C13	0.2377 (3)	0.0333 (2)	0.42326 (8)	0.0324 (5)
C14	0.1868 (3)	0.0686 (2)	0.37086 (8)	0.0263 (5)
C15	−0.3485 (3)	−0.0004 (3)	0.10785 (8)	0.0367 (5)
C16	−0.0282 (3)	0.1318 (2)	0.04014 (7)	0.0336 (5)

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

O1—C14	1.349 (2)	N2—C7	1.298 (2)
N1—C1	1.385 (3)	N2—C6	1.421 (2)
C7—N2—C6	124.6 (2)	N2—C7—C9	116.8 (2)
N1—C1—C2	121.7 (2)	N2—C7—C8	124.2 (2)
N1—C1—C6	120.2 (2)	O1—C14—C13	118.4 (2)
C5—C6—N2	123.0 (2)	O1—C14—C9	121.5 (2)
C1—C6—N2	117.2 (2)		
C6—N2—C7—C9	174.18 (15)	C8—C7—C9—C10	5.1 (2)
C6—N2—C7—C8	−7.7 (3)		

Data were collected from 0.2° ω-rotation exposures of 10 s each, with a crystal–detector distance of 49.37 (8) mm. Crystal decay was found to be negligible by comparison of intensities of repeated reflections. H atoms were added at calculated positions and refined using a riding model, including free rotation of methyl and hydroxy groups. Anisotropic displacement parameters were used for all non-H atoms and H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their attached atoms.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1992). Cell refinement: *MADNES*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1102). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Sulfonyl-Substituted Aziridine

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Abstract

In the title compound, 1-*p*-toluenesulfonylaziridine-(2*S*)-carboxylic acid *tert*-butyl ester, $C_{14}H_{19}NO_4S$, the *p*-toluenesulfonyl substituent on the N atom and the ester substituent on one C atom of the aziridine ring are mutually *trans*, minimizing steric interactions. The two N—C ring bond lengths [1.479 (2) and 1.484 (2) Å] are insignificantly different, consistent with the lack of regioselectivity in reactions with carbon nucleophiles.

Comment

The title compound (I) was originally prepared from the corresponding carboxylic acid, the esterification being achieved by treatment with *tert*-butyl 2,2,2-trichloroacetimidate in the presence of catalytic amounts of boron trifluoride–diethyl ether (Armstrong, Brackenridge, Jackson & Kirk, 1988). It was planned to explore its reactions with organometallic nucleophiles as a potential route to optically active α-amino acids but the results of such studies have been published recently by other researchers, together with details of the preparation and spectroscopic characterization of the compound (Baldwin, Spivey, Schofield & Sweeney, 1993). The compound has also been prepared by dicyclohexylcarbodiimide-mediated esterification of the carboxylic acid using *tert*-butanol (Church & Young, 1994). We report here the crystallographic confirmation of the structure (Fig. 1).

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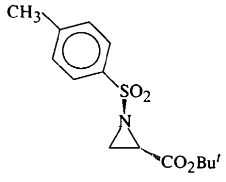

(I)

The configuration at the ring N atom, not surprisingly, places the bulky *p*-toluenesulfonyl substituent *trans* to the ester substituent on one of the ring C atoms. The conformation at the S atom is such as to place one O atom (O1) directly under the three-membered ring, with the larger *p*-tolyl group extended away from the ring. The almost precisely equivalent N—C bond lengths are completely consistent with the observed lack of significant regioselectivity in the reaction of this compound with carbon nucleophiles, specifically copper-catalysed addition of Grignard reagents (Baldwin *et al.*, 1993). These bond lengths, and that for the ring C—C bond, are well within the range found [N—C 1.439–1.551 Å, C—C 1.457–1.508 Å] for 16 aziridines with carboxylate substituents on carbon in the April 1996 release of the Cambridge Structural Database (Allen & Kennard, 1993).

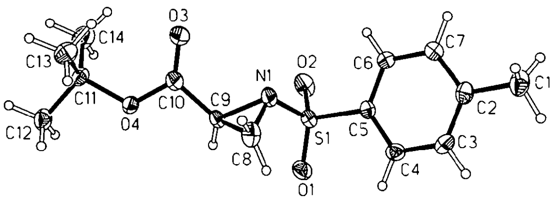

Fig. 1. The molecular structure with atom labels and 50% probability ellipsoids for non-H atoms.

Experimental

The synthesis of the title compound has already been described (Baldwin *et al.*, 1993) using the method of Armstrong *et al.*, (1988). Crystals were obtained by diffusion of petroleum ether into a solution in ethyl acetate.

Crystal data

$C_{14}H_{19}NO_4S$
$M_r = 297.36$
Orthorhombic
$P2_12_12_1$
$a = 5.9653\,(7)$ Å
$b = 13.450\,(2)$ Å
$c = 18.630\,(2)$ Å
$V = 1494.8\,(3)$ Å^3
$Z = 4$
$D_x = 1.321$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 7831 reflections
$\theta = 2.66–28.34°$
$\mu = 0.229$ mm^{-1}
$T = 160\,(2)$ K
Prism
$0.53 \times 0.40 \times 0.40$ mm
Colourless

Data collection

Siemens SMART CCD diffractometer
ω rotation with narrow frames
Absorption correction: none
9374 measured reflections
3410 independent reflections

3306 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0352$
$\theta_{\rm max} = 28.36°$
$h = -7 \rightarrow 7$
$k = -17 \rightarrow 14$
$l = -14 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0260$
$wR(F^2) = 0.0685$
$S = 1.062$
3410 reflections
186 parameters
H atoms riding (see text)
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2 + 0.2985P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.316$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.247$ e Å^{-3}

Extinction correction: *SHELXTL* (Sheldrick, 1994)
Extinction coefficient: 0.0058 (10)
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 = −0.04 (5)

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

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

	x	y	z	$U_{\rm eq}$
C1	0.7034 (3)	−0.29388 (10)	0.40501 (8)	0.0331 (3)
C2	0.5923 (2)	−0.19719 (10)	0.42485 (7)	0.0242 (3)
C3	0.7023 (2)	−0.12858 (10)	0.46797 (7)	0.0257 (3)
C4	0.6103 (2)	−0.03591 (10)	0.48294 (7)	0.0238 (3)
C5	0.4020 (2)	−0.01328 (9)	0.45441 (7)	0.0209 (3)
C6	0.2855 (2)	−0.08121 (9)	0.41241 (7)	0.0243 (3)
C7	0.3803 (2)	−0.17253 (10)	0.39784 (8)	0.0257 (3)
S1	0.28463 (5)	0.10491 (2)	0.46928 (2)	0.02279 (9)
O1	0.4126 (2)	0.15580 (7)	0.52307 (5)	0.0303 (2)
O2	0.0471 (2)	0.09558 (8)	0.47885 (6)	0.0356 (3)
N1	0.3174 (2)	0.15524 (8)	0.38806 (6)	0.0255 (2)
C8	0.5356 (3)	0.20383 (10)	0.37269 (8)	0.0309 (3)
C9	0.3332 (2)	0.26498 (9)	0.38745 (7)	0.0235 (3)
C10	0.2069 (3)	0.31423 (10)	0.32753 (7)	0.0282 (3)
O3	0.1136 (2)	0.27107 (8)	0.27943 (7)	0.0479 (4)
O4	0.2217 (2)	0.41206 (6)	0.33591 (5)	0.0243 (2)
C11	0.1123 (2)	0.48164 (10)	0.28494 (7)	0.0234 (3)
C12	0.1833 (3)	0.58247 (10)	0.31373 (8)	0.0315 (3)
C13	0.2046 (3)	0.46691 (11)	0.20986 (7)	0.0314 (3)
C14	−0.1411 (2)	0.46876 (13)	0.28868 (10)	0.0387 (4)

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

S1—O1	1.4337 (10)	C8—C9	1.487 (2)
S1—O2	1.4339 (10)	C9—C10	1.501 (2)
S1—N1	1.6691 (12)	C10—O3	1.204 (2)
N1—C9	1.479 (2)	C10—O4	1.328 (2)
N1—C8	1.484 (2)	O4—C11	1.4843 (15)
C9—N1—C8	60.22 (9)	N1—C9—C8	60.05 (9)
C9—N1—S1	114.78 (9)	N1—C9—C10	114.47 (11)
C8—N1—S1	117.15 (10)	C8—C9—C10	120.95 (12)
N1—C8—C9	59.73 (8)		

The data collection nominally covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 4.95 cm. Coverage of the unique set is complete to at least 26.4° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections.

H atoms were placed geometrically and refined with a riding model including free rotation about C—C bonds, and with $U_{\rm iso}$ constrained to be 1.2 (1.5 for methyl groups) times $U_{\rm eq}$ of the carrier atom.

Data collection: *SMART* (Siemens, 1995). Cell refinement: local programs. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and local programs.

We thank EPSRC for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1222). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Sulfoximidooxirane

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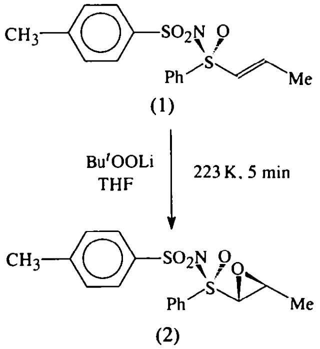
Abstract

The crystal structure of the title compound, *S*-(*trans*-3-methyl-2-oxiranyl)-*S*-phenyl-*N*-(*p*-tolylsulfonyl)sulfoximide, $C_{16}H_{17}NO_4S_2$, has two independent molecules in the asymmetric unit, which differ significantly only in the conformation of the (*p*-tolylsulfonyl)sulfoximide group. The powerful electron-withdrawing properties of the sulfoximide substituent produce a marked difference in the C—O bond lengths of the oxirane ring, the nearer bond being substantially shortened relative to the distal bond. The observed structure further demonstrates the high degree of diastereoselectivity in the nucleophilic epoxidation of vinylsulfoximides with lithium *tert*-butylperoxide.

Comment

The title compound is one of a series prepared in a study of nucleophilic epoxidation of vinylsulfoximides by lithium *tert*-butylperoxide (Bailey, Clegg, Jackson & Meth-Cohn, 1993). As part of that study we reported the crystallographic characterization of the corresponding isopropyl (in place of methyl) derivative, which served to demonstrate a high degree of diastereoselectivity in the epoxidation. The present structure provides additional support for the conclusion that the observed sense of diastereoselectivity in these reactions is a general phenomenon. Preferential addition to the β-face of the double bond of vinylsulfoximides has been rationalized on the basis of steric hindrance of the α-face by the sulfoximide group, as revealed by crystallographic analysis (Dong, Pyne, Skelton & White, 1993).

The two crystallographically independent molecules of the title compound, (2), have essentially the same geometry for the oxirane ring and its immediate substituents. They differ only slightly in the orientations of the phenyl group attached to sulfur, and the main difference is in the conformation of the sulfonyl group at the N atom, with approximately 10° difference in the S—N—S—C torsion angle. In both molecules, the sulfoximide S=O bond approximately eclipses the oxirane ring C—C bond whereas in the isopropyl analogue (Bailey *et al.*, 1993), it eclipses the nearer C—O bond.



For both molecules in the asymmetric unit, as for the isopropyl analogue, the two ring C—O bonds are very different in length. In general, there is no clear correlation between the C—O bond lengths and the electronic properties of the substituents for oxirane structures (Clegg & Kelly, 1994; Clegg, Ranelli & Jackson,

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