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Bis{(*R*)-*N*-[(*R*)-2-benzyloxy-1-(4-*tert*-butylphenyl)ethyl]-2-methylpropane-2-sulfinamide} monohydrate

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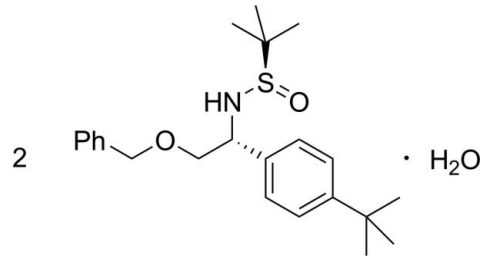
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.110; data-to-parameter ratio = 31.9.

The asymmetric unit of the title compound, $2C_{23}H_{33}NO_2S\cdot H_2O$, contains one organic molecule in a general position and one co-crystallized water molecule on a crystallographic twofold axis. Each water molecule serves as a hydrogen-bond donor to a pair of S=O acceptors on symmetry-related molecules. Thus, each trio of molecules forms one title formula unit. These groupings are further connected along [010] *via* weak non-classical C—H···O hydrogen bonds.

Related literature

For a general method to synthesize the Grignard reagent used in the reaction that generated the title material, see: Tilstam & Weinmann (2002). For in-depth discussions on methods to synthesize the precursor to the title molecule from 2-butene-1,4-diol, see: Evans *et al.* (2008); Tang *et al.* (2001). For the importance of 1,2-aminoalcohols, see: Bergmeier (2000). For methods used to determine the absolute configuration, see: Flack (1983); Parsons & Flack (2004); Parsons *et al.* (2013). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$2C_{23}H_{33}NO_2S\cdot H_2O$
M_r = 793.14
Monoclinic, $C2$
a = 21.5717 (17) Å
b = 6.1097 (5) Å
c = 17.0838 (14) Å
β = 93.2220 (17)°
V = 2248.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 100 K
0.40 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEXII CCD platform diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2012)
T_{min} = 0.662, T_{max} = 0.748
34281 measured reflections
12280 independent reflections
9359 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.110
S = 1.01
12280 reflections
385 parameters
1 restraint
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack parameter determined using 3436 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
Absolute structure parameter: −0.01 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*O*···O2	0.82 (3)	2.02 (3)	2.8420 (19)	171 (3)
C2—H2*A*···O2^{i}	0.96 (2)	2.52 (2)	3.372 (2)	148.1 (19)
C21—H21*C*···O3^{i}	0.97 (2)	2.44 (2)	3.397 (3)	169.0 (19)

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: GK2604).

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supplementary materials

Acta Cryst. (2014). E**70**, o405–o406 [doi:10.1107/S1600536814004589]

Bis{(*R*)-*N*-[(*R*)-2-benzyloxy-1-(4-*tert*-butylphenyl)ethyl]-2-methylpropane-2-sulfinamide} monohydrate

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1. Comment

We report the synthesis, isolation, and characterization of the protected 1,2-aminoalcohol (*R*)-*N*-((*R*)-2-(benzyloxy)-1-(4-(*tert*-butyl)phenyl)ethyl)-2-methylpropane-2-sulfinamide (Fig. 1) from the addition of 4-*tert*-butylphenylmagnesium bromide to an *N*-*tert*-butanesulfinyl imine (Fig. 2) according to the procedure of Ellman (Tang *et al.*, 2001). 1,2-Amino-alcohols are found in a variety of pharmaceutically active compounds and are an important component of the chiral pool (Bergmeier, 2000). The method of Ellman and Tang is one of the most direct to monosubstituted aminoalcohols and relies upon the chiral ammonia equivalent, 2-methyl-2-propanesulfinamide (*tert*-butanesulfinamide). In the original report, the absolute configuration of the products was determined by comparison of optical rotations and no structures of the products of these reactions have been reported in the database. This structure is consistent with the sense of induction reported by Ellman and Tang (Tang *et al.*, 2001). There are 21 structures of *N*-sulfinyl-protected 1,2-aminoalcohols in the Cambridge Stuctural Database, but only two of these structures have no substitution at the 1 position and have substitution at the 2 position (Allen, 2002, refcodes FOKDUF, YEQBOM). Neither of these two structures was made by the method we used and neither has an aryl group at the 2 position.

2. 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., dried over molecular sieves, and stored in a desiccator until use. ^1H and ^{13}C NMR spectra were recorded on an Avance 500 MHz s pectrometer with residual protiated solvent as a reference.

To an oven dried 50 ml Schlenk flask equipped with a magnetic stir bar and a rubber septum, 0.685 g (3.9 mmol) of (*R*)-*N*-(2-(benzyloxy)ethylidene)-2-methylpropane-2-sulfinamide, prepared from 2-butene-1,4-diol (Evans *et al.*, 2008, and Tang *et al.*, 2001), and 20 ml of toluene were added, and the mixture was cooled to 195 K under nitrogen (Fig. 2). Also at 195 K and under positive nitrogen pressure, Grignard reagent 4-*tert*-butylmagnesium bromide (1.5 equivalents, 5.85 mmol), prepared from 4-*tert*-butylbromobenzene (Tilstam and Weinmann, 2002), was added dropwise *via* cannula. The reaction was stirred at 195 K until complete consumption of the imine was confirmed by TLC (25% ethyl acetate in hexanes). After quenching with aqueous saturated sodium sulfate, the mixture was warmed to room temperature, dried over sodium sulfate, and filtered through Celite. By ^1H NMR, the crude product was a 3.6:1 ratio of diastereomers, favoring the title compound. Solvent was removed under reduced pressure and the resultant viscous yellow oil was purified by column chromatography (25% ethyl acetate in hexanes) to yield a white solid (266 mg, 18% yield). Initially single crystals of the unsolvated material were grown from a saturated pentane solution at 243 K. NMR analysis of these

crystals showed only the title compound and none of the minor diastereomer, (*R*)-*N*-((*S*)-2-(benzyloxy)-1-(4-(*tert*- butyl)-phenyl)ethyl)-2-methylpropane-2-sulfinamide. However, the structure suffered from severe disorder. High quality single crystals of the title material were obtained from slow evaporation of a methanol solution at room temperature.

The following characterizations were performed on the unsolvated material: Mp 334–336 K. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37–7.26 (m, 9H), 4.69–4.73 (m, 1H), 4.65 (d, J = 11 Hz, 1H), 4.54 (d, J = 12 Hz, 1H), 4.18 (s, 1H), 3.67–3.57 (m, 2H), 1.31 (s, 9H), 1.23 (s, 9H) p.p.m.. ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 150.90, 137.70, 135.37, 128.47, 128.36, 127.83, 127.54, 125.39, 74.19, 72.67, 56.89, 55.49, 34.55, 31.35, 22.64 p.p.m.. IR (neat): 3306, 1061 cm^{-1}.

Crystal data for the highly disordered unsolvated compound: Orthorhombic, $P2_12_12_1$; Cell constants (Å, °): a = 5.6753 (7), b = 17.305 (2), c = 22.188 (3); V = 2179.2 (5) Å^3; Z = 4; T = 100.0 (5) K; 12121 reflections (9622 for [$I > 2\sigma(I)$]).

3. Refinement

All H atoms were located from difference Fourier maps and freely refined.

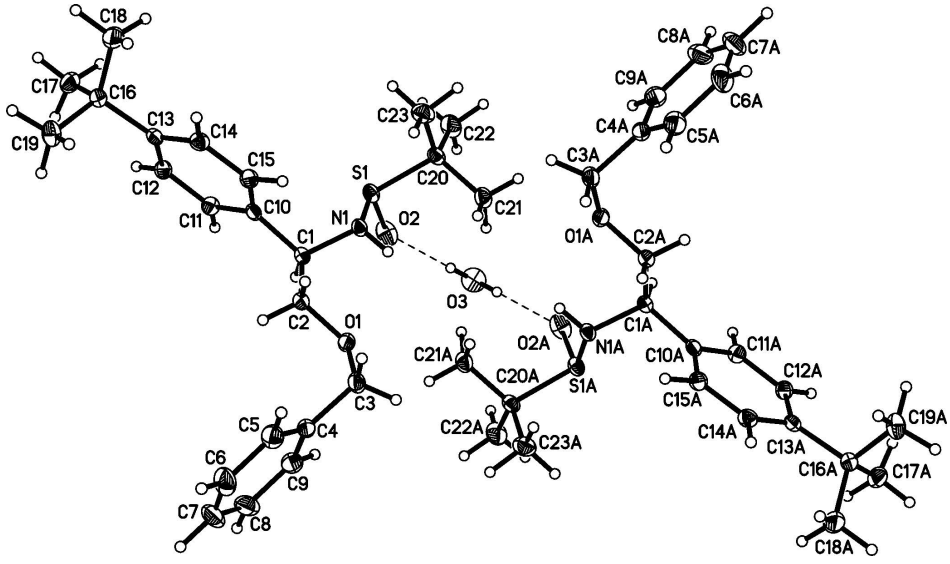

Figure 1

Molecular structure of the title material, showing the atom numbering. The two organic molecules are related by a crystallographic twofold axis (1 - x, y, -z) that includes the water molecule. Intermolecular O–H···O hydrogen bonding is represented with dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

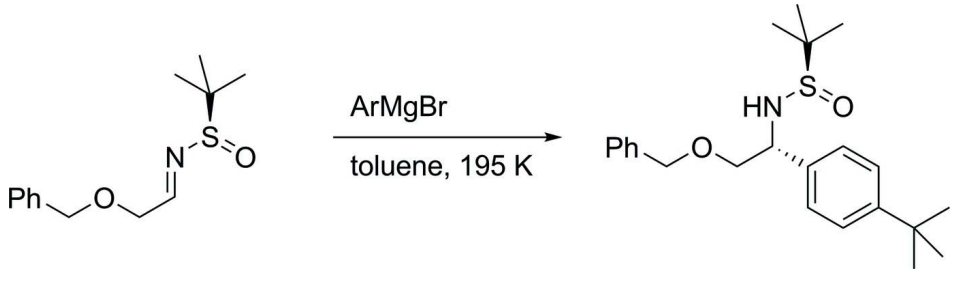

Figure 2

Reaction scheme.

Bis{(*R*)-*N*-[(*R*)-2-benzyloxy-1-(4-*tert*-butylphenyl)ethyl]-2-methylpropane-2-sulfinamide} monohydrate

Crystal data

$2C_{23}H_{33}NO_2S·H_2O$
M_r = 793.14
Monoclinic, *C*2
a = 21.5717 (17) Å
b = 6.1097 (5) Å
c = 17.0838 (14) Å
β = 93.2220 (17)°
V = 2248.0 (3) Å^3
Z = 2
F(000) = 860
D_x = 1.172 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4053 reflections
θ = 2.2–37.9°
μ = 0.16 mm^{-1}
T = 100 K
Needle, colorless
0.40 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEXII CCD platform diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2012)
T_{min} = 0.662, T_{max} = 0.748
34281 measured reflections
12280 independent reflections
9359 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 38.7°, θ_{min} = 1.9°
h = −37→37
k = −10→10
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.110
S = 1.01
12280 reflections
385 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack parameter determined using 3436 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
Absolute structure parameter: −0.01 (3)

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. All hydrogen atoms were found from the difference Fourier map and refined freely.
The absolute configuration was deterimined using 3436 quotients, which gave a Flack parameter of -0.01 (3) (Parsons and Flack, 2004, Parson *et al.*, 2013). This is essentially the same value obtained without D_{obs}(**h**) as a restraint, which resulted in a Flack parameter of -0.01 (4), calculated from 5617 Friedel pairs (Flack, 1983).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.42226 (2)	0.46312 (7)	0.15482 (2)	0.01727 (8)
O1	0.58554 (6)	0.8370 (2)	0.16151 (7)	0.0209 (2)
O2	0.46240 (7)	0.2778 (2)	0.13077 (8)	0.0285 (3)
N1	0.46287 (6)	0.6943 (2)	0.16175 (8)	0.0163 (2)

H1N	0.4839 (11)	0.719 (4)	0.1199 (14)	0.030 (6)*
C1	0.50207 (7)	0.7109 (3)	0.23545 (9)	0.0161 (3)
H1	0.5262 (10)	0.574 (4)	0.2460 (12)	0.016 (5)*
C2	0.54948 (7)	0.8923 (3)	0.22575 (9)	0.0178 (3)
H2A	0.5281 (10)	1.029 (4)	0.2189 (14)	0.023 (6)*
H2B	0.5749 (9)	0.904 (3)	0.2734 (12)	0.012 (5)*
C3	0.62305 (8)	1.0173 (3)	0.13942 (10)	0.0227 (3)
H3A	0.5959 (11)	1.147 (4)	0.1336 (13)	0.022 (6)*
H3B	0.6351 (9)	0.979 (4)	0.0868 (13)	0.022 (5)*
C4	0.67655 (8)	1.0589 (3)	0.19842 (9)	0.0197 (3)
C5	0.68504 (9)	1.2612 (3)	0.23440 (11)	0.0255 (4)
H5	0.6574 (12)	1.379 (5)	0.2226 (15)	0.037 (7)*
C6	0.73526 (11)	1.2977 (4)	0.28735 (12)	0.0341 (5)
H6	0.7411 (11)	1.431 (5)	0.3100 (15)	0.036 (7)*
C7	0.77729 (10)	1.1315 (4)	0.30506 (13)	0.0349 (5)
H7	0.8120 (12)	1.157 (5)	0.3420 (15)	0.041 (7)*
C8	0.76889 (9)	0.9273 (4)	0.27026 (12)	0.0314 (5)
H8	0.7981 (13)	0.816 (5)	0.2848 (16)	0.046 (8)*
C9	0.71873 (8)	0.8916 (3)	0.21733 (11)	0.0242 (3)
H9	0.7099 (12)	0.759 (5)	0.1917 (14)	0.033 (7)*
C10	0.46395 (7)	0.7656 (3)	0.30471 (8)	0.0154 (3)
C11	0.46392 (7)	0.6297 (3)	0.36989 (10)	0.0177 (3)
H11	0.4859 (10)	0.499 (4)	0.3673 (13)	0.024 (6)*
C12	0.43313 (8)	0.6904 (3)	0.43609 (9)	0.0181 (3)
H12	0.4333 (10)	0.594 (4)	0.4788 (13)	0.025 (6)*
C13	0.40197 (7)	0.8894 (3)	0.44018 (9)	0.0161 (3)
C14	0.40172 (7)	1.0238 (3)	0.37349 (10)	0.0184 (3)
H14	0.3801 (11)	1.162 (4)	0.3734 (13)	0.024 (6)*
C15	0.43132 (7)	0.9623 (3)	0.30689 (9)	0.0185 (3)
H15	0.4295 (10)	1.052 (4)	0.2600 (13)	0.024 (6)*
C16	0.37205 (7)	0.9653 (4)	0.51461 (9)	0.0192 (3)
C17	0.35710 (9)	0.7726 (4)	0.56789 (11)	0.0248 (4)
H17A	0.3367 (10)	0.810 (4)	0.6113 (13)	0.025 (6)*
H17B	0.3945 (12)	0.696 (5)	0.5893 (14)	0.034 (7)*
H17C	0.3302 (12)	0.670 (5)	0.5370 (15)	0.039 (7)*
C18	0.31087 (10)	1.0872 (5)	0.49419 (13)	0.0340 (5)
H18A	0.2904 (12)	1.139 (5)	0.5422 (15)	0.038 (7)*
H18B	0.2843 (13)	1.004 (5)	0.4603 (18)	0.051 (8)*
H18C	0.3177 (11)	1.213 (5)	0.4672 (15)	0.032 (7)*
C19	0.41775 (9)	1.1181 (3)	0.55980 (11)	0.0249 (3)
H19A	0.4012 (10)	1.170 (4)	0.6085 (13)	0.020 (5)*
H19B	0.4268 (10)	1.243 (4)	0.5268 (12)	0.018 (5)*
H19C	0.4586 (9)	1.037 (4)	0.5743 (12)	0.017 (5)*
C20	0.37165 (7)	0.5317 (3)	0.06805 (10)	0.0182 (3)
C21	0.40958 (8)	0.5784 (3)	−0.00242 (10)	0.0215 (3)
H21A	0.3833 (11)	0.591 (4)	−0.0478 (14)	0.026 (6)*
H21B	0.4409 (10)	0.450 (5)	−0.0104 (14)	0.030 (6)*
H21C	0.4312 (11)	0.717 (4)	0.0028 (13)	0.023 (6)*
C22	0.33246 (10)	0.3261 (4)	0.05314 (12)	0.0293 (4)

H22A	0.3027 (14)	0.348 (6)	0.0101 (18)	0.052 (8)*
H22B	0.3099 (12)	0.280 (5)	0.0976 (15)	0.030 (6)*
H22C	0.3551 (13)	0.198 (5)	0.0423 (16)	0.049 (8)*
C23	0.33169 (9)	0.7248 (4)	0.09006 (12)	0.0269 (4)
H23A	0.2998 (13)	0.757 (5)	0.0466 (16)	0.047 (8)*
H23B	0.3557 (11)	0.851 (4)	0.0981 (14)	0.029 (6)*
H23C	0.3068 (11)	0.692 (4)	0.1352 (14)	0.028 (6)*
O3	0.5000	0.0337 (4)	0.0000	0.0323 (4)
H3O	0.4903 (13)	0.116 (5)	0.0355 (16)	0.047 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01922 (16)	0.01591 (15)	0.01623 (15)	−0.00308 (15)	−0.00284 (12)	0.00315 (15)
O1	0.0192 (5)	0.0285 (6)	0.0150 (5)	−0.0090 (5)	0.0021 (4)	−0.0026 (4)
O2	0.0358 (7)	0.0181 (6)	0.0304 (7)	0.0067 (5)	−0.0080 (6)	0.0006 (5)
N1	0.0162 (6)	0.0182 (6)	0.0140 (5)	−0.0052 (5)	−0.0017 (4)	0.0013 (5)
C1	0.0150 (6)	0.0194 (7)	0.0137 (6)	0.0000 (5)	−0.0018 (5)	0.0007 (5)
C2	0.0153 (6)	0.0233 (7)	0.0146 (6)	−0.0031 (6)	−0.0005 (5)	−0.0011 (6)
C3	0.0225 (7)	0.0301 (9)	0.0152 (7)	−0.0099 (6)	−0.0015 (6)	0.0035 (6)
C4	0.0181 (7)	0.0251 (8)	0.0157 (6)	−0.0066 (6)	0.0011 (5)	0.0007 (6)
C5	0.0281 (9)	0.0244 (9)	0.0241 (8)	−0.0056 (7)	0.0014 (7)	−0.0009 (7)
C6	0.0412 (11)	0.0354 (11)	0.0253 (9)	−0.0187 (9)	−0.0020 (8)	−0.0050 (8)
C7	0.0265 (9)	0.0502 (13)	0.0267 (9)	−0.0176 (9)	−0.0092 (7)	0.0087 (9)
C8	0.0203 (7)	0.0418 (13)	0.0316 (9)	−0.0043 (8)	−0.0033 (7)	0.0136 (9)
C9	0.0235 (8)	0.0252 (8)	0.0241 (8)	−0.0047 (7)	0.0024 (6)	0.0027 (7)
C10	0.0139 (6)	0.0184 (7)	0.0134 (6)	−0.0015 (5)	−0.0025 (5)	0.0013 (5)
C11	0.0189 (7)	0.0162 (7)	0.0178 (7)	0.0006 (6)	0.0005 (5)	0.0022 (5)
C12	0.0192 (7)	0.0194 (7)	0.0156 (6)	0.0007 (6)	0.0002 (5)	0.0033 (5)
C13	0.0132 (6)	0.0198 (7)	0.0149 (6)	−0.0014 (5)	−0.0023 (5)	0.0000 (5)
C14	0.0168 (6)	0.0184 (7)	0.0197 (7)	0.0030 (5)	−0.0004 (5)	0.0029 (5)
C15	0.0179 (6)	0.0210 (6)	0.0166 (6)	0.0020 (7)	0.0001 (5)	0.0050 (7)
C16	0.0175 (6)	0.0245 (7)	0.0156 (6)	0.0022 (7)	−0.0007 (5)	−0.0018 (7)
C17	0.0237 (8)	0.0308 (9)	0.0205 (8)	−0.0048 (7)	0.0062 (6)	−0.0014 (7)
C18	0.0269 (9)	0.0531 (15)	0.0220 (9)	0.0182 (9)	0.0004 (7)	−0.0013 (9)
C19	0.0328 (9)	0.0221 (8)	0.0195 (8)	−0.0039 (7)	−0.0018 (7)	−0.0022 (6)
C20	0.0157 (6)	0.0195 (7)	0.0189 (7)	−0.0039 (5)	−0.0030 (5)	0.0023 (5)
C21	0.0233 (7)	0.0233 (8)	0.0174 (7)	−0.0027 (6)	−0.0024 (6)	0.0046 (6)
C22	0.0283 (9)	0.0320 (10)	0.0270 (9)	−0.0142 (8)	−0.0033 (7)	−0.0004 (8)
C23	0.0206 (8)	0.0292 (10)	0.0307 (9)	0.0044 (7)	−0.0012 (7)	0.0029 (8)
O3	0.0374 (11)	0.0194 (9)	0.0399 (12)	0.000	0.0008 (10)	0.000

Geometric parameters (Å, º)

S1—O2	1.4967 (14)	C12—H12	0.94 (2)
S1—N1	1.6628 (14)	C13—C14	1.404 (2)
S1—C20	1.8394 (16)	C13—C16	1.530 (2)
O1—C2	1.421 (2)	C14—C15	1.387 (2)
O1—C3	1.430 (2)	C14—H14	0.96 (2)
N1—C1	1.480 (2)	C15—H15	0.97 (2)

N1—H1N	0.88 (2)	C16—C17	1.533 (3)
C1—C10	1.516 (2)	C16—C19	1.534 (3)
C1—C2	1.523 (2)	C16—C18	1.538 (3)
C1—H1	1.00 (2)	C17—H17A	0.91 (2)
C2—H2A	0.96 (2)	C17—H17B	0.99 (3)
C2—H2B	0.96 (2)	C17—H17C	0.99 (3)
C3—C4	1.511 (2)	C18—H18A	1.00 (3)
C3—H3A	0.99 (2)	C18—H18B	0.94 (3)
C3—H3B	0.98 (2)	C18—H18C	0.91 (3)
C4—C5	1.388 (3)	C19—H19A	0.98 (2)
C4—C9	1.394 (3)	C19—H19B	0.97 (2)
C5—C6	1.390 (3)	C19—H19C	1.03 (2)
C5—H5	0.95 (3)	C20—C21	1.520 (2)
C6—C7	1.383 (4)	C20—C23	1.521 (3)
C6—H6	0.91 (3)	C20—C22	1.527 (3)
C7—C8	1.390 (4)	C21—H21A	0.94 (2)
C7—H7	0.96 (3)	C21—H21B	1.05 (3)
C8—C9	1.388 (3)	C21—H21C	0.97 (2)
C8—H8	0.95 (3)	C22—H22A	0.96 (3)
C9—H9	0.94 (3)	C22—H22B	0.97 (3)
C10—C11	1.389 (2)	C22—H22C	0.94 (3)
C10—C15	1.394 (2)	C23—H23A	1.00 (3)
C11—C12	1.394 (2)	C23—H23B	0.94 (3)
C11—H11	0.93 (2)	C23—H23C	0.99 (2)
C12—C13	1.393 (2)	O3—H3O	0.82 (3)

O2—S1—N1	110.59 (8)	C15—C14—C13	121.74 (16)
O2—S1—C20	106.10 (8)	C15—C14—H14	118.7 (14)
N1—S1—C20	98.65 (7)	C13—C14—H14	119.5 (14)
C2—O1—C3	111.30 (14)	C14—C15—C10	120.77 (15)
C1—N1—S1	113.10 (11)	C14—C15—H15	121.8 (13)
C1—N1—H1N	112.5 (16)	C10—C15—H15	117.4 (13)
S1—N1—H1N	112.5 (17)	C13—C16—C17	111.92 (16)
N1—C1—C10	111.72 (12)	C13—C16—C19	108.26 (13)
N1—C1—C2	108.20 (13)	C17—C16—C19	108.61 (14)
C10—C1—C2	108.77 (13)	C13—C16—C18	110.78 (13)
N1—C1—H1	111.3 (12)	C17—C16—C18	107.46 (16)
C10—C1—H1	110.2 (12)	C19—C16—C18	109.78 (18)
C2—C1—H1	106.4 (12)	C16—C17—H17A	114.7 (15)
O1—C2—C1	108.14 (13)	C16—C17—H17B	113.0 (15)
O1—C2—H2A	113.2 (14)	H17A—C17—H17B	103.8 (19)
C1—C2—H2A	109.1 (13)	C16—C17—H17C	107.9 (16)
O1—C2—H2B	111.2 (11)	H17A—C17—H17C	107 (2)
C1—C2—H2B	108.4 (12)	H17B—C17—H17C	110 (2)
H2A—C2—H2B	106.8 (19)	C16—C18—H18A	112.2 (15)
O1—C3—C4	112.08 (14)	C16—C18—H18B	111.4 (19)
O1—C3—H3A	107.7 (13)	H18A—C18—H18B	113 (2)
C4—C3—H3A	111.0 (14)	C16—C18—H18C	111.1 (16)
O1—C3—H3B	103.9 (14)	H18A—C18—H18C	104 (2)

C4—C3—H3B	114.9 (12)	H18B—C18—H18C	105 (2)
H3A—C3—H3B	106.7 (18)	C16—C19—H19A	111.7 (13)
C5—C4—C9	118.82 (17)	C16—C19—H19B	109.2 (13)
C5—C4—C3	121.48 (17)	H19A—C19—H19B	109.6 (18)
C9—C4—C3	119.70 (17)	C16—C19—H19C	110.3 (12)
C4—C5—C6	120.6 (2)	H19A—C19—H19C	107.4 (18)
C4—C5—H5	121.0 (16)	H19B—C19—H19C	108.5 (17)
C6—C5—H5	118.3 (17)	C21—C20—C23	112.84 (15)
C7—C6—C5	120.1 (2)	C21—C20—C22	109.86 (15)
C7—C6—H6	119.5 (16)	C23—C20—C22	111.29 (15)
C5—C6—H6	120.3 (16)	C21—C20—S1	111.06 (11)
C6—C7—C8	119.82 (19)	C23—C20—S1	107.24 (12)
C6—C7—H7	120.2 (17)	C22—C20—S1	104.18 (12)
C8—C7—H7	120.0 (17)	C20—C21—H21A	110.1 (14)
C9—C8—C7	119.9 (2)	C20—C21—H21B	110.0 (13)
C9—C8—H8	122.6 (18)	H21A—C21—H21B	109 (2)
C7—C8—H8	117.6 (18)	C20—C21—H21C	111.6 (13)
C8—C9—C4	120.71 (19)	H21A—C21—H21C	106 (2)
C8—C9—H9	124.8 (16)	H21B—C21—H21C	110.8 (19)
C4—C9—H9	114.5 (16)	C20—C22—H22A	111 (2)
C11—C10—C15	118.08 (14)	C20—C22—H22B	114.0 (16)
C11—C10—C1	121.25 (15)	H22A—C22—H22B	107 (2)
C15—C10—C1	120.51 (13)	C20—C22—H22C	115.1 (18)
C10—C11—C12	120.89 (15)	H22A—C22—H22C	107 (3)
C10—C11—H11	116.9 (14)	H22B—C22—H22C	102 (2)
C12—C11—H11	122.2 (14)	C20—C23—H23A	110.2 (17)
C13—C12—C11	121.75 (15)	C20—C23—H23B	111.1 (15)
C13—C12—H12	119.3 (14)	H23A—C23—H23B	107 (2)
C11—C12—H12	118.9 (14)	C20—C23—H23C	112.1 (15)
C12—C13—C14	116.72 (14)	H23A—C23—H23C	104 (2)
C12—C13—C16	122.24 (15)	H23B—C23—H23C	112 (2)
C14—C13—C16	120.98 (15)		

O2—S1—N1—C1	−79.01 (13)	C15—C10—C11—C12	−1.2 (2)
C20—S1—N1—C1	170.09 (11)	C1—C10—C11—C12	174.35 (15)
S1—N1—C1—C10	−75.83 (16)	C10—C11—C12—C13	−0.8 (3)
S1—N1—C1—C2	164.47 (11)	C11—C12—C13—C14	1.6 (2)
C3—O1—C2—C1	169.06 (13)	C11—C12—C13—C16	−175.44 (15)
N1—C1—C2—O1	−59.44 (17)	C12—C13—C14—C15	−0.4 (2)
C10—C1—C2—O1	179.02 (13)	C16—C13—C14—C15	176.72 (15)
C2—O1—C3—C4	72.42 (18)	C13—C14—C15—C10	−1.7 (3)
O1—C3—C4—C5	−123.42 (18)	C11—C10—C15—C14	2.4 (2)
O1—C3—C4—C9	56.8 (2)	C1—C10—C15—C14	−173.17 (15)
C9—C4—C5—C6	1.1 (3)	C12—C13—C16—C17	−23.2 (2)
C3—C4—C5—C6	−178.68 (17)	C14—C13—C16—C17	159.92 (15)
C4—C5—C6—C7	−0.3 (3)	C12—C13—C16—C19	96.5 (2)
C5—C6—C7—C8	−0.5 (3)	C14—C13—C16—C19	−80.41 (19)
C6—C7—C8—C9	0.6 (3)	C12—C13—C16—C18	−143.07 (19)
C7—C8—C9—C4	0.1 (3)	C14—C13—C16—C18	40.0 (2)

C5—C4—C9—C8	−1.0 (3)	O2—S1—C20—C21	−54.50 (14)
C3—C4—C9—C8	178.79 (16)	N1—S1—C20—C21	59.96 (13)
N1—C1—C10—C11	122.18 (16)	O2—S1—C20—C23	−178.21 (12)
C2—C1—C10—C11	−118.45 (17)	N1—S1—C20—C23	−63.75 (13)
N1—C1—C10—C15	−62.37 (19)	O2—S1—C20—C22	63.72 (14)
C2—C1—C10—C15	56.99 (18)	N1—S1—C20—C22	178.18 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*O*···O2	0.82 (3)	2.02 (3)	2.8420 (19)	171 (3)
C2—H2*A*···O2^{i}	0.96 (2)	2.52 (2)	3.372 (2)	148.1 (19)
C21—H21*C*···O3^{i}	0.97 (2)	2.44 (2)	3.397 (3)	169.0 (19)

Symmetry code: (i) *x*, *y*+1, *z*.