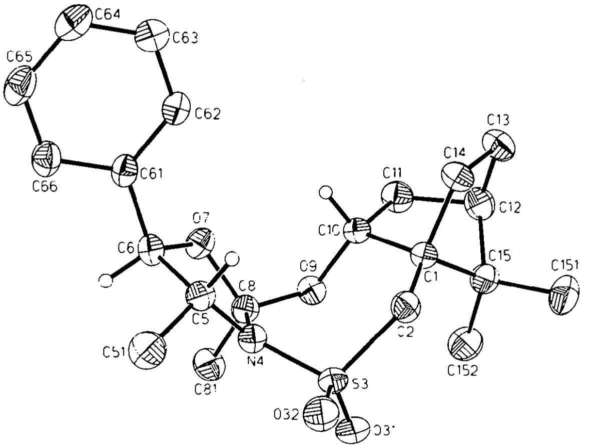


Fig. 1. Perspective view of the title compound with the atom numbering and displacement ellipsoids at the 30% probability level; only tertiary H atoms are shown.

Experimental

Stirring the amide *N*-(2-hydroxy-1-methyl-2-phenylethyl)(2-hydroxy-7,7-dimethylbicyclo[2.2.1]hept-1-yl)methanesulfonamide (see scheme above), derived from (1*S*)-10-camphorsulfonic acid and (1*R*,2*R*)-norpseudoephedrine, with excess trimethyl orthoacetate in the presence of methanesulfonic acid, afforded the diastereomericially homogenous title compound, (I), in 75% yield (Harder, 1995). Single crystals were obtained from an ether solution by diffusion of pentane.

Crystal data

$C_{21}H_{29}NO_4S$
M_r = 391.51
Cu $K\alpha$ radiation
λ = 1.5418 Å

Orthorhombic
$P2_12_12_1$
$a = 9.425\ (1)$ Å
$b = 10.904\ (3)$ Å
$c = 20.034\ (7)$ Å
$V = 2058.9\ (9)$ Å^3
$Z = 4$
$D_x = 1.263$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 30$–$35°$
$\mu = 1.606$ mm^{-1}
$T = 293$ K
Block
0.60 × 0.40 × 0.30 mm
Colourless, transparent

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
ω scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.57$, $T_{\max} = 0.62$
3726 measured reflections
3387 independent reflections

3337 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0128$
$\theta_{\max} = 64.83°$
$h = -10 \rightarrow 0$
$k = 0 \rightarrow 12$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 92 min
intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0307$
$wR(F^2) = 0.0918$
$S = 0.991$
3387 reflections
245 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2 + 0.2445P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.190$ e Å^{-3}
$\Delta\rho_{\min} = -0.295$ e Å^{-3}

Extinction correction: *SHELXL*96 (Sheldrick, 1996)
Extinction coefficient: 0.0103 (5)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.01 (2)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.2517 (2)	0.36057 (16)	0.20116 (8)	0.0395 (4)
C2	0.0908 (2)	0.37206 (19)	0.19763 (9)	0.0454 (4)
S3	−0.00128 (5)	0.48126 (4)	0.24746 (2)	0.04509 (15)
O31	0.04958 (18)	0.60220 (14)	0.23439 (7)	0.0615 (4)
O32	−0.14862 (15)	0.45515 (17)	0.23803 (8)	0.0638 (4)
N4	0.03592 (15)	0.45151 (14)	0.32499 (7)	0.0420 (4)
C5	−0.03043 (18)	0.34930 (17)	0.36131 (9)	0.0419 (4)
C51	−0.1818 (2)	0.3760 (3)	0.38317 (13)	0.0659 (6)
C6	0.0697 (2)	0.33841 (18)	0.42196 (9)	0.0427 (4)
O7	0.20265 (14)	0.38521 (13)	0.39891 (6)	0.0455 (3)
C8	0.17826 (18)	0.48192 (18)	0.35280 (8)	0.0416 (4)
C81	0.1775 (3)	0.6066 (2)	0.38631 (11)	0.0561 (5)
O9	0.28646 (12)	0.48491 (11)	0.30639 (6)	0.0424 (3)
C10	0.32167 (19)	0.37411 (16)	0.27056 (8)	0.0391 (4)
C11	0.4815 (2)	0.3807 (2)	0.25304 (10)	0.0549 (5)
C12	0.4849 (2)	0.3693 (2)	0.17680 (10)	0.0587 (5)
C13	0.4512 (2)	0.2365 (2)	0.16016 (12)	0.0648 (6)
C14	0.2933 (2)	0.22769 (19)	0.17979 (11)	0.0532 (5)
C15	0.3493 (2)	0.4387 (2)	0.15473 (10)	0.0517 (5)
C151	0.3139 (3)	0.4233 (3)	0.08014 (11)	0.0785 (8)
C152	0.3554 (3)	0.5763 (2)	0.16864 (13)	0.0716 (7)
C61	0.0848 (2)	0.21019 (18)	0.44769 (9)	0.0440 (4)
C62	0.1648 (3)	0.1235 (2)	0.41438 (11)	0.0590 (5)
C63	0.1710 (3)	0.0042 (2)	0.43671 (12)	0.0668 (6)
C64	0.0972 (3)	−0.0315 (2)	0.49248 (12)	0.0642 (6)
C65	0.0169 (2)	0.0545 (2)	0.52659 (11)	0.0648 (6)
C66	0.0103 (2)	0.1732 (2)	0.50436 (10)	0.0545 (5)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.523 (3)	N4—C8	1.490 (2)
C1—C10	1.546 (2)	C5—C6	1.543 (3)
C2—S3	1.780 (2)	C6—O7	1.430 (2)
S3—O31	1.4273 (17)	O7—C8	1.421 (2)
S3—O32	1.4302 (15)	C8—O9	1.380 (2)
S3—N4	1.6250 (15)	O9—C10	1.444 (2)
N4—C5	1.471 (2)		
C2—C1—C10	117.30 (15)	C8—N4—S3	120.47 (12)
C1—C2—S3	120.95 (13)	N4—C5—C6	100.83 (14)
O31—S3—O32	119.06 (10)	O7—C6—C5	104.73 (14)
O31—S3—N4	106.71 (9)	C8—O7—C6	109.45 (14)
O32—S3—N4	107.22 (8)	O9—C8—O7	109.63 (14)
O31—S3—C2	110.58 (10)	O9—C8—N4	114.75 (14)
O32—S3—C2	105.43 (10)	O7—C8—N4	102.92 (14)
N4—S3—C2	107.30 (8)	C8—O9—C10	119.00 (13)
C5—N4—C8	111.51 (14)	O9—C10—C1	115.40 (14)
C5—N4—S3	122.17 (12)		
C10—C1—C2—S3	38.5 (2)	C6—O7—C8—N4	−26.51 (17)
C1—C2—S3—N4	58.44 (17)	S3—N4—C8—O9	− 26.3 (2)
C2—S3—N4—C8	73.69 (16)	C5—N4—C8—O7	8.26 (18)
C8—N4—C5—C6	11.09 (18)	N4—C8—O9—C10	−62.4 (2)
N4—C5—C6—O7	−26.41 (18)	C8—O9—C10—C1	95.83 (17)
C5—C6—O7—C8	34.36 (18)	C2—C1—C10—O9	−54.1 (2)

The structure was solved by direct methods and refined by full-matrix least-squares methods. All H atoms were located by a difference Fourier synthesis and refined with fixed individual displacement parameters [$U({\rm H}) = 1.5U_{\rm eq}({\rm C}_{\rm methyl})$ or $U({\rm H}) = 1.2U_{\rm eq}({\rm C})$] using a riding model with C—H(aromatic) = 0.93, C—H(methyl) = 0.96, C—H(secondary) = 0.97 or C—H(tertiary) = 0.98 Å.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991).

We thank Professor Dr D. Hoppe (University of Münster) for providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1144). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **89**, 6193–6200.

Enraf–Nonius (1985). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Harder, T. (1995). PhD thesis, University of Münster, Germany.

Harder, T., Löhl, T., Bolte, M., Wagner, K. & Hoppe, D. (1994). *Tetrahedron Lett.* **35**, 7365–7368.

Herbst-Irmer, R. (1990). PhD thesis, University of Göttingen, Germany.

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

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1996). *SHELXL*96. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.