Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-[(1*S*,2*S*,3*R*)-2-Bromo-3-(*tert*-butyldiphenylsilyloxy)-1-phenyl-4-(phenylsulfonyl)butyl]-4-methylbenzenesulfonamide

K. Ravikumar,[a] G. Y. S. K. Swamy,[a]* S. Raghavan[b] and Ch. Naveenkumar[b]

[a]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [b]Organic Division I, Indian Institute of Chemical Technology, Hyderabad 500 007, India

Correspondence e-mail: swamy@ins.iictnet.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.130
Data-to-parameter ratio = 19.9

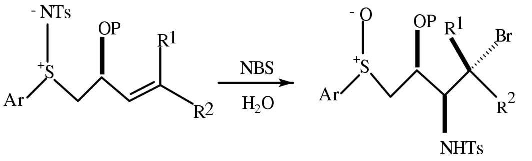
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 November 2004
Accepted 3 November 2004
Online 13 November 2004

In the title compound, $C_{39}H_{42}BrNO_5S_2Si$, the Si and S atoms have tetrahedrally geometry. In the crystal structure, the molecules form centrosymmetric dimers through rather long N—H···O hydrogen-bonding interactions.

Comment

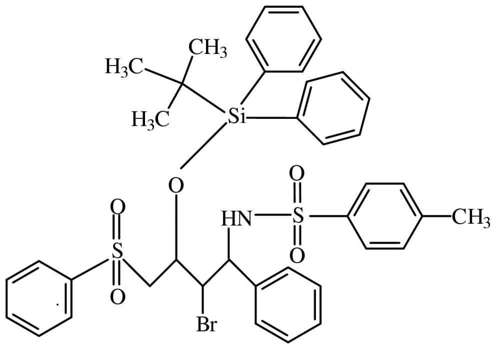
Vicinal (Babine & Bender, 1997) and 1,3-amino alcohols (Benedetti & Norbedo, 2001) are structural motifs present in a variety of natural products and pharmaceutical agents. Interest in these structural units has led to the development of a variety of methods for their synthesis (Petasis & Zavialov, 1998). We recently disclosed (Raghavan *et al.*, 2004) an efficient, novel and general method for the preparation of amino alcohol derivatives, (2), from readily accessible β-hydroxy (silyloxy) γ,δ-unsaturated sulfilimines, (1), *via* intramolecular nucleophilic assistance, by the sulfilimine group. The crystal structure determination of the title compound, (I), was carried out in order to elucidate the relative stereochemistry at atoms C1, C2 and C3 (Fig. 1.)



1 2

P = H, SiR_3
R^1 = H, Alkyl
R^2 = H, Alkyl, Aryl

Ts = p- toluenesulfonyl
NBS = N-bromosuccsuccinimide



(I)

The S—O, S—C, S—N and C—Br bond lengths are comparable with the reported mean literature values of 1.436 (1), 1.78 (2), 1.663 (19) and 1.97 (3) Å, respectively (Allen *et al.*, 1987). The bond distances around Si are comparable with those in reported structures (Ravikumar *et al.*, 2004; Lewinski, 1997; Tomlins *et al.*, 1985).

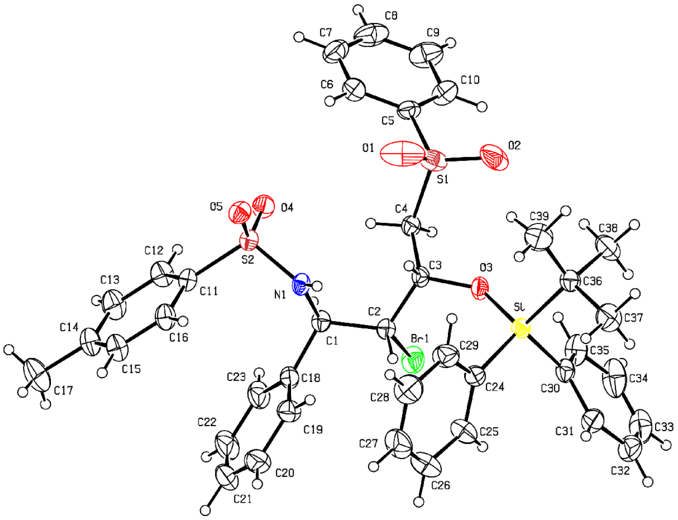


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Atoms S2 and Si have tetrahedral geometry (Table 1) and S1 has distorted tetrahedral geometry. The widening of the O1—S1—O2 angle to 119.1 (2)° and the resulting narrowing of the C5—S1—C4 angle to 102.1 (1)° from the ideal tetrahedral value is attributed to the Thorpe—Ingold effect (Bassindale, 1984). The torsion angle C3—O3—Si—C36 [−113.1 (2)°] differs from an ideal *trans* conformation to a greater extent than observed in the majority of structures containing *tert*-butyldiphenylsilyloxy group (Lewinski, 1997). We have previously reported the structure of a very closely related compound, *viz*. *N*-[(1*S*,2*S*,3*R*)-2-bromo-3-(*tert*-butyldiphenylsiloxy)-1-phenyl-4-(phenylsulfinyl)butyl]benzenesulfonamide (SFIL), where atom S1 carries just one O atom (Ravikumar *et al.*, 2004). In the title compound, the molecules form centrosymmetric dimers *via* rather long N—H···O hydrogen bonds. The structure is further stabilized by weak C—H···O hydrogen bonding (Table 2).

On comparing (I) with SFIL, the relative torsion angles about the bonds S2—N1 [63.3 (2)° in (I) *versus* 78.6 (3)° in SFIL], C4—C3 [162.9 (2) *versus* 168.1 (2)°] and N1—C1 [144.9 (2) *versus* 141.0 (2)°] are very similar, however, the torsion angle about C1—C2 [−39.1 (3) *versus* −168.5 (2)°] is significantly different. A least-squares fit for the atoms C3/C4/S1/C5–C10 (r.m.s. deviation = 0.084 Å) of the two compounds shows significant similarities (Fig. 2), differing only in the orientation of the *p*-toluenesulfonyl group.

Experimental

To a solution of sulfoxide (Ravikumar *et al.*, 2004) (0.1 mmol) in dichloromethane (0.4 ml) was added *m*-chloroperoxybenzoic acid (0.12 mmol) and the mixture was stirred for 30 min. After completion, the reaction mixture was diluted with dichloromethane and washed successively with aqueous sodium bisulfite, aqueous sodium bicarbonate, water, brine and dried over Na_2SO_4. Evaporation of the solvent under reduced pressure afforded the crude product, which was purified by column chromatography using 20% EtOAc/petroleum ether (*v*/*v*) as the eluent to afford the title compound. Suitable colourless crystals were obtained from aqueous methanol.

Crystal data

$C_{39}H_{42}BrNO_5S_2Si$
$M_r = 776.86$
Monoclinic, $P2_1/n$
$a = 13.9704$ (8) Å
$b = 19.5590$ (12) Å
$c = 14.1646$ (9) Å
$\beta = 96.9000$ (10)°
$V = 3842.4$ (4) Å^3
$Z = 4$

$D_x = 1.345$ Mg m^{-3}
Mo *K*α radiation
Cell parameters from 4427 reflections
$\theta = 2.4$–22.8°
$\mu = 1.25$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
0.24 × 0.20 × 0.14 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
23909 measured reflections
8859 independent reflections

5593 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\rm max} = 28.0°$
$h = -17 \rightarrow 18$
$k = -25 \rightarrow 23$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.130$
$S = 1.02$
8859 reflections
446 parameters
H atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.62$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.56$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.960 (3)	N1—S2	1.622 (2)
C4—S1	1.773 (3)	O1—S1	1.428 (3)
C5—S1	1.764 (3)	O2—S1	1.427 (3)
C11—S2	1.759 (3)	O3—Si	1.6446 (19)
C24—Si	1.878 (3)	O4—S2	1.4275 (19)
C30—Si	1.866 (3)	O5—S2	1.4325 (19)
C36—Si	1.887 (3)		
O1—S1—C4	107.12 (15)	N1—S2—C11	108.25 (12)
C5—S1—C4	102.12 (13)	O3—Si—C30	105.24 (12)
O4—S2—N1	107.62 (11)	O3—Si—C24	109.46 (11)
O4—S2—C11	107.95 (12)	C30—Si—C24	111.28 (13)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O4	0.98	2.58	3.027 (5)	108
C4—H4*A*···O4	0.97	2.57	3.493 (3)	160
C10—H10···O2	0.93	2.54	2.904 (2)	104
C12—H12···O4	0.93	2.52	2.896 (2)	105
N1—H1*A*···O5$^{\rm i}$	0.86	2.40	2.966 (3)	124

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The H atoms were positioned geometrically and were treated as riding on their parent C and N atoms, with aromatic C—H distance of 0.93 Å, N—H distances of 0.86 Å, methine C—H distances of 0.98 Å, methylene C—H distances of 0.97 Å and methyl C—H distances of 0.96 Å, and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ for other H atoms. The methyl groups were allowed to rotate but not to tip.

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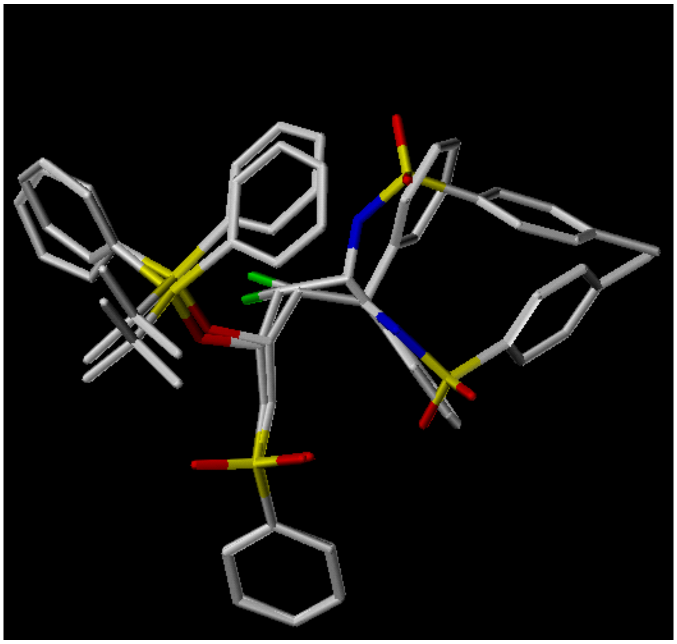

Figure 2
Least-squares fit of (I) and SFIL based on the atoms C3/C4/S1/C5–C10

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

The authors thank Dr J. S. Yadav, Director, IICT, Hyderabad, for his kind encouragement.

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