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N-[(1*S*,2*S*,3*R*)-2-Bromo-3-(*tert*-butyldiphenylsiloxy)-1-phenyl-4-(phenylsulfinyl)butyl]benzenesulfonamide

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In the title compound, $C_{39}H_{42}BrNO_4S_2Si$, the Si atom shows a tetrahedral geometry. The phenyl rings attached to the Si atom form a dihedral angle of 54.9 (1)°. The molecular packing is stabilized by N—H···O hydrogen bonds.

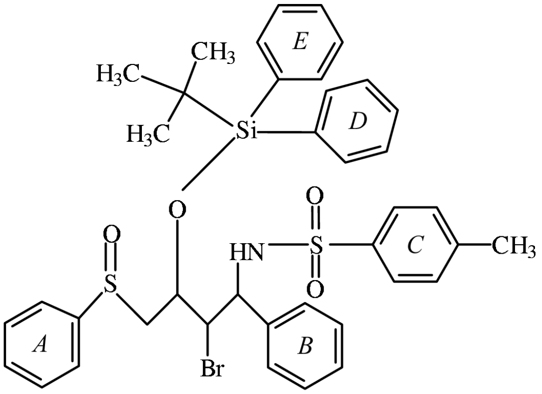
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.054
wR factor = 0.130
Data-to-parameter ratio = 20.8

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

Comment

The structure of the title compound, (I), with the atom-numbering scheme is shown in Fig. 1. The S—O, S—C, S—N and C—Br distances are comparable with the reported mean literature values of 1.436 (1), 1.779 (20), 1.663 (19) and 1.966 (29) Å, respectively (Allen *et al.*, 1987). The bond distances around Si are comparable with those in reported structures (Lewinski, 1997; Tomlins *et al.*, 1985). The S1—O1 bond distance of 1.498 (2) Å confirms the double-bond character (Allen *et al.*, 1987).



(I)

Atoms S2 and Si have tetrahedral geometry (Table 1). The torsion angle C3—O2—Si1—C36 [−120.7 (2)°] differs from an ideal *trans* conformation to a greater extent than observed in the majority of structures containing a *tert*-butyldiphenylsilyloxy group (Lewinski, 1997).

The S atoms are displaced by 0.050 (1) Å for S1 and 0.020 (1) Å for S2 from the mean planes of the corresponding benzene rings *A* and *C* (see scheme). Benzene rings *C* and *D* are nearly perpendicular to one another, with a dihedral angle of 78.6 (1)°. The dihedral angle between phenyl rings *D* and *E* is 54.9 (1)°.

Along the *b* axis, one-dimensional chains are formed through strong N—H···O hydrogen bonds (Fig. 2). Furthermore, the molecules in the chains are arranged parallel to one another through N—H···Br intramolecular hydrogen bonding. In addition, the crystal structure is further stabilized

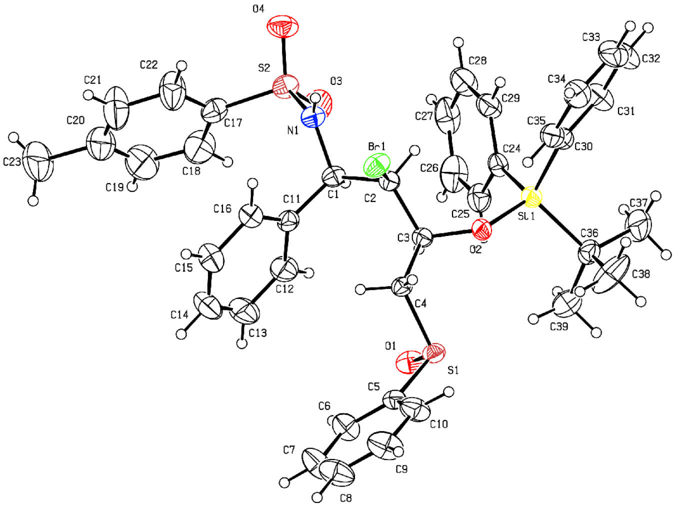


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

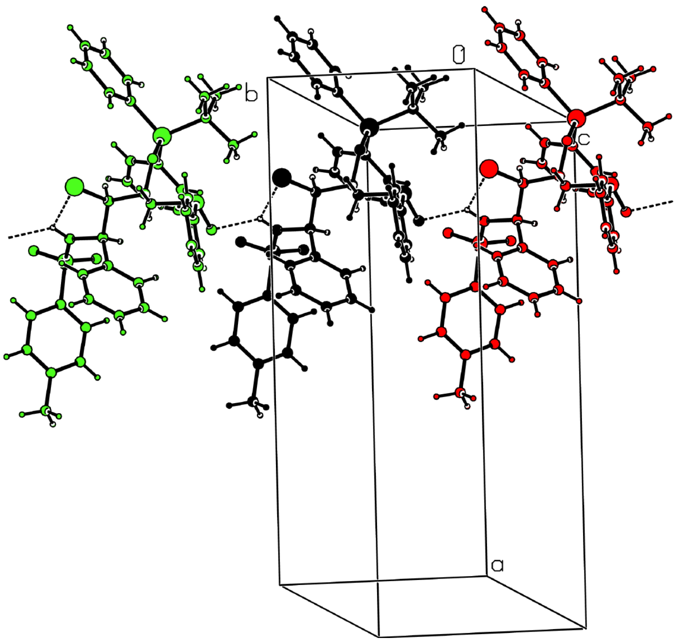


Figure 2
Part of the packing of (I).

by intra- and weak intermolecular C—H···O hydrogen-bonding interactions (Table 2).

Experimental

To a solution of the sulfilimine (332 mg, 0.5 mmol) in dry toluene (5 ml) was added water (0.6 mmol) followed by *N*-bromosuccinimide (0.65 mmol) at room temperature and stirred for 1 h under a nitrogen atmosphere. After completion, the reaction mixture was diluted with ethyl acetate and washed successively with aqueous saturated $NaHCO_3$, water, dried over Na_2SO_4 and evaporated under reduced pressure to afford the crude mixture which was purified by column chromatography using 20% EtOAc/petroleum ether (*v*/*v*) as eluant to efford the title compound bromosulfonamide.

Crystal data

$C_{39}H_{42}BrNO_4S_2Si$
$M_r = 760.86$
Monoclinic, $P2_1/c$
$a = 20.0567$ (11) Å
$b = 8.2412$ (5) Å
$c = 23.8154$ Å
$\beta = 91.263$ (1)°
$V = 3935.5$ (4) Å^3
$Z = 4$
$D_x = 1.284$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3196 reflections
$\theta = 2.6$–20.6°
$\mu = 1.22$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.22 × 0.19 × 0.14 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
24266 measured reflections
9091 independent reflections
5188 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\rm max} = 28.0°$
$h = -18 \rightarrow 26$
$k = -10 \rightarrow 10$
$l = -30 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.130$
$S = 0.95$
9091 reflections
437 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.014$
$\Delta\rho_{\rm max} = 0.56$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.961 (3)	C36—Si1	1.886 (3)
C1—N1	1.470 (4)	N1—S2	1.607 (3)
C4—S1	1.809 (3)	O1—S1	1.498 (2)
C5—S1	1.788 (3)	O2—Si1	1.662 (2)
C17—S2	1.760 (4)	O3—S2	1.425 (3)
C24—Si1	1.868 (3)	O4—S2	1.431 (3)
C30—Si1	1.866 (3)		
O3—S2—N1	107.36 (15)	O2—Si1—C30	106.84 (14)
O3—S2—C17	106.75 (18)	O2—Si1—C24	110.02 (13)
N1—S2—C17	109.02 (15)	C30—Si1—C24	109.21 (15)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br1	0.86	2.87	3.261 (2)	110
C1—H1···O3	0.98	2.38	2.906 (4)	113
C4—H4*B*···Br1	0.97	2.81	3.140 (3)	101
C6—H6···O1	0.93	2.54	2.931 (4)	106
C18—H18···O3	0.93	2.53	2.893 (6)	104
N1—H1*A*···O1$^{\rm i}$	0.86	2.19	2.883 (3)	137
C25—H25···O4$^{\rm ii}$	0.93	2.55	3.271 (5)	135

Symmetry codes: (i) *x*, 1 + *y*, *z*; (ii) *x*, *y* − 1, *z*.

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

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

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used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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