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# (*R*)-*N*-{2-*tert*-Butyl-2-[(*R*)-*tert*-butylsulfonamido]ethylidene}-*tert*-butanesulfonamide

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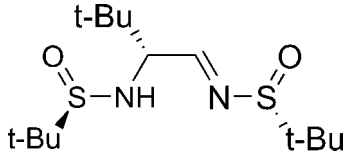
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.039; $wR$ factor = 0.084; data-to-parameter ratio = 17.7.

The title compound, $C_{14}H_{30}N_2O_2S_2$, is the product of the monoaddition reaction of *tert*-butyl magnesium chloride with bis-[(*R*)-*N*-*tert*-butanesulfinyl]ethanediimine. There are two almost identical molecules in the asymmetric unit, the molecular conformation of which is stabilized by an intramolecular N—H···N hydrogen bond.

## Related literature

For general background, see: Sun *et al.* (2005). Alexakis *et al.* (2000); Alvaro *et al.* (1997). For related structures, see: Bambridge *et al.* (1994); Lucet *et al.* (1998); Roland & Mangeney (2000); Roland *et al.* (1999).




## Experimental

### Crystal data

$C_{14}H_{30}N_2O_2S_2$
$M_r$ = 322.52
Monoclinic, $P2_1$
$a$ = 9.714 (2) Å
$b$ = 18.489 (3) Å
$c$ = 11.169 (2) Å
$\beta$ = 109.23 (1)°
$V$ = 1894.0 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.28 mm$^{-1}$
$T$ = 291 (2) K
0.52 × 0.42 × 0.38 mm

### Data collection

Siemens P4 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.936, $T_{max}$ = 0.975
(expected range = 0.862–0.897)
7793 measured reflections
6860 independent reflections
5010 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.020
3 standard reflections
every 97 reflections
intensity decay: 3.1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.084
$S$ = 0.93
6860 reflections
388 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$
Absolute structure: Flack (1983), 3214 Friedel pairs
Flack parameter: −0.04 (5)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*···N2 | 0.84 (2) | 2.19 (2) | 2.697 (3) | 118.5 (17) |
| N1′—H1′*N*···N2′ | 0.84 (2) | 2.18 (3) | 2.672 (3) | 118 (2) |

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2777).

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# supporting information

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## (*R*)-*N*-{2-*tert*-Butyl-2-[(*R*)-*tert*-butylsulfonamido]ethylidene}-*tert*-butanesulfonamide

**Yu Hu, Xiao-Xia Sun and Cong-Bin Fan**

### S1. Comment

The title compound is an important imtermediate for the synthesis of unsymmetrically disubstituted 1,2-diamines and $C_2$-symmetric vicinal diamines. There are two almost identical molecules in the asymmetric unit whose molecular conformation is stabilized by an intramolecular N—H···N hydrogen bond.

### S2. Experimental

R-1-Amino-1-tert-butyl-N,N′-bis[(R)- N-tert-butanesulfinyl]-2-iminoethane was prepared from bis-[(R)-N-tert-butanesulfinyl]ethanediimine (264 mg, 1.00 mmol). The solution of bis-[(R)-N-tert-butanesulfinyl]ethanediimine was cooled to 195 K under a argon atmosphere. 3 mol/l t-BuMgCl in diethyl ether (0.5 ml) was added slowly to the solution and stirred for 3–5 h. The combined organic layers were dried over magnesium sulfate, filtered and concentrated.

Single crystals suitable for X-ray diffraction analysis were obtained by slow diffusion of diethyl ether into the solution. $^1$HNMR (300 MHz, $CDCl_3$, TMS): $\delta$ 1.03 (s, 9H, –$3CH_3$), 1.22 (s, 9H, –$3CH_3$), 1.26 (s, 9H, –$3CH_3$), 3.98 (m, 1H, –NH), 4.84 (d, 1H, $J$ = 5.6 Hz, –CH), 8.28 (d, 1H, $J$ = 2.9 Hz, –CH); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 22.55, 22.78, 26.64, 36.58, 56.41, 57.12, 66.70, 168.39; FT–IR (KBr, cm-1): 1065, 1070, 1621, 3278.

### S3. Refinement

Hydrogen atoms bonded to C were positioned geometrically and refined using a riding model with fixed individual displacement parameters [U(H) = 1.2$U_{eq}$(C) or U(H) = 1.5 $U_{eq}$($C_{methyl}$)] using a riding model with $C_{sp2}$—H = 0.95 Å, tertiary C—H = 0.98 Å, or methyl C—H = 0.96 Å, respectively. The methyl groups were allowed to rotate but not to tip. The H atoms bonded to N were refined isotropically with a distance restraint of 0.84 (1) Å.

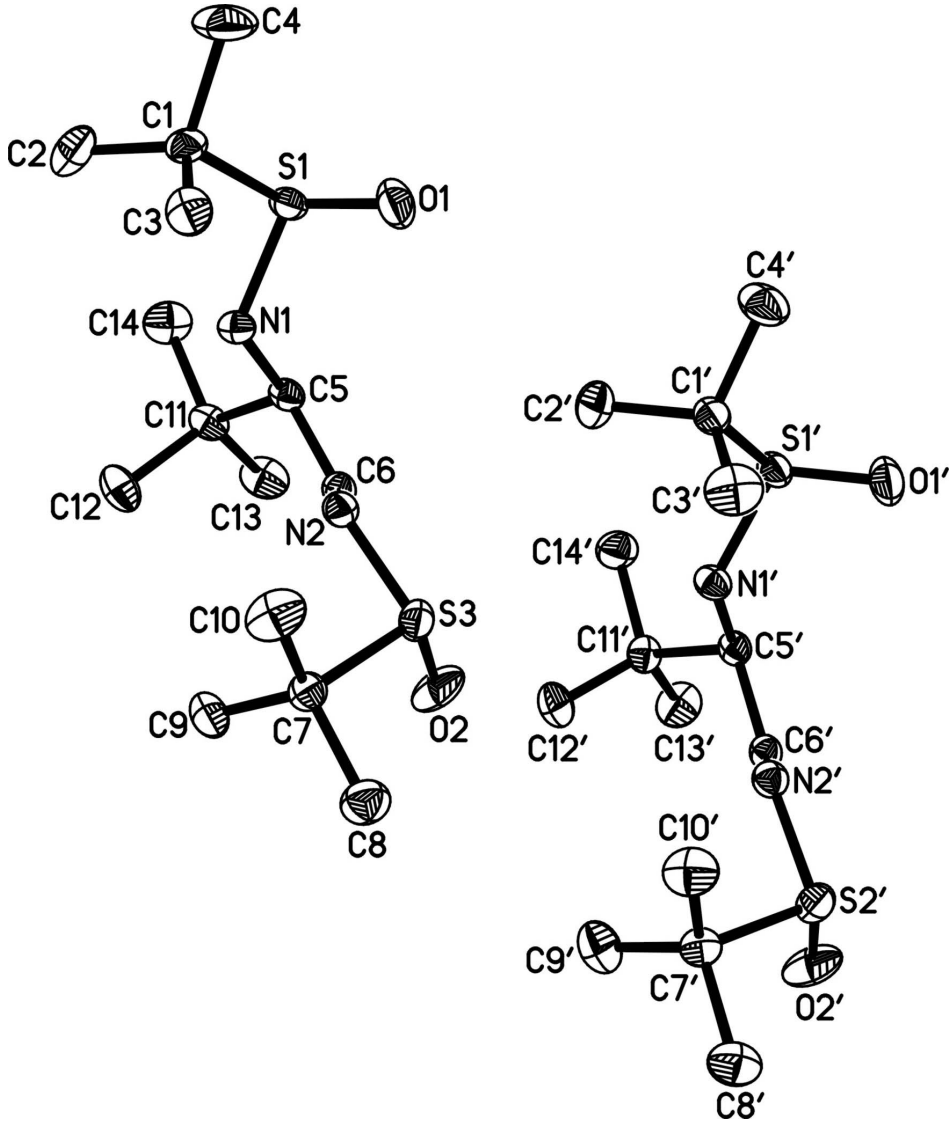


**Figure 1**

Molecular structure of title compound in the solid state with 50% probability ellipsoids showing the labelling scheme. H atoms omitted for clarity.

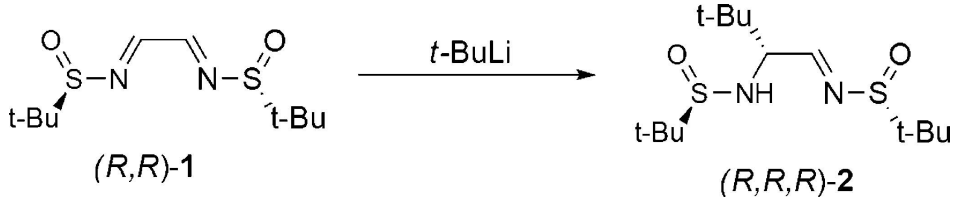


**Figure 2**

Synthesis of the title compound.

**(*R*)-*N*-{2-*tert*-Butyl-2-[(*R*)-*tert*- butylsulfonamido]ethylidene}-*tert*-butanesulfonamide**

*Crystal data*

$C_{14}H_{30}N_2O_2S_2$
$M_r = 322.52$
Monoclinic, $P2_1$
$a = 9.714$ (2) Å
$b = 18.489$ (3) Å
$c = 11.169$ (2) Å
$\beta = 109.23$ (1)°
$V = 1894.0$ (7) Å$^3$
$Z = 4$
$F(000) = 704$
$D_x = 1.131$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 33 reflections
$\theta = 4.4$–14.3°
$\mu = 0.29$ mm$^{-1}$
$T = 291$ K
Block, colourless
0.52 × 0.42 × 0.38 mm

*Data collection*

Siemens P4 diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.936$, $T_{max} = 0.975$
7793 measured reflections
6860 independent reflections
5010 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.9°$
$h = -11 \rightarrow 11$
$k = -21 \rightarrow 22$
$l = -13 \rightarrow 12$
3 standard reflections every 97 reflections
intensity decay: 3.1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.084$
$S = 0.93$
6860 reflections
388 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0019 (5)
Absolute structure: Flack (1983), 3214 Friedel pairs
Absolute structure parameter: −0.04 (5)

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.62185 (9) | 0.13531 (4) | 0.09753 (7) | 0.0524 (2) |

| | | | | |
|---|---|---|---|---|
| S3 | 0.76902 (9) | 0.41227 (5) | 0.24900 (7) | 0.0597 (2) |
| O1 | 0.7320 (3) | 0.13867 (14) | 0.22466 (19) | 0.0841 (7) |
| O2 | 0.6577 (3) | 0.45966 (12) | 0.2718 (2) | 0.0877 (8) |
| N1 | 0.5765 (3) | 0.21702 (12) | 0.0354 (2) | 0.0438 (6) |
| N2 | 0.6898 (3) | 0.33949 (12) | 0.1602 (2) | 0.0483 (6) |
| C1 | 0.7187 (4) | 0.10552 (16) | −0.0102 (3) | 0.0573 (8) |
| C2 | 0.6127 (4) | 0.1053 (2) | −0.1445 (3) | 0.0836 (11) |
| H2A | 0.6594 | 0.0853 | −0.2005 | 0.100* |
| H2B | 0.5824 | 0.1540 | −0.1699 | 0.100* |
| H2C | 0.5291 | 0.0766 | −0.1482 | 0.100* |
| C3 | 0.8487 (3) | 0.15443 (18) | 0.0034 (3) | 0.0688 (9) |
| H3A | 0.9114 | 0.1322 | −0.0368 | 0.083* |
| H3B | 0.9017 | 0.1618 | 0.0917 | 0.083* |
| H3C | 0.8152 | 0.2002 | −0.0362 | 0.083* |
| C4 | 0.7696 (5) | 0.02897 (19) | 0.0361 (5) | 0.1111 (17) |
| H4A | 0.8332 | 0.0310 | 0.1225 | 0.133* |
| H4B | 0.8210 | 0.0085 | −0.0159 | 0.133* |
| H4C | 0.6865 | −0.0005 | 0.0309 | 0.133* |
| C5 | 0.4821 (3) | 0.25941 (14) | 0.0876 (3) | 0.0442 (7) |
| H5 | 0.4620 | 0.2294 | 0.1522 | 0.053* |
| C6 | 0.5596 (3) | 0.32531 (15) | 0.1532 (2) | 0.0490 (7) |
| H6 | 0.5110 | 0.3570 | 0.1901 | 0.059* |
| C7 | 0.8329 (3) | 0.45638 (15) | 0.1300 (3) | 0.0511 (7) |
| C8 | 0.9063 (4) | 0.52479 (18) | 0.1961 (3) | 0.0762 (10) |
| H8A | 0.9412 | 0.5524 | 0.1392 | 0.091* |
| H8B | 0.9868 | 0.5121 | 0.2699 | 0.091* |
| H8C | 0.8374 | 0.5531 | 0.2209 | 0.091* |
| C9 | 0.7062 (4) | 0.47343 (19) | 0.0140 (3) | 0.0759 (10) |
| H9A | 0.6643 | 0.4292 | −0.0272 | 0.091* |
| H9B | 0.7390 | 0.5021 | −0.0431 | 0.091* |
| H9C | 0.6342 | 0.4999 | 0.0378 | 0.091* |
| C10 | 0.9417 (4) | 0.4065 (2) | 0.1017 (4) | 0.0860 (11) |
| H10A | 0.9910 | 0.4321 | 0.0528 | 0.103* |
| H10B | 0.8916 | 0.3655 | 0.0544 | 0.103* |
| H10C | 1.0116 | 0.3904 | 0.1798 | 0.103* |
| C11 | 0.3333 (3) | 0.27616 (16) | −0.0148 (3) | 0.0535 (8) |
| C12 | 0.3570 (3) | 0.3235 (2) | −0.1180 (3) | 0.0729 (10) |
| H12A | 0.4012 | 0.3684 | −0.0815 | 0.087* |
| H12B | 0.2650 | 0.3331 | −0.1820 | 0.087* |
| H12C | 0.4199 | 0.2990 | −0.1553 | 0.087* |
| C13 | 0.2335 (4) | 0.3140 (2) | 0.0462 (3) | 0.0789 (10) |
| H13A | 0.2351 | 0.2882 | 0.1213 | 0.095* |
| H13B | 0.1358 | 0.3148 | −0.0126 | 0.095* |
| H13C | 0.2669 | 0.3626 | 0.0682 | 0.095* |
| C14 | 0.2615 (3) | 0.20401 (18) | −0.0705 (4) | 0.0776 (10) |
| H14A | 0.1650 | 0.2130 | −0.1280 | 0.093* |
| H14B | 0.2561 | 0.1731 | −0.0031 | 0.093* |
| H14C | 0.3186 | 0.1809 | −0.1152 | 0.093* |

| | | | | |
|---|---|---|---|---|
| S1′ | 0.65069 (9) | 0.31033 (4) | 0.68082 (7) | 0.0563 (2) |
| S2′ | 0.77418 (9) | 0.60677 (4) | 0.72095 (7) | 0.0623 (2) |
| O1′ | 0.7423 (3) | 0.31879 (14) | 0.81314 (18) | 0.0963 (9) |
| O2′ | 0.6581 (3) | 0.65720 (13) | 0.7277 (3) | 0.0954 (9) |
| N1′ | 0.6110 (3) | 0.38924 (13) | 0.6060 (2) | 0.0512 (6) |
| N2′ | 0.7029 (3) | 0.52435 (12) | 0.6713 (2) | 0.0524 (6) |
| C1′ | 0.7708 (3) | 0.27604 (16) | 0.5946 (3) | 0.0561 (8) |
| C2′ | 0.6810 (4) | 0.2677 (2) | 0.4578 (3) | 0.0813 (11) |
| H2′1 | 0.7416 | 0.2508 | 0.4110 | 0.098* |
| H2′2 | 0.6393 | 0.3135 | 0.4246 | 0.098* |
| H2′3 | 0.6044 | 0.2334 | 0.4502 | 0.098* |
| C3′ | 0.8974 (4) | 0.3270 (2) | 0.6125 (4) | 0.0946 (12) |
| H3′1 | 0.9740 | 0.3023 | 0.5924 | 0.113* |
| H3′2 | 0.9329 | 0.3430 | 0.6991 | 0.113* |
| H3′3 | 0.8659 | 0.3680 | 0.5576 | 0.113* |
| C4′ | 0.8225 (5) | 0.20221 (19) | 0.6538 (4) | 0.0909 (13) |
| H4′1 | 0.8828 | 0.2085 | 0.7406 | 0.109* |
| H4′2 | 0.8777 | 0.1788 | 0.6078 | 0.109* |
| H4′3 | 0.7396 | 0.1730 | 0.6503 | 0.109* |
| C5′ | 0.5098 (3) | 0.43696 (14) | 0.6406 (3) | 0.0470 (7) |
| H5′ | 0.4925 | 0.4164 | 0.7153 | 0.056* |
| C6′ | 0.5765 (3) | 0.51004 (15) | 0.6755 (2) | 0.0501 (7) |
| H6′ | 0.5246 | 0.5458 | 0.7008 | 0.060* |
| C7′ | 0.8184 (3) | 0.63050 (17) | 0.5785 (3) | 0.0563 (8) |
| C8′ | 0.8951 (4) | 0.70297 (18) | 0.6136 (4) | 0.0860 (11) |
| H8′1 | 0.9151 | 0.7225 | 0.5414 | 0.103* |
| H8′2 | 0.9850 | 0.6964 | 0.6820 | 0.103* |
| H8′3 | 0.8336 | 0.7357 | 0.6395 | 0.103* |
| C9′ | 0.6820 (4) | 0.6364 (2) | 0.4637 (3) | 0.0875 (11) |
| H9′1 | 0.6433 | 0.5889 | 0.4383 | 0.105* |
| H9′2 | 0.7054 | 0.6589 | 0.3954 | 0.105* |
| H9′3 | 0.6107 | 0.6650 | 0.4847 | 0.105* |
| C10′ | 0.9242 (4) | 0.57470 (19) | 0.5606 (4) | 0.0842 (11) |
| H10D | 1.0049 | 0.5700 | 0.6378 | 0.101* |
| H10E | 0.9591 | 0.5898 | 0.4936 | 0.101* |
| H10F | 0.8755 | 0.5290 | 0.5392 | 0.101* |
| C11′ | 0.3593 (3) | 0.44309 (16) | 0.5317 (3) | 0.0534 (8) |
| C12′ | 0.3752 (4) | 0.4788 (2) | 0.4167 (3) | 0.0739 (10) |
| H12D | 0.3947 | 0.5294 | 0.4333 | 0.089* |
| H12E | 0.2866 | 0.4730 | 0.3466 | 0.089* |
| H12F | 0.4544 | 0.4570 | 0.3965 | 0.089* |
| C13′ | 0.2532 (4) | 0.4833 (2) | 0.5827 (4) | 0.0854 (11) |
| H13D | 0.1572 | 0.4816 | 0.5211 | 0.102* |
| H13E | 0.2835 | 0.5328 | 0.5990 | 0.102* |
| H13F | 0.2522 | 0.4609 | 0.6599 | 0.102* |
| C14′ | 0.3006 (4) | 0.36648 (17) | 0.4980 (3) | 0.0754 (10) |
| H14D | 0.2041 | 0.3687 | 0.4373 | 0.090* |
| H14E | 0.2976 | 0.3423 | 0.5732 | 0.090* |

| | | | | |
|---|---|---|---|---|
| H14F | 0.3633 | 0.3403 | 0.4625 | 0.090* |
| H1N | 0.6536 (17) | 0.2404 (12) | 0.046 (2) | 0.043 (8)* |
| H1′N | 0.686 (2) | 0.4126 (14) | 0.607 (3) | 0.059 (10)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0687 (5) | 0.0399 (4) | 0.0566 (5) | 0.0111 (4) | 0.0316 (4) | 0.0094 (3) |
| S3 | 0.0751 (6) | 0.0619 (5) | 0.0430 (4) | −0.0116 (5) | 0.0207 (4) | −0.0050 (4) |
| O1 | 0.1006 (17) | 0.0986 (17) | 0.0521 (12) | 0.0434 (15) | 0.0237 (13) | 0.0181 (13) |
| O2 | 0.1073 (19) | 0.0810 (17) | 0.1057 (19) | −0.0254 (14) | 0.0769 (17) | −0.0468 (14) |
| N1 | 0.0430 (15) | 0.0394 (13) | 0.0545 (15) | −0.0005 (12) | 0.0234 (12) | 0.0015 (11) |
| N2 | 0.0558 (16) | 0.0456 (14) | 0.0470 (14) | 0.0005 (12) | 0.0218 (12) | 0.0029 (10) |
| C1 | 0.074 (2) | 0.0438 (16) | 0.0651 (19) | 0.0105 (17) | 0.0382 (17) | −0.0022 (16) |
| C2 | 0.097 (3) | 0.088 (3) | 0.076 (2) | −0.018 (2) | 0.043 (2) | −0.035 (2) |
| C3 | 0.060 (2) | 0.081 (3) | 0.071 (2) | 0.0117 (18) | 0.0299 (18) | 0.0029 (18) |
| C4 | 0.161 (4) | 0.051 (2) | 0.157 (4) | 0.037 (3) | 0.101 (4) | 0.014 (2) |
| C5 | 0.0469 (17) | 0.0418 (16) | 0.0514 (17) | 0.0042 (13) | 0.0263 (14) | 0.0074 (13) |
| C6 | 0.062 (2) | 0.0476 (18) | 0.0451 (16) | 0.0084 (15) | 0.0278 (15) | 0.0036 (13) |
| C7 | 0.0535 (18) | 0.0515 (17) | 0.0517 (17) | −0.0040 (15) | 0.0220 (15) | −0.0024 (14) |
| C8 | 0.080 (2) | 0.068 (2) | 0.083 (2) | −0.015 (2) | 0.031 (2) | −0.0081 (19) |
| C9 | 0.095 (3) | 0.074 (2) | 0.053 (2) | −0.004 (2) | 0.016 (2) | 0.0147 (17) |
| C10 | 0.071 (2) | 0.087 (3) | 0.119 (3) | −0.005 (2) | 0.058 (2) | −0.024 (3) |
| C11 | 0.0447 (18) | 0.0539 (18) | 0.0653 (19) | 0.0076 (15) | 0.0225 (16) | 0.0109 (15) |
| C12 | 0.061 (2) | 0.088 (3) | 0.064 (2) | 0.0156 (19) | 0.0134 (17) | 0.0272 (19) |
| C13 | 0.063 (2) | 0.077 (2) | 0.106 (3) | 0.019 (2) | 0.041 (2) | 0.014 (2) |
| C14 | 0.052 (2) | 0.075 (2) | 0.097 (3) | −0.0094 (18) | 0.013 (2) | −0.005 (2) |
| S1′ | 0.0768 (6) | 0.0515 (5) | 0.0470 (4) | 0.0158 (4) | 0.0288 (4) | 0.0094 (4) |
| S2′ | 0.0675 (6) | 0.0626 (5) | 0.0584 (5) | −0.0102 (5) | 0.0230 (4) | −0.0121 (4) |
| O1′ | 0.139 (2) | 0.0970 (18) | 0.0443 (12) | 0.0514 (18) | 0.0189 (13) | 0.0050 (13) |
| O2′ | 0.1054 (19) | 0.0705 (17) | 0.141 (2) | −0.0142 (15) | 0.0817 (18) | −0.0438 (15) |
| N1′ | 0.0530 (17) | 0.0495 (15) | 0.0553 (15) | 0.0082 (14) | 0.0238 (13) | 0.0069 (11) |
| N2′ | 0.0523 (16) | 0.0532 (15) | 0.0516 (15) | 0.0041 (13) | 0.0168 (13) | 0.0018 (12) |
| C1′ | 0.068 (2) | 0.0542 (19) | 0.0505 (18) | 0.0160 (17) | 0.0261 (16) | 0.0047 (15) |
| C2′ | 0.108 (3) | 0.087 (3) | 0.055 (2) | 0.007 (2) | 0.034 (2) | −0.0077 (19) |
| C3′ | 0.068 (2) | 0.095 (3) | 0.125 (3) | 0.007 (2) | 0.038 (2) | −0.004 (3) |
| C4′ | 0.123 (3) | 0.074 (2) | 0.091 (3) | 0.046 (2) | 0.056 (3) | 0.021 (2) |
| C5′ | 0.0494 (17) | 0.0515 (18) | 0.0452 (16) | 0.0010 (14) | 0.0223 (14) | 0.0036 (13) |
| C6′ | 0.060 (2) | 0.0524 (18) | 0.0418 (16) | 0.0087 (15) | 0.0217 (15) | 0.0009 (14) |
| C7′ | 0.0511 (17) | 0.0549 (19) | 0.0652 (19) | 0.0032 (16) | 0.0223 (16) | −0.0015 (16) |
| C8′ | 0.081 (3) | 0.072 (3) | 0.107 (3) | −0.005 (2) | 0.033 (2) | 0.008 (2) |
| C9′ | 0.076 (2) | 0.107 (3) | 0.076 (2) | 0.006 (2) | 0.019 (2) | 0.021 (2) |
| C10′ | 0.083 (3) | 0.082 (3) | 0.108 (3) | 0.007 (2) | 0.059 (2) | −0.006 (2) |
| C11′ | 0.0448 (18) | 0.063 (2) | 0.0516 (18) | 0.0070 (16) | 0.0143 (15) | 0.0002 (15) |
| C12′ | 0.066 (2) | 0.088 (3) | 0.058 (2) | 0.007 (2) | 0.0077 (18) | 0.0159 (19) |
| C13′ | 0.062 (2) | 0.095 (3) | 0.099 (3) | 0.021 (2) | 0.025 (2) | −0.013 (2) |
| C14′ | 0.066 (2) | 0.065 (2) | 0.082 (2) | −0.0054 (18) | 0.0073 (19) | −0.0018 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O1 | 1.471 (2) | S1′—O1′ | 1.461 (2) |
| S1—N1 | 1.661 (2) | S1′—N1′ | 1.662 (2) |
| S1—C1 | 1.839 (3) | S1′—C1′ | 1.852 (3) |
| S3—O2 | 1.478 (2) | S2′—O2′ | 1.484 (2) |
| S3—N2 | 1.698 (2) | S2′—N2′ | 1.690 (3) |
| S3—C7 | 1.833 (3) | S2′—C7′ | 1.833 (3) |
| N1—C5 | 1.465 (3) | N1′—C5′ | 1.464 (3) |
| N1—H1N | 0.838 (10) | N1′—H1′N | 0.845 (10) |
| N2—C6 | 1.268 (3) | N2′—C6′ | 1.272 (3) |
| C1—C2 | 1.514 (4) | C1′—C2′ | 1.497 (4) |
| C1—C3 | 1.520 (4) | C1′—C3′ | 1.509 (5) |
| C1—C4 | 1.532 (4) | C1′—C4′ | 1.528 (4) |
| C2—H2A | 0.9600 | C2′—H2′1 | 0.9600 |
| C2—H2B | 0.9600 | C2′—H2′2 | 0.9600 |
| C2—H2C | 0.9600 | C2′—H2′3 | 0.9600 |
| C3—H3A | 0.9600 | C3′—H3′1 | 0.9600 |
| C3—H3B | 0.9600 | C3′—H3′2 | 0.9600 |
| C3—H3C | 0.9600 | C3′—H3′3 | 0.9600 |
| C4—H4A | 0.9600 | C4′—H4′1 | 0.9600 |
| C4—H4B | 0.9600 | C4′—H4′2 | 0.9600 |
| C4—H4C | 0.9600 | C4′—H4′3 | 0.9600 |
| C5—C6 | 1.492 (4) | C5′—C6′ | 1.493 (4) |
| C5—C11 | 1.551 (4) | C5′—C11′ | 1.568 (4) |
| C5—H5 | 0.9800 | C5′—H5′ | 0.9800 |
| C6—H6 | 0.9300 | C6′—H6′ | 0.9300 |
| C7—C9 | 1.497 (4) | C7′—C9′ | 1.514 (4) |
| C7—C10 | 1.513 (4) | C7′—C10′ | 1.515 (4) |
| C7—C8 | 1.519 (4) | C7′—C8′ | 1.520 (4) |
| C8—H8A | 0.9600 | C8′—H8′1 | 0.9600 |
| C8—H8B | 0.9600 | C8′—H8′2 | 0.9600 |
| C8—H8C | 0.9600 | C8′—H8′3 | 0.9600 |
| C9—H9A | 0.9600 | C9′—H9′1 | 0.9600 |
| C9—H9B | 0.9600 | C9′—H9′2 | 0.9600 |
| C9—H9C | 0.9600 | C9′—H9′3 | 0.9600 |
| C10—H10A | 0.9600 | C10′—H10D | 0.9600 |
| C10—H10B | 0.9600 | C10′—H10E | 0.9600 |
| C10—H10C | 0.9600 | C10′—H10F | 0.9600 |
| C11—C12 | 1.524 (4) | C11′—C12′ | 1.497 (4) |
| C11—C13 | 1.525 (4) | C11′—C13′ | 1.525 (4) |
| C11—C14 | 1.539 (4) | C11′—C14′ | 1.527 (4) |
| C12—H12A | 0.9600 | C12′—H12D | 0.9600 |
| C12—H12B | 0.9600 | C12′—H12E | 0.9600 |
| C12—H12C | 0.9600 | C12′—H12F | 0.9600 |
| C13—H13A | 0.9600 | C13′—H13D | 0.9600 |
| C13—H13B | 0.9600 | C13′—H13E | 0.9600 |
| C13—H13C | 0.9600 | C13′—H13F | 0.9600 |

| | | | |
|---|---|---|---|
| C14—H14A | 0.9600 | C14′—H14D | 0.9600 |
| C14—H14B | 0.9600 | C14′—H14E | 0.9600 |
| C14—H14C | 0.9600 | C14′—H14F | 0.9600 |
| | | | |
| O1—S1—N1 | 111.94 (14) | O1′—S1′—N1′ | 112.20 (14) |
| O1—S1—C1 | 106.14 (14) | O1′—S1′—C1′ | 106.52 (15) |
| N1—S1—C1 | 96.97 (13) | N1′—S1′—C1′ | 96.92 (13) |
| O2—S3—N2 | 110.71 (13) | O2′—S2′—N2′ | 110.32 (13) |
| O2—S3—C7 | 107.61 (15) | O2′—S2′—C7′ | 107.03 (15) |
| N2—S3—C7 | 97.23 (12) | N2′—S2′—C7′ | 96.83 (13) |
| C5—N1—S1 | 115.78 (18) | C5′—N1′—S1′ | 117.44 (19) |
| C5—N1—H1N | 108.2 (18) | C5′—N1′—H1′N | 110 (2) |
| S1—N1—H1N | 107.9 (18) | S1′—N1′—H1′N | 113 (2) |
| C6—N2—S3 | 116.8 (2) | C6′—N2′—S2′ | 118.1 (2) |
| C2—C1—C3 | 111.8 (3) | C2′—C1′—C3′ | 112.1 (3) |
| C2—C1—C4 | 111.8 (3) | C2′—C1′—C4′ | 110.3 (3) |
| C3—C1—C4 | 110.6 (3) | C3′—C1′—C4′ | 111.3 (3) |
| C2—C1—S1 | 108.7 (2) | C2′—C1′—S1′ | 108.1 (2) |
| C3—C1—S1 | 110.1 (2) | C3′—C1′—S1′ | 110.1 (2) |
| C4—C1—S1 | 103.5 (2) | C4′—C1′—S1′ | 104.7 (2) |
| C1—C2—H2A | 109.5 | C1′—C2′—H2′1 | 109.5 |
| C1—C2—H2B | 109.5 | C1′—C2′—H2′2 | 109.5 |
| H2A—C2—H2B | 109.5 | H2′1—C2′—H2′2 | 109.5 |
| C1—C2—H2C | 109.5 | C1′—C2′—H2′3 | 109.5 |
| H2A—C2—H2C | 109.5 | H2′1—C2′—H2′3 | 109.5 |
| H2B—C2—H2C | 109.5 | H2′2—C2′—H2′3 | 109.5 |
| C1—C3—H3A | 109.5 | C1′—C3′—H3′1 | 109.5 |
| C1—C3—H3B | 109.5 | C1′—C3′—H3′2 | 109.5 |
| H3A—C3—H3B | 109.5 | H3′1—C3′—H3′2 | 109.5 |
| C1—C3—H3C | 109.5 | C1′—C3′—H3′3 | 109.5 |
| H3A—C3—H3C | 109.5 | H3′1—C3′—H3′3 | 109.5 |
| H3B—C3—H3C | 109.5 | H3′2—C3′—H3′3 | 109.5 |
| C1—C4—H4A | 109.5 | C1′—C4′—H4′1 | 109.5 |
| C1—C4—H4B | 109.5 | C1′—C4′—H4′2 | 109.5 |
| H4A—C4—H4B | 109.5 | H4′1—C4′—H4′2 | 109.5 |
| C1—C4—H4C | 109.5 | C1′—C4′—H4′3 | 109.5 |
| H4A—C4—H4C | 109.5 | H4′1—C4′—H4′3 | 109.5 |
| H4B—C4—H4C | 109.5 | H4′2—C4′—H4′3 | 109.5 |
| N1—C5—C6 | 110.6 (2) | N1′—C5′—C6′ | 110.1 (2) |
| N1—C5—C11 | 111.5 (2) | N1′—C5′—C11′ | 112.0 (2) |
| C6—C5—C11 | 113.3 (2) | C6′—C5′—C11′ | 110.5 (2) |
| N1—C5—H5 | 107.0 | N1′—C5′—H5′ | 108.1 |
| C6—C5—H5 | 107.0 | C6′—C5′—H5′ | 108.1 |
| C11—C5—H5 | 107.0 | C11′—C5′—H5′ | 108.1 |
| N2—C6—C5 | 122.1 (2) | N2′—C6′—C5′ | 121.3 (2) |
| N2—C6—H6 | 119.0 | N2′—C6′—H6′ | 119.4 |
| C5—C6—H6 | 119.0 | C5′—C6′—H6′ | 119.4 |
| C9—C7—C10 | 112.3 (3) | C9′—C7′—C10′ | 112.3 (3) |

| | | | |
|---|---|---|---|
| C9—C7—C8 | 111.5 (3) | C9′—C7′—C8′ | 111.9 (3) |
| C10—C7—C8 | 111.1 (3) | C10′—C7′—C8′ | 109.8 (3) |
| C9—C7—S3 | 110.1 (2) | C9′—C7′—S2′ | 111.2 (2) |
| C10—C7—S3 | 107.9 (2) | C10′—C7′—S2′ | 108.3 (2) |
| C8—C7—S3 | 103.6 (2) | C8′—C7′—S2′ | 102.8 (2) |
| C7—C8—H8A | 109.5 | C7′—C8′—H8′1 | 109.5 |
| C7—C8—H8B | 109.5 | C7′—C8′—H8′2 | 109.5 |
| H8A—C8—H8B | 109.5 | H8′1—C8′—H8′2 | 109.5 |
| C7—C8—H8C | 109.5 | C7′—C8′—H8′3 | 109.5 |
| H8A—C8—H8C | 109.5 | H8′1—C8′—H8′3 | 109.5 |
| H8B—C8—H8C | 109.5 | H8′2—C8′—H8′3 | 109.5 |
| C7—C9—H9A | 109.5 | C7′—C9′—H9′1 | 109.5 |
| C7—C9—H9B | 109.5 | C7′—C9′—H9′2 | 109.5 |
| H9A—C9—H9B | 109.5 | H9′1—C9′—H9′2 | 109.5 |
| C7—C9—H9C | 109.5 | C7′—C9′—H9′3 | 109.5 |
| H9A—C9—H9C | 109.5 | H9′1—C9′—H9′3 | 109.5 |
| H9B—C9—H9C | 109.5 | H9′2—C9′—H9′3 | 109.5 |
| C7—C10—H10A | 109.5 | C7′—C10′—H10D | 109.5 |
| C7—C10—H10B | 109.5 | C7′—C10′—H10E | 109.5 |
| H10A—C10—H10B | 109.5 | H10D—C10′—H10E | 109.5 |
| C7—C10—H10C | 109.5 | C7′—C10′—H10F | 109.5 |
| H10A—C10—H10C | 109.5 | H10D—C10′—H10F | 109.5 |
| H10B—C10—H10C | 109.5 | H10E—C10′—H10F | 109.5 |
| C12—C11—C13 | 110.6 (3) | C12′—C11′—C13′ | 112.2 (3) |
| C12—C11—C14 | 110.7 (3) | C12′—C11′—C14′ | 109.5 (3) |
| C13—C11—C14 | 107.9 (3) | C13′—C11′—C14′ | 107.5 (3) |
| C12—C11—C5 | 109.6 (2) | C12′—C11′—C5′ | 111.4 (3) |
| C13—C11—C5 | 109.7 (2) | C13′—C11′—C5′ | 108.5 (2) |
| C14—C11—C5 | 108.3 (2) | C14′—C11′—C5′ | 107.5 (2) |
| C11—C12—H12A | 109.5 | C11′—C12′—H12D | 109.5 |
| C11—C12—H12B | 109.5 | C11′—C12′—H12E | 109.5 |
| H12A—C12—H12B | 109.5 | H12D—C12′—H12E | 109.5 |
| C11—C12—H12C | 109.5 | C11′—C12′—H12F | 109.5 |
| H12A—C12—H12C | 109.5 | H12D—C12′—H12F | 109.5 |
| H12B—C12—H12C | 109.5 | H12E—C12′—H12F | 109.5 |
| C11—C13—H13A | 109.5 | C11′—C13′—H13D | 109.5 |
| C11—C13—H13B | 109.5 | C11′—C13′—H13E | 109.5 |
| H13A—C13—H13B | 109.5 | H13D—C13′—H13E | 109.5 |
| C11—C13—H13C | 109.5 | C11′—C13′—H13F | 109.5 |
| H13A—C13—H13C | 109.5 | H13D—C13′—H13F | 109.5 |
| H13B—C13—H13C | 109.5 | H13E—C13′—H13F | 109.5 |
| C11—C14—H14A | 109.5 | C11′—C14′—H14D | 109.5 |
| C11—C14—H14B | 109.5 | C11′—C14′—H14E | 109.5 |
| H14A—C14—H14B | 109.5 | H14D—C14′—H14E | 109.5 |
| C11—C14—H14C | 109.5 | C11′—C14′—H14F | 109.5 |
| H14A—C14—H14C | 109.5 | H14D—C14′—H14F | 109.5 |
| H14B—C14—H14C | 109.5 | H14E—C14′—H14F | 109.5 |

| | | | |
|---|---|---|---|
| O1—S1—N1—C5 | −74.9 (2) | O1′—S1′—N1′—C5′ | −71.0 (3) |
| C1—S1—N1—C5 | 174.6 (2) | C1′—S1′—N1′—C5′ | 178.0 (2) |
| O2—S3—N2—C6 | −17.2 (2) | O2′—S2′—N2′—C6′ | −16.3 (3) |
| C7—S3—N2—C6 | −129.1 (2) | C7′—S2′—N2′—C6′ | −127.3 (2) |
| O1—S1—C1—C2 | −176.6 (2) | O1′—S1′—C1′—C2′ | −179.2 (2) |
| N1—S1—C1—C2 | −61.3 (2) | N1′—S1′—C1′—C2′ | −63.5 (2) |
| O1—S1—C1—C3 | −53.7 (2) | O1′—S1′—C1′—C3′ | −56.5 (3) |
| N1—S1—C1—C3 | 61.5 (2) | N1′—S1′—C1′—C3′ | 59.1 (3) |
| O1—S1—C1—C4 | 64.4 (3) | O1′—S1′—C1′—C4′ | 63.2 (3) |
| N1—S1—C1—C4 | 179.7 (3) | N1′—S1′—C1′—C4′ | 178.8 (2) |
| S1—N1—C5—C6 | 115.5 (2) | S1′—N1′—C5′—C6′ | 126.5 (2) |
| S1—N1—C5—C11 | −117.4 (2) | S1′—N1′—C5′—C11′ | −110.2 (2) |
| S3—N2—C6—C5 | −174.66 (18) | S2′—N2′—C6′—C5′ | −176.3 (2) |
| N1—C5—C6—N2 | −0.4 (3) | N1′—C5′—C6′—N2′ | 0.9 (4) |
| C11—C5—C6—N2 | −126.5 (3) | C11′—C5′—C6′—N2′ | −123.3 (3) |
| O2—S3—C7—C9 | −54.5 (3) | O2′—S2′—C7′—C9′ | −49.8 (3) |
| N2—S3—C7—C9 | 59.9 (2) | N2′—S2′—C7′—C9′ | 64.0 (3) |
| O2—S3—C7—C10 | −177.4 (2) | O2′—S2′—C7′—C10′ | −173.7 (2) |
| N2—S3—C7—C10 | −62.9 (2) | N2′—S2′—C7′—C10′ | −59.9 (2) |
| O2—S3—C7—C8 | 64.7 (2) | O2′—S2′—C7′—C8′ | 70.2 (2) |
| N2—S3—C7—C8 | 179.2 (2) | N2′—S2′—C7′—C8′ | −176.1 (2) |
| N1—C5—C11—C12 | −63.8 (3) | N1′—C5′—C11′—C12′ | −65.0 (3) |
| C6—C5—C11—C12 | 61.8 (3) | C6′—C5′—C11′—C12′ | 58.1 (3) |
| N1—C5—C11—C13 | 174.6 (2) | N1′—C5′—C11′—C13′ | 171.0 (3) |
| C6—C5—C11—C13 | −59.8 (3) | C6′—C5′—C11′—C13′ | −65.9 (3) |
| N1—C5—C11—C14 | 57.1 (3) | N1′—C5′—C11′—C14′ | 54.9 (3) |
| C6—C5—C11—C14 | −177.3 (2) | C6′—C5′—C11′—C14′ | 178.0 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···N2 | 0.84 (2) | 2.19 (2) | 2.697 (3) | 119 (2) |
| N1′—H1′*N*···N2′ | 0.84 (2) | 2.18 (3) | 2.672 (3) | 118 (2) |