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# 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. Moskowitz, Angelo M. Verderame, Mikael S. Olezeski, Louis J. Papa, Stephanie C. M. Dorn, William W. Brennessel and Daniel J. Weix***

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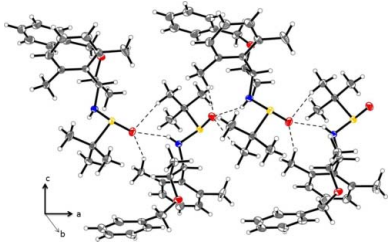
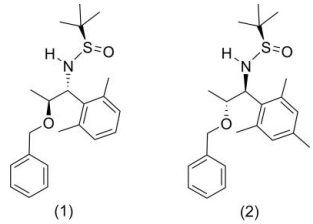
The title compounds, $C_{22}H_{31}NO_2S$, (1), and $C_{23}H_{33}NO_2S$, (2), are related protected 1,2-amino alcohols. They differ in the substituents on the benzene ring, *viz.* 2,6-dimethylphenyl in (1) and 2,4,6-trimethylphenyl in (2). The plane of the phenyl ring is inclined to that of the benzene 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-propanesulfinamide (*tert*-butanesulfinamide), 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 $^1$H 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),

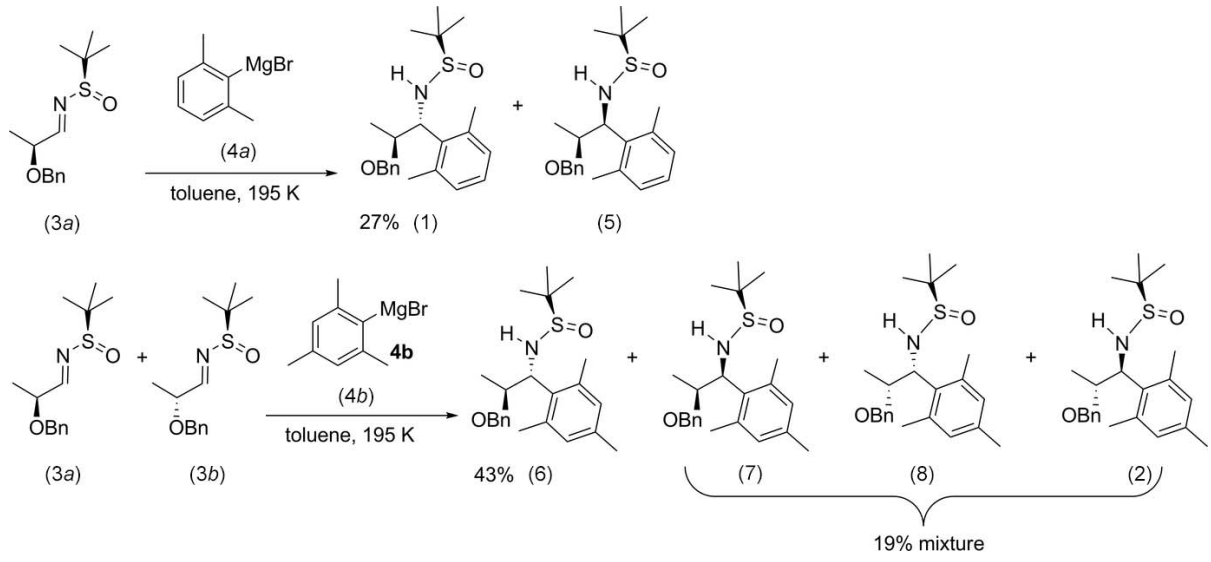



**Figure 1**
(Top) Reaction scheme depicting the synthesis of (1) and (5) from (3*a*), for which (1) is the major product of the reaction. (Bottom) Reaction scheme depicting the synthesis of (6) and (7) from (3*a*), and (8) and (2) from (3*b*), for which (6) is the major product of the reaction from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

from the addition of an arylmagnesium bromide to an *N*-*tert*-butanesulfinyl imine (Evans & Ellman, 2003). The reaction of imine (3*a*) with xylylmagnesium bromide, (4*a*), (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, (4*b*), 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

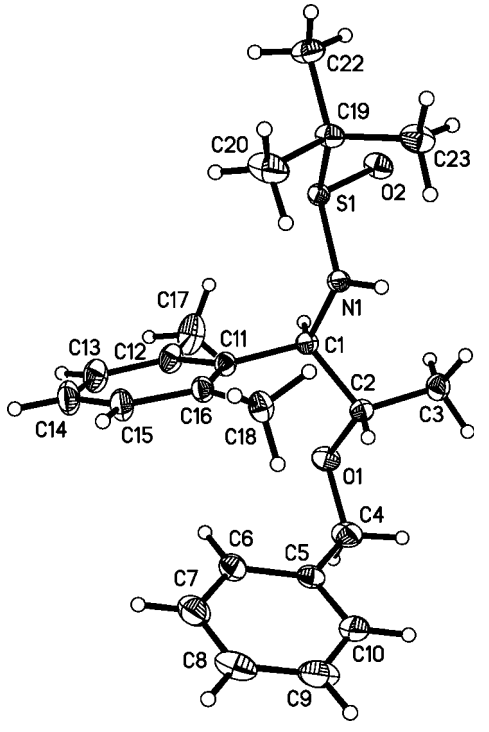



**Figure 2**
The molecular structure of compound (1), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

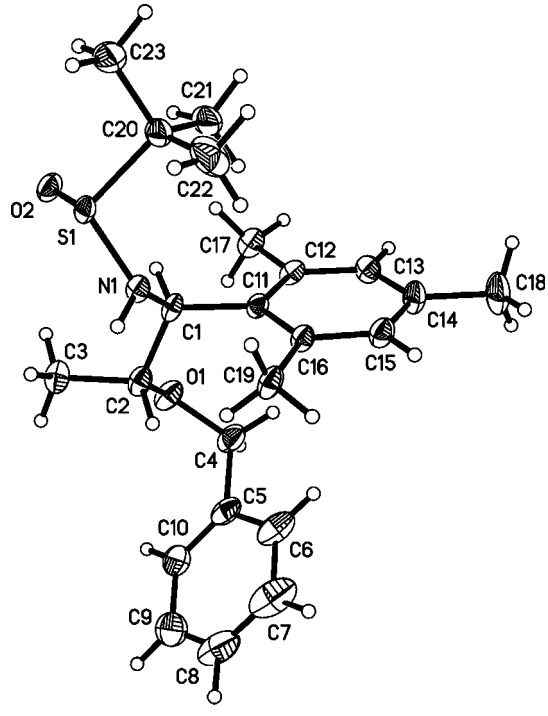



**Figure 3**
The molecular structure of compound (2), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.84 (2) | 2.23 (2) | 3.0039 (15) | 152.8 (7) |
| C18—H18$A$···O2$^{i}$ | 0.98 | 2.52 | 3.4077 (17) | 150 |
| C23—H23$B$···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···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.83 (4) | 2.08 (4) | 2.890 (4) | 169 (4) |
| C7—H7$A$···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 (3*a*). Upon further investigation, we discovered that the starting material, which we had assumed was pure (3*a*), contained the minor diastereomer, (3*b*), in about 8% (determined by $^{1}$H NMR; Fontenelle *et al.*, 2014), which had formed due to racemization in the synthesis of (3*a*). Based on the work of Evans & Ellman (2003), it was deduced that (2) is the *minor* product expected from the reaction of (3*b*) 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 (3*a*), and (8), the major product from the reaction with the slight impurity of (3*b*).

## 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).

## 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···O═S) and non-classical (C—H···O═S) hydrogen bonds (Table 1 and Fig. 4). In (2), molecules are linked along the [010] direction also by classical (N—H···O═S) and non-classical (C—H···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, (3*a*), was prepared from *S*-ethyl lactate (Enders *et al.*, 2002; Evans & Ellman, 2003). Grignard reagents (4*a*) and (4*b*) 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 (3*a*) and

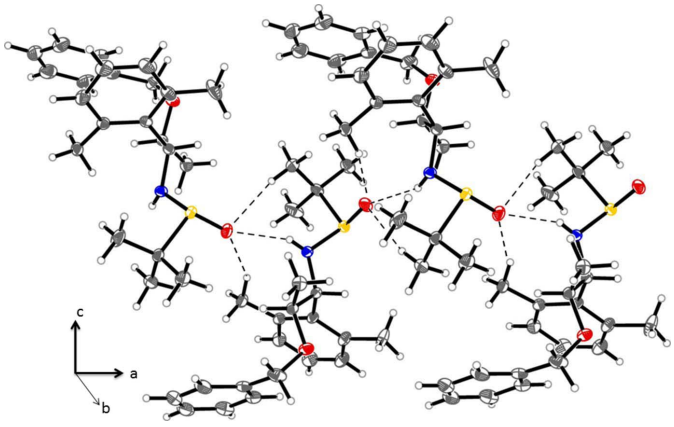



**Figure 4**
A partial view of the crystal packing of compound (1), illustrating the formation of the hydrogen-bonded chains along [100] (hydrogen bonds are shown as dashed lines; see Table 1 for details). Displacement ellipsoids are drawn at the 50% probability level.

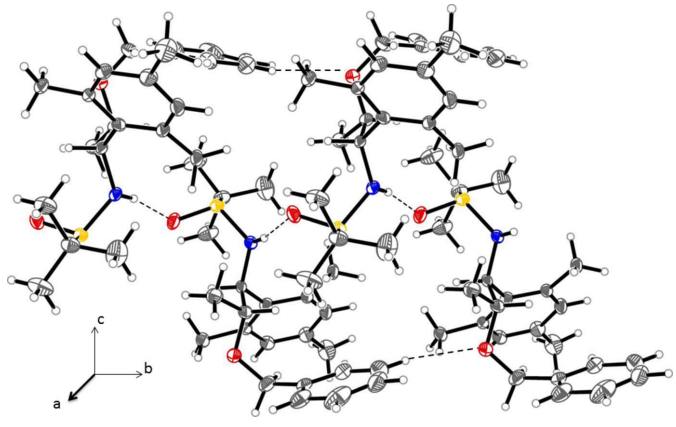



**Figure 5**
A partial view of the crystal packing of compound (2), illustrating the formation of the hydrogen-bonded chains along [010] (hydrogen bonds are shown as dashed lines; see Table 2 for details). Displacement ellipsoids are drawn at the 50% probability level.

**Table 3**
Experimental details.

| | (1) | (2) |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{22}H_{31}NO_2S$ | $C_{23}H_{33}NO_2S$ |
| $M_r$ | 373.54 | 387.56 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ | Monoclinic, $P2_1$ |
| Temperature (K) | 100 | 100 |
| $a, b, c$ (Å) | 9.1567 (13), 10.2951 (15), 22.494 (3) | 10.535 (3), 7.984 (2), 13.481 (4) |
| $\alpha, \beta, \gamma$ (°) | 90, 90, 90 | 90, 103.519 (5), 90 |
| $V$ (Å$^3$) | 2120.5 (5) | 1102.5 (5) |
| $Z$ | 4 | 2 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (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 (*SADABS*; Bruker, 2014) | Multi-scan (*SADABS*; Bruker, 2014) |
| $T_{min}$, $T_{max}$ | 0.642, 0.748 | 0.564, 0.746 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 74315, 11731, 10413 | 18025, 6191, 4675 |
| $R_{int}$ | 0.041 | 0.074 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.879 | 0.695 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 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 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.40, −0.30 | 0.72, −0.32 |
| Absolute structure | Flack $x$ determined using 4260 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) | Flack $x$ determined using 1713 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 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 (4*a*) or (4*b*) 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 $^1$H 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-sulfinamide (1):**

The reaction of sulfinamide (3*a*) (0.631 g, 2.36 mmol) with xylylmagnesium bromide [(4*a*), 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, $^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 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). $^{13}$C NMR (126 MHz, $CDCl_3$): $\delta$ 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 $C_{22}H_{31}NO_2S$ (%), 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-sulfinamide (2)**:

The reaction of sulfinamide (3*a*) (0.757 g, 2.83 mmol), which contained an impurity (8%) of sulfinamide (3*b*), with mesitylmagnesium bromide [(4*b*), 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 $^1$H 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)**: $^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 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, $CDCl_3$): $\delta$ 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 $C_{23}H_{33}NO_2S$ (%), 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 $D_{obs}$(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 $D_{obs}$(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 $U_{iso(}$H) = 1.5$U_{eq}$(C) for methyl H atoms and = 1.2$U_{eq}$(C) for other H atoms.

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# supporting information

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# 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-benzyl-oxy-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. Moskowitz, 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*

$C_{22}H_{31}NO_2S$
$M_r$ = 373.54
Orthorhombic, $P2_12_12_1$
$a$ = 9.1567 (13) Å
$b$ = 10.2951 (15) Å
$c$ = 22.494 (3) Å
$V$ = 2120.5 (5) Å$^3$
$Z$ = 4
$F$(000) = 808
$D_x$ = 1.170 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3707 reflections
$\theta$ = 2.4–38.1°
$\mu$ = 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
$\varphi$ and $\omega$ 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 $I > 2\sigma(I)$
$R_{int}$ = 0.041
$\theta_{max}$ = 38.7°, $\theta_{min}$ = 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 methods
Secondary 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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ e 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. $^1$H 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 $U_{iso}$(H) = 1.2$U_{eq}$(C), methylene, C—H = 0.99 Å with $U_{iso}$(H) = 1.2$U_{eq}$(C), methyl, C—H = 0.98 Å with $U_{iso}$(H) = 1.5$U_{eq}$(C), $sp^2$, C—H = 0.95 Å with $U_{iso}$(H) = 1.2$U_{eq}$(C).
The absolute configuration was deterimined using 4260 quotients, which gave a Flack parameter of 0.005 (12) (Parsons and Flack, 2004, Parsons *et al.*, 2013). The value obtained without $D_{obs}$(**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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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 (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.84 (2) | 2.23 (2) | 3.0039 (15) | 152.8 (7) |
| C18—H18*A*···O2$^{i}$ | 0.98 | 2.52 | 3.4077 (17) | 150 |
| C23—H23*B*···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*

$C_{23}H_{33}NO_2S$
$M_r = 387.56$
Monoclinic, $P2_1$
$a = 10.535$ (3) Å
$b = 7.984$ (2) Å
$c = 13.481$ (4) Å
$\beta = 103.519$ (5)°
$V = 1102.5$ (5) Å$^3$
$Z = 2$
$F(000) = 420$
$D_x = 1.167$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4086 reflections
$\theta = 2.2$–$28.7°$
$\mu = 0.16$ 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
$\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 29.6°$, $\theta_{min} = 2.0°$
$h = -14 \rightarrow 14$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$

*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 methods
Secondary 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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ e Å$^{-3}$
Absolute structure: Flack *x* determined using 1713 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 $U_{iso}$(H) = 1.2$U_{eq}$(C), methylene, C—H = 0.99 Å with $U_{iso}$(H) = 1.2$U_{eq}$(C), methyl, C—H = 0.98 Å with $U_{iso}$(H) = 1.5$U_{eq}$(C), $sp^2$, C—H = 0.95 Å with $U_{iso}$(H) = 1.2$U_{eq}$(C).

The absolute configuration was deterimined 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)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.83 (4) | 2.08 (4) | 2.890 (4) | 169 (4) |
| C7—H7*A*···O1$^{ii}$ | 0.95 | 2.59 | 3.501 (6) | 160 |

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