

Keywords: crystal structure; sulfonamide;
diastereomer; amino alcohol; hydrogen
bonding; NMR; column chromatography

CCDC references: 1029177; 1029178

Supporting information: this article has
supporting information at journals.iucr.org/e

Crystal structures of (*R*_S)-*N*-[(1*R*,2*S*)-2-benzyloxy-1-(2,6-dimethylphenyl)propyl]-2-methylpropane-2-sulfonamide and (*R*_S)-*N*-[(1*S*,2*R*)-2-benzyloxy-1-(2,4,6-trimethylphenyl)propyl]-2-methylpropane-2-sulfonamide: two related protected 1,2-amino alcohols

Matthew R. Carbone, Garrick A. Centola, Adam Haas, Kevin P. McClelland, Michael D. Moskowitz, Angelo M. Verderame, Mikael S. Oleszki, Louis J. Papa, Stephanie C. M. Dorn, William W. Brennessel and Daniel J. Weix*

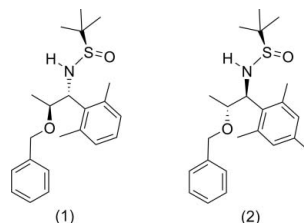
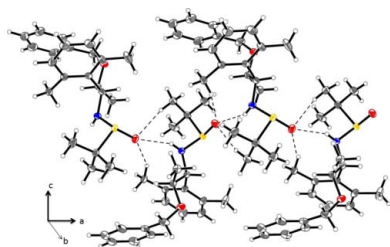
Department of Chemistry, 120 Trustee Road, 412 Hutchison Hall, University of Rochester, Rochester, NY 14627, USA.

*Correspondence e-mail: weix@chem.rochester.edu

The title compounds, C₂₂H₃₁NO₂S, (1), and C₂₃H₃₃NO₂S, (2), are related protected 1,2-amino alcohols. They differ in the substituents on the benzene ring, *viz.* 2,6-dimethylphenyl in (1) and 2,4,6-trimethylphenyl in (2). The plane of the phenyl ring is inclined to that of the benzene ring by 28.52 (7)° in (1) and by 44.65 (19)° in (2). In the crystal of (1), N—H···O=S and C—H···O=S hydrogen bonds link molecules, forming chains along [100], while in (2), similar hydrogen bonds link molecules into chains along [010]. The absolute structures of both compounds were determined by resonance scattering.

1. Chemical context

1,2-Amino alcohols are found in a variety of pharmaceutically active compounds (Lee & Kang, 2004) and have been used extensively as components of chiral ligands and auxiliaries in asymmetric synthesis (Ager *et al.*, 1996; Pu & Yu, 2001). In order to develop new chiral ligands and as part of an advanced undergraduate laboratory course, we sought to make a series of 2-aryl-1-methyl-1,2-amino alcohols. The most straightforward synthesis of these compounds was reported by Ellman (Tang *et al.*, 2001; Evans & Ellman, 2003). The method relies upon the chiral ammonia equivalent, 2-methyl-2-propane-sulfonamide (*tert*-butanesulfonamide), which is readily available from a variety of commercial sources or easily synthesized on scale (Weix *et al.*, 2005). In the original Ellman report, the absolute configuration of the products was determined by deprotection of the amine and alcohol, cyclization to form the corresponding oxazolidinone, and correlation of the ¹H NMR spectra with the literature (Zietlow & Steckhan, 1994).



We report herein on the syntheses and structures of two different but related protected 1,2-amino alcohols, (1) and (2),

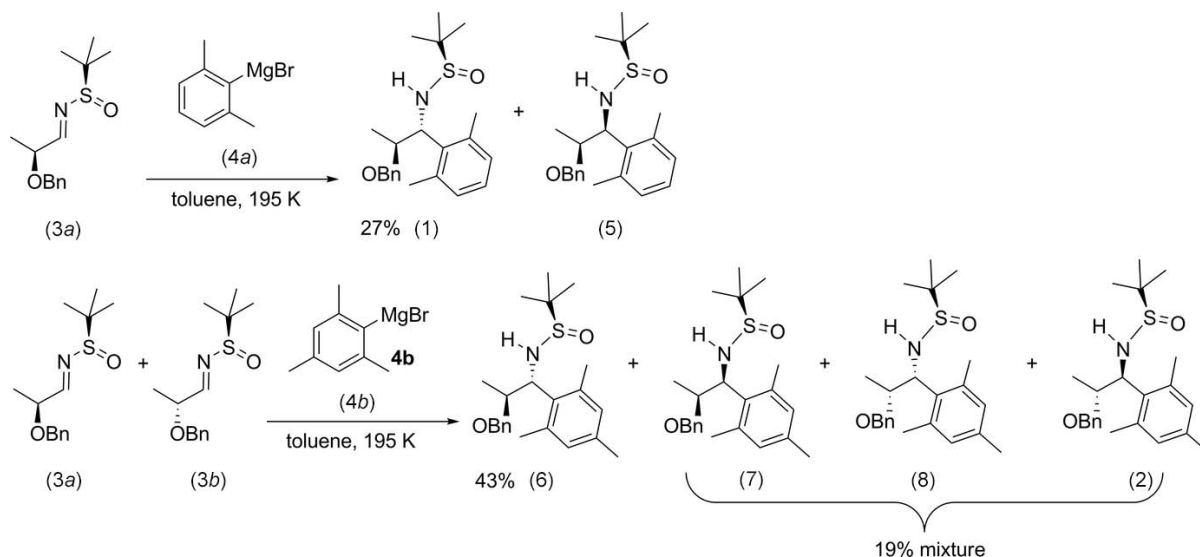


Figure 1

(Top) Reaction scheme depicting the synthesis of (1) and (5) from (3a), for which (1) is the major product of the reaction. (Bottom) Reaction scheme depicting the synthesis of (6) and (7) from (3a), and (8) and (2) from (3b), for which (6) is the major product of the reaction from (3a), and (8) is the major product from (3b).

from the addition of an arylmagnesium bromide to an *N*-*tert*-butanesulfinyl imine (Evans & Ellman, 2003). The reaction of imine (3a) with xylylmagnesium bromide, (4a), (see Fig. 1) resulted in a mixture of amino alcohol products from which the major product of the reaction, (1), was isolated in 27% yield after chromatographic separation of the diastereomers. The stereochemistry of this major product was

confirmed by X-ray diffraction and the result is consistent with the sense of induction reported by Evans & Ellman (2003).

The analogous reaction with mesitylmagnesium bromide, (4b), also resulted in a mixture of products, from which the major product, (6), was isolated in 43% yield. A mixture of other diastereomers was also isolated, from which a crystal suitable for X-ray diffraction was grown. Unexpectedly, X-ray

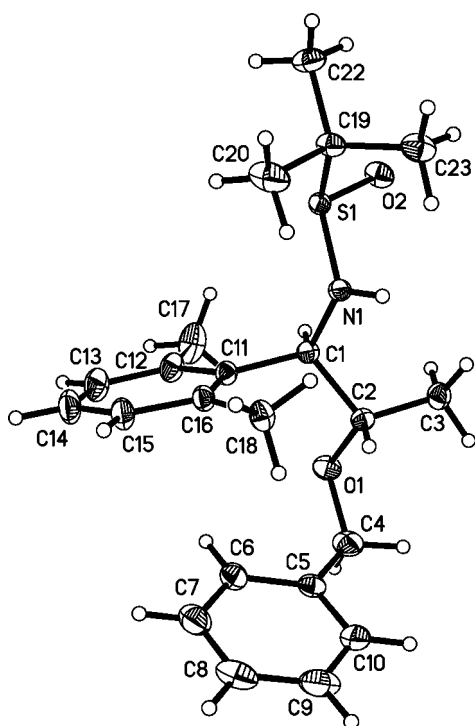


Figure 2

The molecular structure of compound (1), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

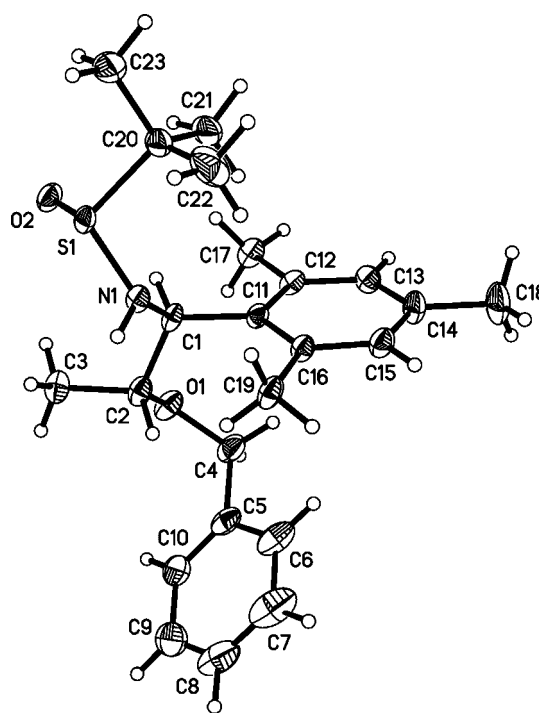


Figure 3

The molecular structure of compound (2), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

Table 1
Hydrogen-bond geometry (Å, °) for (1).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O2^i$	0.84 (2)	2.23 (2)	3.0039 (15)	152.8 (7)
$C18-H18A\cdots O2^i$	0.98	2.52	3.4077 (17)	150
$C23-H23B\cdots O2^i$	0.98	2.59	3.5534 (17)	167

Symmetry code: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z$.

Table 2
Hydrogen-bond geometry (Å, °) for (2).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O2^i$	0.83 (4)	2.08 (4)	2.890 (4)	169 (4)
$C7-H7A\cdots O1^{ii}$	0.95	2.59	3.501 (6)	160

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

analysis showed this crystal to be (2), a product that could only have derived from a diastereomerically different isomer of (3a). Upon further investigation, we discovered that the starting material, which we had assumed was pure (3a), contained the minor diastereomer, (3b), in about 8% (determined by $^1\text{H NMR}$; Fontenelle *et al.*, 2014), which had formed due to racemization in the synthesis of (3a). Based on the work of Evans & Ellman (2003), it was deduced that (2) is the *minor* product expected from the reaction of (3b) with an arylmagnesium bromide. Although no further separations were performed on this mixture that contained (2), it follows that the other diastereomers present were (7), the minor product from the reaction with (3a), and (8), the major product from the reaction with the slight impurity of (3b).

2. Structural commentary

The molecular structures of compounds (1) and (2) are illustrated in Figs. 2 and 3, respectively. The essential difference in the conformation of the two compounds is that the phenyl ring (C5–C10) is inclined to the benzene ring (C11–C16) by 28.52 (7)° in (1) and by 44.65 (19)° in (2).

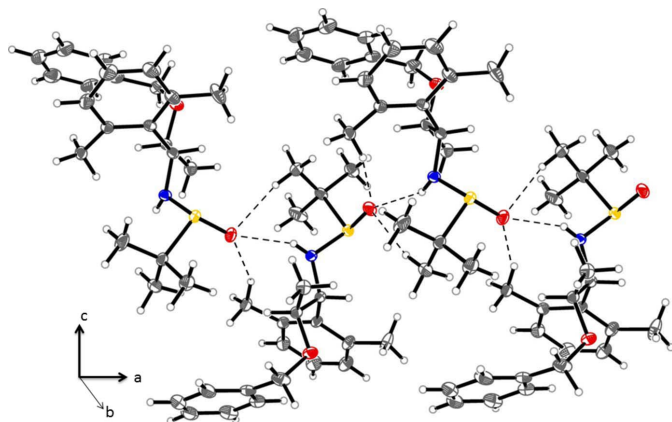


Figure 4
A partial view of the crystal packing of compound (1), illustrating the formation of the hydrogen-bonded chains along [100] (hydrogen bonds are shown as dashed lines; see Table 1 for details). Displacement ellipsoids are drawn at the 50% probability level.

3. Supramolecular features

In the crystals of both (1) and (2), chains are formed *via* intermolecular hydrogen bonding (Tables 1 and 2). In (1), molecules are linked along the [100] direction by a combination of classical ($N-H\cdots O=S$) and non-classical ($C-H\cdots O=S$) hydrogen bonds (Table 1 and Fig. 4). In (2), molecules are linked along the [010] direction also by classical ($N-H\cdots O=S$) and non-classical ($C-H\cdots O=S$) hydrogen bonds (Table 2 and Fig. 5).

4. Database survey

Although there are 78 structures of *N*-sulfinyl-protected 1,2-amino alcohols in the Cambridge Structural Database (CSD, Version 5.35, last update May 2014; Groom & Allen, 2014), only seven of these structures have substitution at the 1-position and an aryl group at the 2-position. Of these compounds, only three have a *tert*-butanesulfinyl group [CSD refcodes CAVQOG (Zhong *et al.*, 2005), FIZBIB (Jiang *et al.*, 2014) and WOBNEI (Buesking & Ellman, 2014)], and the other four contain *p*-toluenesulfinyl groups [CSD refcodes PAQZIR (Zhao *et al.*, 2005), RUXZUG (Ghorai *et al.*, 2010), WADYOR (Fadlalla *et al.*, 2010) and SICSII (Guo *et al.*, 2012)]. However, none of these seven compounds were synthesized by our method of interest.

5. Synthesis and crystallization

The starting sulfinamide, (*R,E*)-*N*-(2-(benzyloxy)propylidene)-2-methylpropane-2-sulfinamide, (3a), was prepared from *S*-ethyl lactate (Enders *et al.*, 2002; Evans & Ellman, 2003). Grignard reagents (4a) and (4b) were prepared from 2-bromoxylene and 2-bromomesitylene, respectively (Tilstam & Weinmann, 2002). The synthesis of the title compounds is illustrated in Fig. 1.

General procedure

To an oven-dried 50 ml Schlenk flask equipped with a magnetic stirrer bar and a rubber septum, sulfinamide (3a) and

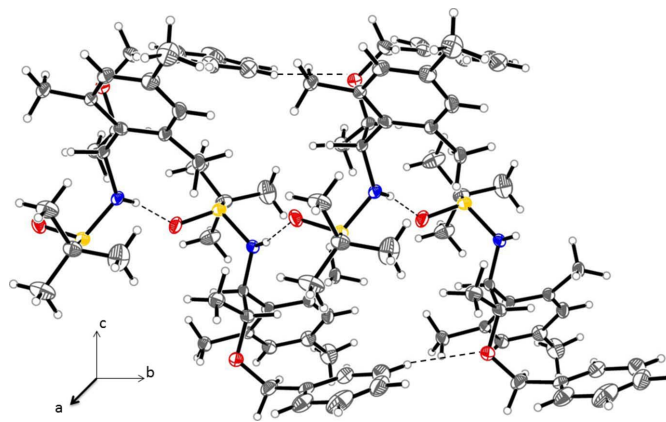


Figure 5
A partial view of the crystal packing of compound (2), illustrating the formation of the hydrogen-bonded chains along [010] (hydrogen bonds are shown as dashed lines; see Table 2 for details). Displacement ellipsoids are drawn at the 50% probability level.

Table 3
Experimental details.

	(1)	(2)
Crystal data		
Chemical formula	C ₂₂ H ₃₁ NO ₂ S	C ₂₃ H ₃₃ NO ₂ S
<i>M_r</i>	373.54	387.56
Crystal system, space group	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	100	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.1567 (13), 10.2951 (15), 22.494 (3)	10.535 (3), 7.984 (2), 13.481 (4)
α , β , γ (°)	90, 90, 90	90, 103.519 (5), 90
<i>V</i> (Å ³)	2120.5 (5)	1102.5 (5)
<i>Z</i>	4	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.17	0.16
Crystal size (mm)	0.40 × 0.25 × 0.20	0.50 × 0.14 × 0.10
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker SMART APEXII CCD platform
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2014)	Multi-scan (<i>SADABS</i> ; Bruker, 2014)
<i>T</i> _{min} , <i>T</i> _{max}	0.642, 0.748	0.564, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	74315, 11731, 10413	18025, 6191, 4675
<i>R</i> _{int}	0.041	0.074
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.879	0.695
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.039, 0.096, 1.09	0.055, 0.126, 1.01
No. of reflections	11731	6191
No. of parameters	245	255
No. of restraints	0	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.40, -0.30	0.72, -0.32
Absolute structure	Flack <i>x</i> determined using 4260 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)	Flack <i>x</i> determined using 1713 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.005 (12)	0.03 (6)

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS2013*, *SHELXL2014* and *SHELXTL* (Sheldrick, 2008).

toluene (20 ml) were added and the mixture was cooled to 195 K under nitrogen. The Grignard reagent (*4a*) or (*4b*) in toluene was placed under positive nitrogen pressure and was added to the Schlenk flask dropwise by cannula at 195 K. The reaction was stirred at 195 K and stopped when complete consumption of the imine was confirmed by thin-layer chromatography (30% ethyl acetate in hexanes, stained with ceric ammonium molybdate). The reaction was quenched with aqueous saturated sodium sulfate (1.5 ml), then the mixture was warmed to room temperature, dried over sodium sulfate, filtered through Celite, and the solvent was removed under reduced pressure. The ratio of diastereomers was determined by ¹H NMR of the crude material, specifically by examining the amine (N–H) proton resonances. The chemical shifts of *anti* diastereomers like (1) and (6) were found around $\delta = 3.78$ p.p.m., while those for *syn* diastereomers were found slightly further upfield at $\delta = 3.61$ (mixture, see below) and 3.66 (5) p.p.m.. The crude viscous yellow oil was purified by column chromatography. Crystals suitable for single-crystal X-ray diffraction were obtained from slow evaporation of methanol solutions.

(*R_S*)-*N*-[(1*R*,2*S*)-2-benzyloxy-1-(2,6-dimethylphenyl)propyl]-2-methylpropane-2-sulfonamide (1):

The reaction of sulfonamide (*3a*) (0.631 g, 2.36 mmol) with xylilmagnesium bromide [(*4a*), 3.80 equiv, 8.87 mmol],

performed according to the general procedure, yielded a 2.5:1 ratio of diastereomers, (1) to (5), respectively (see Fig. 1). The light-yellow oil was purified by column chromatography (100% diethyl ether) to yield a light-yellow solid (239 mg, 27%).

(1): m.p.: 346–348 K, ¹H NMR (500 MHz, CDCl₃): δ 1.20 (*d*, *J* = 0.3, 9H), 1.32 (*d*, *J* = 6.1, 3H), 2.36 (*s*, 3H), 2.43 (*s*, 3H), 3.71–3.70 (*m*, 1H), 3.99 (*td*, *J* = 6.7, 0.3, 1H), 4.27 (*d*, *J* = 11.8, 1H), 4.39 (*d*, *J* = 11.8, 1H), 4.92–4.89 (*m*, 1H), 6.96–6.94 (*m*, 1H), 7.02–7.01 (*m*, 3H), 7.08 (*d*, *J* = 7.6, 1H), 7.22 (*d*, *J* = 4.6, 3H). ¹³C NMR (126 MHz, CDCl₃): δ 17.65, 21.62, 21.77, 22.71, 55.48, 59.01, 71.27, 76.41, 127.49, 127.60, 127.85, 128.35, 128.50, 130.43, 134.91, 137.22, 138.32, 138.57. IR (neat): 3271, 1084, 1041 cm⁻¹. Analysis calculated for C₂₂H₃₁NO₂S (%), 70.74 C, 8.36 H, 3.75 N, found (%) 70.99 C, 8.58 H, 3.66 N.

(*R_S*)-*N*-[(1*S*,2*R*)-2-benzyloxy-1-(2,4,6-trimethylphenyl)propyl]-2-methylpropane-2-sulfonamide (2):

The reaction of sulfonamide (*3a*) (0.757 g, 2.83 mmol), which contained an impurity (8%) of sulfonamide (*3b*), with mesitylmagnesium bromide [(*4b*), 3.00 equiv, 8.50 mmol] in toluene, performed according to the general procedure, yielded a mixture of *anti* and *syn* diastereomers. The light-yellow oil was purified by column chromatography (80% diethyl ether in hexanes) to yield two white solids. The first was the expected major product (6) (467 mg, 43%). The

second (207 mg, 19%) was determined to be a mixture of diastereomers (based on ^1H NMR) that contained (2) (confirmed by X-ray crystallography) and two others, likely (7) and (8) (see Fig. 1). No further characterization or separation was performed on this mixture.

(6): ^1H NMR (500 MHz, CDCl_3): δ 1.17 (s, 9H), 1.29 (d, $J = 6.1$, 3H), 2.26 (s, 3H), 2.33 (s, 3H), 2.39 (s, 3H), 3.72–3.71 (m, 1H), 3.98–3.95 (m, 1H), 4.29 (d, $J = 11.9$, 1H), 4.39 (d, $J = 11.8$, 1H), 4.88–4.86 (m, 1H), 6.77 (s, 1H), 6.84 (s, 1H), 7.06 (d, $J = 4.3$, 2H), 7.22 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3): δ 17.61, 20.97, 21.56, 21.65, 22.76, 55.44, 58.65, 58.67, 71.30, 76.66, 127.58, 127.88, 128.34, 129.38, 130.80, 131.22, 137.13, 138.45. IR (neat): 3271, 1057 cm^{-1} . Analysis calculated for $\text{C}_{23}\text{H}_{33}\text{NO}_2\text{S}$ (%), 71.27 C, 8.58 H, 3.61 N, found (%) 70.55 C, 8.62 H, 3.49 N.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For (1), the absolute configuration was determined using 4260 quotients, which gave a Flack parameter of 0.005 (12). The value obtained without $D_{\text{obs}}(\text{h})$ as a restraint was -0.02 (3), calculated from 5203 Friedel pairs. For (2), the absolute configuration was determined using 1713 quotients, which gave a Flack parameter of 0.03 (6). The value obtained without $D_{\text{obs}}(\text{h})$ as a restraint was -0.04 (8), calculated from 2882 Friedel pairs. In (2), the needle-shaped crystal diffracted weakly at higher angles. The cut-off resolution of 0.72 Å was chosen to maximize the number of enantiomer-determining reflections, while limiting the inclusion of very weak high-angle data. The largest residual peak of 0.72 e Å $^{-3}$ is located in the S1–C20 bond.

For both structures, the amine H atoms were located from difference Fourier maps and freely refined. The C-bound H atoms were placed geometrically and treated as riding with C–H = 0.95–1.00 Å and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and = $1.2U_{\text{eq}}(\text{C})$ for other H atoms.

Acknowledgements

The authors thank Keywan Johnson and Kierra Huihui for their expert guidance with the synthetic work and the University of Rochester Chemistry Department (CHM 234) for financial support.

References

- Ager, D. J., Prakash, I. & Schaad, D. R. (1996). *Chem. Rev.* **96**, 835–876.
- Bruker (2014). *APEX2, SAINT and SADABS*. Bruker AXS, Inc., Madison, Wisconsin, USA.
- Buesking, A. W. & Ellman, J. A. (2014). *Chem. Sci.* **5**, 1983–1987.
- Enders, D., von Berg, S., Jandeleit, B., Savall, B. M. & Roush, W. R. (2002). *Org. Synth.* **78**, 177–182.
- Evans, J. W. & Ellman, J. A. (2003). *J. Org. Chem.* **68**, 9948–9957.
- Fadlalla, M. I., Friedrich, H. B., Maguire, G. E. M. & Omondi, B. (2010). *Acta Cryst.* **E66**, o3279–o3280.
- Fontenelle, C. Q., Conroy, M., Light, M., Poisson, T., Pannecoucke, X. & Linclau, B. (2014). *J. Org. Chem.* **79**, 4186–4195.
- Ghorai, M. K., Kumar, A. & Tiwari, D. P. (2010). *J. Org. Chem.* **75**, 137–151.
- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed. Engl.* **53**, 662–671.
- Guo, Y.-L., Bai, J.-F., Peng, L., Wang, L.-L., Jia, L.-N., Luo, X.-Y., Tian, F., Xu, X.-Y. & Wang, L.-X. (2012). Private communication (refcode SICSII). CCDC, Cambridge, England.
- Jiang, J.-L., Yao, M. & Lu, C.-D. (2014). *Org. Lett.* **16**, 318–321.
- Lee, H.-S. & Kang, S. H. (2004). *Synlett*, pp. 1673–1685.
- Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* **B69**, 249–259.
- Pu, L. & Yu, H.-B. (2001). *Chem. Rev.* **101**, 757–824.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tang, T. P., Volkman, S. K. & Ellman, J. A. (2001). *J. Org. Chem.* **66**, 8772–8778.
- Tilstam, U. & Weinmann, H. (2002). *Org. Process Res. Dev.* **6**, 906–910.
- Weix, D. J., Ellman, J. A., Wang, X. & Curran, D. P. (2005). *Org. Synth.* **82**, 157–165.
- Zhao, Y., Jiang, N., Chen, S., Peng, C., Zhang, X., Zou, Y., Zhang, S. & Wang, J. (2005). *Tetrahedron*, **61**, 6546–6552.
- Zhong, Y.-W., Dong, Y.-Z., Fang, K., Izumi, K., Xu, M.-H. & Lin, G.-Q. (2005). *J. Am. Chem. Soc.* **127**, 11956–11957.
- Zietlow, A. & Steckhan, E. (1994). *J. Org. Chem.* **59**, 5658–5661.

supporting information

Acta Cryst. (2014). E70, 365-369 [doi:10.1107/S1600536814022570]

Crystal structures of (*R*_S)-*N*-[(1*R*,2*S*)-2-benzyloxy-1-(2,6-dimethylphenyl)propyl]-2-methylpropane-2-sulfinamide and (*R*_S)-*N*-[(1*S*,2*R*)-2-benzyloxy-1-(2,4,6-trimethylphenyl)propyl]-2-methylpropane-2-sulfinamide: two related protected 1,2-amino alcohols

Matthew R. Carbone, Garrick A. Centola, Adam Haas, Kevin P. McClelland, Michael D. Moskowicz, Angelo M. Verderame, Mikael S. Olezeski, Louis J. Papa, Stephanie C. M. Dorn, William W. Brennessel and Daniel J. Weix

Computing details

For both compounds, data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(1) (*R*_S)-*N*-[(1*R*,2*S*)-2-Benzyloxy-1-(2,6-dimethylphenyl)propyl]-2-methylpropane-2-sulfinamide

Crystal data

C₂₂H₃₁NO₂S

*M*_r = 373.54

Orthorhombic, *P*2₁2₁2₁

a = 9.1567 (13) Å

b = 10.2951 (15) Å

c = 22.494 (3) Å

V = 2120.5 (5) Å³

Z = 4

F(000) = 808

*D*_x = 1.170 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3707 reflections

θ = 2.4–38.1°

μ = 0.17 mm⁻¹

T = 100 K

Block, colourless

0.40 × 0.25 × 0.20 mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2014)

*T*_{min} = 0.642, *T*_{max} = 0.748

74315 measured reflections

11731 independent reflections

10413 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.041

θ_{max} = 38.7°, θ_{min} = 1.8°

h = -16→15

k = -17→17

l = -39→39

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.096$ $S = 1.09$

11731 reflections

245 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.2202P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.40 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -0.30 \text{ e } \text{Å}^{-3}$ Absolute structure: Flack x determined using
4260 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: 0.005 (12)

*Special details***Experimental.** Dry solvents were prepared from ACS grade, inhibitor free solvents by passage through activated molecular sieves in an Innovative Technology solvent purification system. CDCl_3 was purchased from Cambridge Isotope Laboratories, Inc., and dried over molecular sieves. ^1H and ^{13}C NMR spectra were recorded on an Avance 500 MHz spectrometer with residual protiated solvent as a reference.**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.**Refinement.** The amine H atom was found from the difference Fourier map and refined freely. All other H atoms were placed geometrically and treated as riding atoms: methine, C—H = 1.00 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, methylene, C—H = 0.99 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, methyl, C—H = 0.98 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, sp^2 , C—H = 0.95 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.The absolute configuration was determined using 4260 quotients, which gave a Flack parameter of 0.005 (12) (Parsons and Flack, 2004, Parsons *et al.*, 2013). The value obtained without $D_{\text{obs}}(\mathbf{h})$ as a restraint was -0.02 (3), calculated from 5203 Friedel pairs (Flack, 1983).*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.79149 (3)	0.10142 (3)	0.03041 (2)	0.01557 (5)
O1	0.91190 (11)	0.06660 (10)	-0.18104 (4)	0.02226 (18)
O2	0.68782 (10)	0.21396 (10)	0.02920 (4)	0.02241 (17)
N1	0.92290 (11)	0.11541 (10)	-0.01896 (4)	0.01623 (16)
H1	0.977 (2)	0.1819 (19)	-0.0167 (8)	0.021 (4)*
C1	0.89269 (12)	0.05350 (11)	-0.07694 (5)	0.01571 (17)
H1A	0.7843	0.0558	-0.0823	0.019*
C2	0.95786 (13)	0.13364 (12)	-0.12820 (5)	0.01660 (18)
H2A	1.0669	0.1337	-0.1255	0.020*
C3	0.90089 (15)	0.27252 (13)	-0.12894 (6)	0.0217 (2)
H3A	0.9377	0.3191	-0.0940	0.033*
H3B	0.7939	0.2716	-0.1281	0.033*
H3C	0.9344	0.3162	-0.1652	0.033*
C4	1.00691 (18)	0.08699 (16)	-0.23019 (6)	0.0281 (3)
H4A	1.0292	0.1809	-0.2332	0.034*
H4B	0.9559	0.0609	-0.2671	0.034*
C5	1.14836 (16)	0.01255 (14)	-0.22566 (5)	0.0225 (2)

C6	1.14793 (17)	-0.11740 (14)	-0.20832 (6)	0.0253 (2)
H6A	1.0585	-0.1582	-0.1977	0.030*
C7	1.2770 (2)	-0.18760 (17)	-0.20640 (7)	0.0326 (3)
H7A	1.2755	-0.2765	-0.1951	0.039*
C8	1.40849 (19)	-0.12847 (19)	-0.22088 (7)	0.0349 (4)
H8A	1.4968	-0.1769	-0.2198	0.042*
C9	1.4105 (2)	0.0017 (2)	-0.23699 (8)	0.0382 (4)
H9A	1.5004	0.0428	-0.2463	0.046*
C10	1.2812 (2)	0.07163 (15)	-0.23950 (7)	0.0319 (3)
H10A	1.2831	0.1606	-0.2507	0.038*
C11	0.93628 (12)	-0.08950 (11)	-0.07699 (5)	0.01664 (18)
C12	0.82885 (15)	-0.18286 (13)	-0.09100 (6)	0.0229 (2)
C13	0.86506 (17)	-0.31517 (14)	-0.09039 (7)	0.0290 (3)
H13A	0.7934	-0.3778	-0.1009	0.035*
C14	1.00387 (18)	-0.35576 (14)	-0.07467 (8)	0.0300 (3)
H14A	1.0265	-0.4458	-0.0733	0.036*
C15	1.10962 (16)	-0.26432 (13)	-0.06090 (7)	0.0247 (2)
H15A	1.2046	-0.2925	-0.0499	0.030*
C16	1.07903 (13)	-0.13087 (11)	-0.06295 (5)	0.01810 (19)
C17	0.67364 (17)	-0.14536 (17)	-0.10624 (9)	0.0355 (4)
H17A	0.6180	-0.2234	-0.1166	0.053*
H17B	0.6740	-0.0855	-0.1401	0.053*
H17C	0.6283	-0.1028	-0.0719	0.053*
C18	1.20616 (14)	-0.04049 (12)	-0.05286 (6)	0.0209 (2)
H18A	1.1704	0.0421	-0.0368	0.031*
H18B	1.2564	-0.0248	-0.0907	0.031*
H18C	1.2743	-0.0800	-0.0246	0.031*
C19	0.90048 (13)	0.12273 (13)	0.09851 (5)	0.0195 (2)
C20	1.01500 (19)	0.01501 (19)	0.09980 (7)	0.0349 (4)
H20A	1.0883	0.0312	0.0689	0.052*
H20B	0.9677	-0.0689	0.0925	0.052*
H20C	1.0625	0.0135	0.1388	0.052*
C22	0.78986 (16)	0.10520 (16)	0.14911 (5)	0.0259 (2)
H22A	0.7145	0.1725	0.1463	0.039*
H22B	0.8401	0.1126	0.1874	0.039*
H22C	0.7443	0.0193	0.1460	0.039*
C23	0.96837 (16)	0.25766 (16)	0.10044 (6)	0.0267 (3)
H23A	0.8923	0.3232	0.0941	0.040*
H23B	1.0423	0.2651	0.0691	0.040*
H23C	1.0142	0.2715	0.1393	0.040*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01487 (10)	0.01513 (10)	0.01672 (10)	0.00088 (9)	0.00119 (9)	0.00003 (9)
O1	0.0245 (4)	0.0271 (5)	0.0152 (3)	-0.0001 (3)	-0.0034 (3)	-0.0033 (3)
O2	0.0182 (4)	0.0259 (4)	0.0231 (4)	0.0091 (3)	-0.0017 (3)	-0.0013 (3)
N1	0.0168 (4)	0.0165 (4)	0.0154 (3)	-0.0027 (3)	0.0016 (3)	-0.0019 (3)

C1	0.0152 (4)	0.0166 (4)	0.0154 (4)	-0.0020 (3)	-0.0009 (3)	-0.0014 (3)
C2	0.0169 (4)	0.0184 (5)	0.0146 (4)	-0.0004 (3)	-0.0011 (3)	0.0000 (3)
C3	0.0229 (5)	0.0184 (5)	0.0239 (5)	0.0019 (4)	-0.0007 (4)	0.0023 (4)
C4	0.0382 (7)	0.0317 (7)	0.0144 (4)	0.0072 (6)	0.0002 (4)	0.0015 (4)
C5	0.0301 (6)	0.0240 (6)	0.0135 (4)	0.0004 (5)	0.0017 (4)	-0.0037 (4)
C6	0.0323 (6)	0.0226 (6)	0.0209 (5)	-0.0008 (5)	0.0010 (4)	-0.0038 (4)
C7	0.0416 (8)	0.0305 (7)	0.0257 (6)	0.0083 (6)	0.0018 (6)	-0.0029 (5)
C8	0.0314 (7)	0.0483 (10)	0.0250 (6)	0.0096 (7)	0.0002 (5)	-0.0110 (6)
C9	0.0316 (7)	0.0491 (10)	0.0340 (8)	-0.0079 (7)	0.0110 (6)	-0.0155 (7)
C10	0.0405 (8)	0.0281 (7)	0.0271 (6)	-0.0050 (6)	0.0128 (6)	-0.0057 (5)
C11	0.0179 (4)	0.0146 (4)	0.0174 (4)	-0.0029 (3)	-0.0008 (3)	-0.0011 (3)
C12	0.0229 (5)	0.0199 (5)	0.0260 (5)	-0.0079 (4)	-0.0033 (4)	0.0004 (4)
C13	0.0312 (7)	0.0193 (6)	0.0365 (7)	-0.0105 (5)	-0.0017 (6)	-0.0015 (5)
C14	0.0348 (7)	0.0145 (5)	0.0406 (8)	-0.0033 (5)	0.0023 (6)	-0.0007 (5)
C15	0.0250 (6)	0.0156 (5)	0.0336 (6)	0.0002 (4)	0.0010 (5)	0.0000 (4)
C16	0.0190 (5)	0.0144 (4)	0.0209 (5)	-0.0013 (3)	0.0002 (4)	-0.0014 (3)
C17	0.0242 (6)	0.0292 (7)	0.0531 (9)	-0.0119 (5)	-0.0154 (6)	0.0066 (7)
C18	0.0168 (4)	0.0177 (5)	0.0283 (5)	-0.0006 (4)	-0.0026 (4)	-0.0021 (4)
C19	0.0182 (4)	0.0241 (6)	0.0162 (4)	0.0074 (4)	-0.0004 (3)	0.0003 (4)
C20	0.0365 (8)	0.0441 (9)	0.0242 (6)	0.0263 (7)	-0.0023 (5)	0.0011 (6)
C22	0.0270 (5)	0.0338 (6)	0.0169 (4)	0.0053 (6)	0.0028 (4)	0.0038 (4)
C23	0.0219 (5)	0.0349 (7)	0.0232 (5)	-0.0027 (5)	-0.0020 (4)	-0.0066 (5)

Geometric parameters (Å, °)

S1—O2	1.4980 (9)	C11—C16	1.4106 (17)
S1—N1	1.6436 (10)	C11—C12	1.4109 (16)
S1—C19	1.8415 (12)	C12—C13	1.402 (2)
O1—C4	1.4225 (17)	C12—C17	1.512 (2)
O1—C2	1.4374 (14)	C13—C14	1.384 (2)
N1—C1	1.4778 (14)	C13—H13A	0.9500
N1—H1	0.84 (2)	C14—C15	1.386 (2)
C1—C11	1.5253 (17)	C14—H14A	0.9500
C1—C2	1.5383 (16)	C15—C16	1.4029 (18)
C1—H1A	1.0000	C15—H15A	0.9500
C2—C3	1.5221 (18)	C16—C18	1.5075 (17)
C2—H2A	1.0000	C17—H17A	0.9800
C3—H3A	0.9800	C17—H17B	0.9800
C3—H3B	0.9800	C17—H17C	0.9800
C3—H3C	0.9800	C18—H18A	0.9800
C4—C5	1.508 (2)	C18—H18B	0.9800
C4—H4A	0.9900	C18—H18C	0.9800
C4—H4B	0.9900	C19—C23	1.522 (2)
C5—C6	1.394 (2)	C19—C20	1.5266 (18)
C5—C10	1.395 (2)	C19—C22	1.5342 (17)
C6—C7	1.386 (2)	C20—H20A	0.9800
C6—H6A	0.9500	C20—H20B	0.9800
C7—C8	1.388 (3)	C20—H20C	0.9800

C7—H7A	0.9500	C22—H22A	0.9800
C8—C9	1.388 (3)	C22—H22B	0.9800
C8—H8A	0.9500	C22—H22C	0.9800
C9—C10	1.387 (3)	C23—H23A	0.9800
C9—H9A	0.9500	C23—H23B	0.9800
C10—H10A	0.9500	C23—H23C	0.9800
O2—S1—N1	112.57 (5)	C13—C12—C11	119.66 (13)
O2—S1—C19	105.45 (5)	C13—C12—C17	118.20 (12)
N1—S1—C19	98.91 (5)	C11—C12—C17	122.14 (13)
C4—O1—C2	113.13 (10)	C14—C13—C12	120.87 (13)
C1—N1—S1	114.92 (8)	C14—C13—H13A	119.6
C1—N1—H1	120.8 (13)	C12—C13—H13A	119.6
S1—N1—H1	117.1 (13)	C13—C14—C15	119.59 (13)
N1—C1—C11	111.58 (9)	C13—C14—H14A	120.2
N1—C1—C2	110.96 (9)	C15—C14—H14A	120.2
C11—C1—C2	114.56 (9)	C14—C15—C16	121.23 (13)
N1—C1—H1A	106.4	C14—C15—H15A	119.4
C11—C1—H1A	106.4	C16—C15—H15A	119.4
C2—C1—H1A	106.4	C15—C16—C11	119.22 (11)
O1—C2—C3	109.98 (10)	C15—C16—C18	116.44 (11)
O1—C2—C1	104.41 (9)	C11—C16—C18	124.26 (11)
C3—C2—C1	112.28 (10)	C12—C17—H17A	109.5
O1—C2—H2A	110.0	C12—C17—H17B	109.5
C3—C2—H2A	110.0	H17A—C17—H17B	109.5
C1—C2—H2A	110.0	C12—C17—H17C	109.5
C2—C3—H3A	109.5	H17A—C17—H17C	109.5
C2—C3—H3B	109.5	H17B—C17—H17C	109.5
H3A—C3—H3B	109.5	C16—C18—H18A	109.5
C2—C3—H3C	109.5	C16—C18—H18B	109.5
H3A—C3—H3C	109.5	H18A—C18—H18B	109.5
H3B—C3—H3C	109.5	C16—C18—H18C	109.5
O1—C4—C5	113.43 (11)	H18A—C18—H18C	109.5
O1—C4—H4A	108.9	H18B—C18—H18C	109.5
C5—C4—H4A	108.9	C23—C19—C20	112.44 (13)
O1—C4—H4B	108.9	C23—C19—C22	110.84 (11)
C5—C4—H4B	108.9	C20—C19—C22	110.73 (11)
H4A—C4—H4B	107.7	C23—C19—S1	110.71 (9)
C6—C5—C10	118.92 (14)	C20—C19—S1	107.55 (9)
C6—C5—C4	120.28 (13)	C22—C19—S1	104.21 (9)
C10—C5—C4	120.80 (13)	C19—C20—H20A	109.5
C7—C6—C5	120.45 (15)	C19—C20—H20B	109.5
C7—C6—H6A	119.8	H20A—C20—H20B	109.5
C5—C6—H6A	119.8	C19—C20—H20C	109.5
C6—C7—C8	120.25 (15)	H20A—C20—H20C	109.5
C6—C7—H7A	119.9	H20B—C20—H20C	109.5
C8—C7—H7A	119.9	C19—C22—H22A	109.5
C7—C8—C9	119.74 (16)	C19—C22—H22B	109.5

C7—C8—H8A	120.1	H22A—C22—H22B	109.5
C9—C8—H8A	120.1	C19—C22—H22C	109.5
C10—C9—C8	120.05 (16)	H22A—C22—H22C	109.5
C10—C9—H9A	120.0	H22B—C22—H22C	109.5
C8—C9—H9A	120.0	C19—C23—H23A	109.5
C9—C10—C5	120.57 (15)	C19—C23—H23B	109.5
C9—C10—H10A	119.7	H23A—C23—H23B	109.5
C5—C10—H10A	119.7	C19—C23—H23C	109.5
C16—C11—C12	119.36 (11)	H23A—C23—H23C	109.5
C16—C11—C1	122.26 (10)	H23B—C23—H23C	109.5
C12—C11—C1	118.37 (11)		
O2—S1—N1—C1	-92.54 (9)	N1—C1—C11—C12	123.09 (11)
C19—S1—N1—C1	156.53 (9)	C2—C1—C11—C12	-109.78 (12)
S1—N1—C1—C11	-86.77 (10)	C16—C11—C12—C13	0.43 (19)
S1—N1—C1—C2	144.18 (8)	C1—C11—C12—C13	-178.90 (12)
C4—O1—C2—C3	85.24 (13)	C16—C11—C12—C17	179.78 (14)
C4—O1—C2—C1	-154.11 (10)	C1—C11—C12—C17	0.45 (19)
N1—C1—C2—O1	-176.40 (9)	C11—C12—C13—C14	1.7 (2)
C11—C1—C2—O1	56.15 (12)	C17—C12—C13—C14	-177.64 (16)
N1—C1—C2—C3	-57.29 (13)	C12—C13—C14—C15	-1.7 (2)
C11—C1—C2—C3	175.25 (10)	C13—C14—C15—C16	-0.4 (2)
C2—O1—C4—C5	74.91 (15)	C14—C15—C16—C11	2.6 (2)
O1—C4—C5—C6	44.88 (17)	C14—C15—C16—C18	-174.29 (13)
O1—C4—C5—C10	-136.05 (14)	C12—C11—C16—C15	-2.54 (18)
C10—C5—C6—C7	-1.68 (19)	C1—C11—C16—C15	176.77 (11)
C4—C5—C6—C7	177.41 (12)	C12—C11—C16—C18	174.06 (12)
C5—C6—C7—C8	1.0 (2)	C1—C11—C16—C18	-6.63 (18)
C6—C7—C8—C9	0.4 (2)	O2—S1—C19—C23	-53.16 (10)
C7—C8—C9—C10	-1.0 (2)	N1—S1—C19—C23	63.36 (9)
C8—C9—C10—C5	0.3 (2)	O2—S1—C19—C20	-176.36 (10)
C6—C5—C10—C9	1.0 (2)	N1—S1—C19—C20	-59.85 (11)
C4—C5—C10—C9	-178.05 (13)	O2—S1—C19—C22	66.06 (10)
N1—C1—C11—C16	-56.23 (14)	N1—S1—C19—C22	-177.43 (9)
C2—C1—C11—C16	70.90 (14)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O2 ⁱ	0.84 (2)	2.23 (2)	3.0039 (15)	152.8 (7)
C18—H18A \cdots O2 ⁱ	0.98	2.52	3.4077 (17)	150
C23—H23B \cdots O2 ⁱ	0.98	2.59	3.5534 (17)	167

Symmetry code: (i) $x+1/2, -y+1/2, -z$.

(2) (*R*_s)-*N*-[(1*S*,2*R*)-2-Benzyloxy-1-(2,4,6-trimethylphenyl)propyl]-2-methylpropane-2-sulfinamide

Crystal data

C₂₃H₃₃NO₂S*M*_r = 387.56Monoclinic, *P*2₁*a* = 10.535 (3) Å*b* = 7.984 (2) Å*c* = 13.481 (4) Å β = 103.519 (5)°*V* = 1102.5 (5) Å³*Z* = 2*F*(000) = 420*D*_x = 1.167 Mg m⁻³Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 4086 reflections

 θ = 2.2–28.7° μ = 0.16 mm⁻¹*T* = 100 K

Needle, colorless

0.50 × 0.14 × 0.10 mm

Data collection

Bruker SMART APEXII CCD platform
diffractometer

Radiation source: fine-focus sealed tube

 ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2014)

*T*_{min} = 0.564, *T*_{max} = 0.746

18025 measured reflections

6191 independent reflections

4675 reflections with *I* > 2σ(*I*)*R*_{int} = 0.074 θ_{\max} = 29.6°, θ_{\min} = 2.0°*h* = -14→14*k* = -11→11*l* = -18→18

Refinement

Refinement on *F*²

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.055$ $wR(F^2) = 0.126$ *S* = 1.01

6191 reflections

255 parameters

1 restraint

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent

and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0563P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.72 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.32 \text{ e \AA}^{-3}$ Absolute structure: Flack *x* determined using1713 quotients [(*I*+)–(*I*-)]/[(*I*+) + (*I*-)] (Parsons *et al.*, 2013)

Absolute structure parameter: 0.03 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. The amine H atom was found from the difference Fourier map and refined freely. All other H atoms were placed geometrically and treated as riding atoms: methine, C—H = 1.00 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, methylene, C—H = 0.99 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, methyl, C—H = 0.98 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, *sp*², C—H = 0.95 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

The absolute configuration was determined using 1713 quotients, which gave a Flack parameter of 0.03 (6) (Parsons and Flack, 2004, Parsons *et al.*, 2013). The value obtained without *D*_{obs}(**h**) as a restraint was -0.04 (8), calculated from 2882 Friedel pairs (Flack, 1983).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
S1	0.14253 (7)	0.70038 (11)	0.52944 (5)	0.01937 (18)

O1	-0.1200 (3)	0.7675 (3)	0.79000 (17)	0.0277 (6)
O2	0.1009 (2)	0.5256 (3)	0.54810 (18)	0.0255 (5)
N1	0.0861 (3)	0.8363 (4)	0.6010 (2)	0.0193 (6)
H1	0.029 (4)	0.895 (5)	0.565 (3)	0.029 (11)*
C1	0.0582 (3)	0.7907 (4)	0.7007 (2)	0.0187 (7)
H1A	0.0758	0.6681	0.7107	0.022*
C2	-0.0893 (4)	0.8173 (5)	0.6958 (2)	0.0228 (7)
H2A	-0.1121	0.9380	0.6822	0.027*
C3	-0.1748 (3)	0.7098 (6)	0.6136 (2)	0.0281 (7)
H3A	-0.2659	0.7182	0.6187	0.042*
H3B	-0.1462	0.5929	0.6228	0.042*
H3C	-0.1675	0.7490	0.5463	0.042*
C4	-0.1156 (4)	0.8932 (5)	0.8670 (3)	0.0309 (9)
H4A	-0.0238	0.9283	0.8935	0.037*
H4B	-0.1465	0.8434	0.9243	0.037*
C5	-0.1973 (4)	1.0465 (5)	0.8290 (3)	0.0277 (8)
C6	-0.1330 (4)	1.1992 (6)	0.8314 (3)	0.0399 (9)
H6A	-0.0414	1.2050	0.8587	0.048*
C7	-0.2017 (6)	1.3429 (6)	0.7941 (4)	0.0542 (14)
H7A	-0.1575	1.4467	0.7949	0.065*
C8	-0.3341 (6)	1.3334 (6)	0.7562 (3)	0.0516 (14)
H8A	-0.3812	1.4306	0.7284	0.062*
C9	-0.4009 (5)	1.1822 (8)	0.7580 (3)	0.0538 (14)
H9A	-0.4931	1.1777	0.7342	0.065*
C10	-0.3300 (4)	1.0372 (6)	0.7954 (3)	0.0375 (10)
H10A	-0.3739	0.9337	0.7972	0.045*
C11	0.1486 (3)	0.8807 (4)	0.7901 (2)	0.0191 (7)
C12	0.2023 (4)	0.7877 (4)	0.8796 (2)	0.0203 (7)
C13	0.2873 (3)	0.8671 (5)	0.9617 (2)	0.0238 (7)
H13A	0.3229	0.8043	1.0216	0.029*
C14	0.3208 (4)	1.0342 (5)	0.9582 (2)	0.0276 (8)
C15	0.2652 (3)	1.1243 (5)	0.8705 (3)	0.0253 (8)
H15A	0.2866	1.2395	0.8672	0.030*
C16	0.1790 (3)	1.0514 (4)	0.7869 (2)	0.0196 (7)
C17	0.1703 (4)	0.6060 (5)	0.8916 (3)	0.0264 (8)
H17A	0.2246	0.5630	0.9557	0.040*
H17B	0.1876	0.5416	0.8343	0.040*
H17C	0.0780	0.5953	0.8927	0.040*
C18	0.4126 (4)	1.1175 (6)	1.0483 (3)	0.0416 (11)
H18A	0.4920	1.0499	1.0695	0.062*
H18B	0.3696	1.1268	1.1051	0.062*
H18C	0.4356	1.2295	1.0285	0.062*
C19	0.1231 (4)	1.1646 (4)	0.6973 (2)	0.0254 (8)
H19A	0.1332	1.2817	0.7194	0.038*
H19B	0.0303	1.1394	0.6712	0.038*
H19C	0.1697	1.1459	0.6433	0.038*
C20	0.3203 (3)	0.7019 (6)	0.5841 (2)	0.0256 (7)
C21	0.3559 (4)	0.6297 (6)	0.6915 (3)	0.0344 (9)

H21A	0.4510	0.6197	0.7141	0.052*
H21B	0.3160	0.5188	0.6916	0.052*
H21C	0.3236	0.7041	0.7380	0.052*
C22	0.3660 (4)	0.8809 (6)	0.5811 (4)	0.0463 (12)
H22A	0.4615	0.8846	0.6018	0.069*
H22B	0.3292	0.9493	0.6278	0.069*
H22C	0.3368	0.9247	0.5116	0.069*
C23	0.3749 (4)	0.5895 (7)	0.5121 (3)	0.0415 (11)
H23A	0.4705	0.5892	0.5329	0.062*
H23B	0.3469	0.6321	0.4422	0.062*
H23C	0.3422	0.4751	0.5150	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0237 (4)	0.0206 (4)	0.0128 (3)	−0.0007 (4)	0.0020 (3)	−0.0017 (4)
O1	0.0448 (16)	0.0204 (13)	0.0200 (12)	0.0009 (11)	0.0122 (11)	0.0008 (10)
O2	0.0313 (14)	0.0179 (13)	0.0265 (12)	−0.0014 (10)	0.0053 (10)	−0.0064 (10)
N1	0.0276 (16)	0.0165 (14)	0.0120 (12)	0.0023 (12)	0.0010 (11)	0.0034 (11)
C1	0.0311 (18)	0.0125 (16)	0.0119 (13)	−0.0027 (13)	0.0042 (13)	0.0000 (12)
C2	0.0339 (19)	0.0198 (17)	0.0145 (14)	−0.0023 (14)	0.0055 (13)	0.0005 (13)
C3	0.0316 (18)	0.0297 (19)	0.0234 (15)	−0.0088 (19)	0.0072 (13)	−0.0046 (18)
C4	0.041 (2)	0.029 (2)	0.0231 (18)	0.0047 (17)	0.0093 (15)	−0.0058 (15)
C5	0.042 (2)	0.026 (2)	0.0195 (16)	0.0040 (17)	0.0152 (15)	−0.0025 (15)
C6	0.058 (2)	0.030 (2)	0.039 (2)	0.002 (2)	0.0265 (18)	−0.005 (2)
C7	0.094 (4)	0.035 (3)	0.043 (3)	0.012 (3)	0.033 (3)	0.007 (2)
C8	0.095 (4)	0.037 (3)	0.023 (2)	0.027 (3)	0.015 (2)	0.0048 (19)
C9	0.057 (3)	0.072 (4)	0.0263 (19)	0.023 (3)	−0.0033 (18)	−0.015 (2)
C10	0.041 (2)	0.045 (3)	0.0253 (19)	0.007 (2)	0.0037 (17)	−0.0105 (18)
C11	0.0244 (17)	0.0188 (17)	0.0131 (14)	0.0019 (13)	0.0025 (12)	−0.0016 (12)
C12	0.0300 (19)	0.0162 (17)	0.0155 (15)	0.0033 (14)	0.0067 (13)	0.0017 (13)
C13	0.0283 (19)	0.0253 (19)	0.0154 (15)	0.0030 (15)	0.0000 (13)	0.0037 (14)
C14	0.033 (2)	0.028 (2)	0.0179 (16)	−0.0045 (16)	−0.0009 (14)	−0.0029 (14)
C15	0.032 (2)	0.0177 (17)	0.0251 (17)	−0.0022 (15)	0.0038 (15)	−0.0023 (14)
C16	0.0262 (18)	0.0167 (17)	0.0147 (14)	0.0003 (13)	0.0023 (13)	0.0007 (12)
C17	0.040 (2)	0.0201 (18)	0.0175 (16)	0.0024 (16)	0.0044 (15)	0.0042 (14)
C18	0.049 (3)	0.039 (2)	0.027 (2)	−0.010 (2)	−0.0111 (18)	−0.0025 (19)
C19	0.040 (2)	0.0143 (19)	0.0203 (16)	−0.0027 (14)	0.0040 (14)	−0.0018 (12)
C20	0.0226 (16)	0.0331 (18)	0.0203 (14)	0.0020 (18)	0.0032 (12)	−0.0016 (19)
C21	0.029 (2)	0.051 (3)	0.0208 (17)	0.0104 (18)	0.0002 (15)	−0.0013 (17)
C22	0.027 (2)	0.041 (3)	0.068 (3)	−0.0101 (19)	0.004 (2)	0.003 (2)
C23	0.030 (2)	0.064 (3)	0.031 (2)	0.008 (2)	0.0078 (17)	−0.011 (2)

Geometric parameters (Å, °)

S1—O2	1.501 (3)	C12—C13	1.402 (5)
S1—N1	1.652 (3)	C12—C17	1.507 (5)
S1—C20	1.845 (3)	C13—C14	1.384 (5)

O1—C4	1.437 (4)	C13—H13A	0.9500
O1—C2	1.437 (4)	C14—C15	1.391 (5)
N1—C1	1.487 (4)	C14—C18	1.518 (5)
N1—H1	0.82 (4)	C15—C16	1.398 (4)
C1—C11	1.529 (4)	C15—H15A	0.9500
C1—C2	1.554 (5)	C16—C19	1.513 (4)
C1—H1A	1.0000	C17—H17A	0.9800
C2—C3	1.520 (5)	C17—H17B	0.9800
C2—H2A	1.0000	C17—H17C	0.9800
C3—H3A	0.9800	C18—H18A	0.9800
C3—H3B	0.9800	C18—H18B	0.9800
C3—H3C	0.9800	C18—H18C	0.9800
C4—C5	1.515 (5)	C19—H19A	0.9800
C4—H4A	0.9900	C19—H19B	0.9800
C4—H4B	0.9900	C19—H19C	0.9800
C5—C10	1.367 (6)	C20—C22	1.511 (6)
C5—C6	1.391 (6)	C20—C21	1.522 (5)
C6—C7	1.387 (7)	C20—C23	1.529 (5)
C6—H6A	0.9500	C21—H21A	0.9800
C7—C8	1.371 (7)	C21—H21B	0.9800
C7—H7A	0.9500	C21—H21C	0.9800
C8—C9	1.400 (8)	C22—H22A	0.9800
C8—H8A	0.9500	C22—H22B	0.9800
C9—C10	1.406 (7)	C22—H22C	0.9800
C9—H9A	0.9500	C23—H23A	0.9800
C10—H10A	0.9500	C23—H23B	0.9800
C11—C16	1.403 (5)	C23—H23C	0.9800
C11—C12	1.417 (4)		
O2—S1—N1	110.67 (15)	C14—C13—C12	122.0 (3)
O2—S1—C20	104.37 (18)	C14—C13—H13A	119.0
N1—S1—C20	103.45 (16)	C12—C13—H13A	119.0
C4—O1—C2	118.0 (3)	C13—C14—C15	117.9 (3)
C1—N1—S1	122.8 (2)	C13—C14—C18	121.0 (3)
C1—N1—H1	113 (3)	C15—C14—C18	121.1 (4)
S1—N1—H1	110 (3)	C14—C15—C16	122.3 (3)
N1—C1—C11	112.3 (3)	C14—C15—H15A	118.8
N1—C1—C2	109.6 (3)	C16—C15—H15A	118.8
C11—C1—C2	113.7 (3)	C15—C16—C11	119.4 (3)
N1—C1—H1A	107.0	C15—C16—C19	116.9 (3)
C11—C1—H1A	107.0	C11—C16—C19	123.7 (3)
C2—C1—H1A	107.0	C12—C17—H17A	109.5
O1—C2—C3	105.7 (3)	C12—C17—H17B	109.5
O1—C2—C1	110.7 (3)	H17A—C17—H17B	109.5
C3—C2—C1	111.7 (3)	C12—C17—H17C	109.5
O1—C2—H2A	109.5	H17A—C17—H17C	109.5
C3—C2—H2A	109.5	H17B—C17—H17C	109.5
C1—C2—H2A	109.5	C14—C18—H18A	109.5

C2—C3—H3A	109.5	C14—C18—H18B	109.5
C2—C3—H3B	109.5	H18A—C18—H18B	109.5
H3A—C3—H3B	109.5	C14—C18—H18C	109.5
C2—C3—H3C	109.5	H18A—C18—H18C	109.5
H3A—C3—H3C	109.5	H18B—C18—H18C	109.5
H3B—C3—H3C	109.5	C16—C19—H19A	109.5
O1—C4—C5	113.6 (3)	C16—C19—H19B	109.5
O1—C4—H4A	108.8	H19A—C19—H19B	109.5
C5—C4—H4A	108.8	C16—C19—H19C	109.5
O1—C4—H4B	108.8	H19A—C19—H19C	109.5
C5—C4—H4B	108.8	H19B—C19—H19C	109.5
H4A—C4—H4B	107.7	C22—C20—C21	112.0 (3)
C10—C5—C6	120.7 (4)	C22—C20—C23	111.6 (4)
C10—C5—C4	121.6 (4)	C21—C20—C23	109.6 (4)
C6—C5—C4	117.7 (4)	C22—C20—S1	107.2 (3)
C7—C6—C5	120.4 (4)	C21—C20—S1	112.3 (2)
C7—C6—H6A	119.8	C23—C20—S1	103.9 (2)
C5—C6—H6A	119.8	C20—C21—H21A	109.5
C8—C7—C6	119.3 (5)	C20—C21—H21B	109.5
C8—C7—H7A	120.4	H21A—C21—H21B	109.5
C6—C7—H7A	120.4	C20—C21—H21C	109.5
C7—C8—C9	120.8 (4)	H21A—C21—H21C	109.5
C7—C8—H8A	119.6	H21B—C21—H21C	109.5
C9—C8—H8A	119.6	C20—C22—H22A	109.5
C8—C9—C10	119.3 (4)	C20—C22—H22B	109.5
C8—C9—H9A	120.3	H22A—C22—H22B	109.5
C10—C9—H9A	120.3	C20—C22—H22C	109.5
C5—C10—C9	119.4 (5)	H22A—C22—H22C	109.5
C5—C10—H10A	120.3	H22B—C22—H22C	109.5
C9—C10—H10A	120.3	C20—C23—H23A	109.5
C16—C11—C12	119.1 (3)	C20—C23—H23B	109.5
C16—C11—C1	122.5 (3)	H23A—C23—H23B	109.5
C12—C11—C1	118.4 (3)	C20—C23—H23C	109.5
C13—C12—C11	119.3 (3)	H23A—C23—H23C	109.5
C13—C12—C17	117.9 (3)	H23B—C23—H23C	109.5
C11—C12—C17	122.8 (3)		
O2—S1—N1—C1	27.5 (3)	C2—C1—C11—C12	98.0 (4)
C20—S1—N1—C1	-83.8 (3)	C16—C11—C12—C13	-1.7 (5)
S1—N1—C1—C11	114.3 (3)	C1—C11—C12—C13	178.9 (3)
S1—N1—C1—C2	-118.3 (3)	C16—C11—C12—C17	177.2 (3)
C4—O1—C2—C3	-146.4 (3)	C1—C11—C12—C17	-2.2 (5)
C4—O1—C2—C1	92.5 (3)	C11—C12—C13—C14	-0.1 (5)
N1—C1—C2—O1	177.6 (3)	C17—C12—C13—C14	-179.0 (4)
C11—C1—C2—O1	-55.9 (4)	C12—C13—C14—C15	1.3 (6)
N1—C1—C2—C3	60.1 (3)	C12—C13—C14—C18	179.8 (4)
C11—C1—C2—C3	-173.4 (3)	C13—C14—C15—C16	-0.7 (6)
C2—O1—C4—C5	53.4 (4)	C18—C14—C15—C16	-179.2 (4)

O1—C4—C5—C10	63.7 (5)	C14—C15—C16—C11	-1.2 (5)
O1—C4—C5—C6	-117.6 (4)	C14—C15—C16—C19	178.6 (3)
C10—C5—C6—C7	-3.7 (6)	C12—C11—C16—C15	2.3 (5)
C4—C5—C6—C7	177.6 (3)	C1—C11—C16—C15	-178.3 (3)
C5—C6—C7—C8	0.9 (6)	C12—C11—C16—C19	-177.4 (3)
C6—C7—C8—C9	2.2 (7)	C1—C11—C16—C19	2.0 (5)
C7—C8—C9—C10	-2.6 (6)	O2—S1—C20—C22	-172.3 (3)
C6—C5—C10—C9	3.3 (5)	N1—S1—C20—C22	-56.5 (3)
C4—C5—C10—C9	-178.0 (3)	O2—S1—C20—C21	-48.9 (3)
C8—C9—C10—C5	-0.2 (6)	N1—S1—C20—C21	66.9 (3)
N1—C1—C11—C16	43.8 (4)	O2—S1—C20—C23	69.4 (3)
C2—C1—C11—C16	-81.3 (4)	N1—S1—C20—C23	-174.7 (3)
N1—C1—C11—C12	-136.8 (3)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...O2 ⁱ	0.83 (4)	2.08 (4)	2.890 (4)	169 (4)
C7—H7A...O1 ⁱⁱ	0.95	2.59	3.501 (6)	160

Symmetry codes: (i) $-x, y+1/2, -z+1$; (ii) $x, y+1, z$.