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# (*R*)-*N*-(Biphenyl-4-yl)-*tert*-butanesulfinamide

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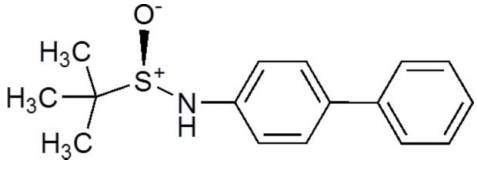
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.047; $wR$ factor = 0.097; data-to-parameter ratio = 15.8.

In the title compound, $C_{16}H_{19}NOS$, the dihedral angle between the two aromatic rings is 38.98 (8)°. The crystal structure is stabilized by N—H···O hydrogen bonds, which link neighbouring molecules into chains running parallel to the *a* axis.

## Related literature

For related structures, see: Sun *et al.* (2012); Jasinski *et al.* (2012); Gainsford *et al.* (2011).



## Experimental

### Crystal data

$C_{16}H_{19}NOS$
$M_r$ = 273.38
Orthorhombic, $P2_12_12_1$
$a$ = 9.3588 (5) Å
$b$ = 11.9452 (5) Å
$c$ = 13.3136 (7) Å
$V$ = 1488.36 (12) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.21 mm$^{-1}$
$T$ = 293 K
0.43 × 0.41 × 0.40 mm

### Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min}$ = 0.988, $T_{max}$ = 1.000
3983 measured reflections
2766 independent reflections
2325 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.097
$S$ = 1.06
2766 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$
Absolute structure: assigned from the known absolute structure of the (*R*)-tert-butanesulfinamide starting material; the Flack (1983) parameter is consistent with this assignment, 1017 Friedel pairs
Flack parameter: 0.03 (10)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.86 | 2.35 | 3.144 (3) | 154 |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2735).

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# supporting information

*Acta Cryst.* (2012). E**68**, o1389 [doi:10.1107/S1600536812015127]

## (*R*)-*N*-(Biphenyl-4-yl)-*tert*-butanesulfinamide

**Binbin Zhang, Yan Wang, Xiaofei Sun, Wenguo Wang and Qingle Zeng**

### S1. Comment

Sulfinamides, especially chiral sulfinamides, are an important class of organic compounds in modern organic chemistry, and a great number of such compounds have been synthesized. In our continuous study on chiral *N*-aryl-*tert*-butanesulfinamides (Sun *et al.*, 2012), we have prepared the title compound and report its crystal structure herein.

In the molecule of the title compound (Fig. 1) the aromatic rings of the biphenyl are tilted to form a dihedral angle of 38.98 (8)°, which is comparable to the value observed in other related compounds containing the biphenyl group (Jasinski *et al.*, 2012; Gainsford *et al.*, 2011). In the crystal packing (Fig. 2), the molecules are linked by intermolecular N—H···O hydrogen bonds (Table 1) into one-dimensional chains running parallel to the the *a* axis.

### S2. Experimental

A oven-dried ground test tube, which was equipped with a magnetic stir bar and fitted with a rubber septum, was charged with (*R*)-*tert*-butanesulfinamide (0.121 g, 1.0 mmol), Pd2(dba)3 (0.018 g, 0.02 mmol; dba is dibenzylideneacetone), 2-di-*tert*-butylphosphino-2′,4′,6′-triisopropylbiphenyl (0.0212 g, 0.05 mmol) and NaOH (0.08 g, 2 mmol). The vessel was evacuated and backfilled with argon three times, then 4-biphenyl bromide (1.3 mmol), toluene (10 ml) and degassed water (0.3 ml) were added *via* syringe. The solution was stirred at 90°C for 20 h. The reaction mixture was then cooled to room temperature, quenched by water, and extracted with chloroform (20 ml) for twice. The organic layers were combined, and dried over anhydrous sodium sulfate and filtrated. The filterate was condensed under vacuum. The residual was purified with silica gel column chromatography with a solution of petroleum ether and ethyl acetate (5:1 *v*/*v*) as eluent to give the title compound (*R*)-*N*-(4-biphenyl)-*tert*-butanesulfinamide. A test tube containing a petroleum ether and ethyl acetate (1:1 *v*/*v*) solution of the title compound was covered with a piece of filter paper and placed motionless at room temperature, and a single-crystal was cultured in the bottom of the test tube. Spectroscopic analysis: $^{1}$H NMR (300 MHz, $CDCl_3$), $\delta$ (ppm): 7.49–7.29 (m, 7H), 7.06 (d, J = 8.5 Hz, 2H), 6.03 (d, J = 3.9 Hz, 1H), 1.37 (s, 9H). $^{13}$C NMR (300 MHz, $CDCl_3$), $\delta$ (ppm): 114.6, 140.4, 135.6, 128.6, 127.9, 126.8, 126.6, 118.4, 56.5, 22.4. FT—IR (KBr) ($cm^{-1}$): 3453, 3252, 2926, 1610, 1519, 1485, 1386, 1305, 1286, 1268, 1228, 1191, 1057, 912, 880, 838, 767. [$\alpha$]D = -110.8 (c 0.15, ethyl acetate). ESI-MS (negative mode), m/*z* = 272 [M—H]-. Anal. Calcd for $C_{16}H_{19}NOS$: C, 70.29; H, 7.00; N, 5.12. Found: C, 70.43; H, 7.16; N 5.01.

### S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with N—H = 0.86 Å, C—H = 0.93–0.96 Å, and with $U_{iso}$(H) = 1.2 $U_{eq}$(C, N) or 1.5 $U_{eq}$(C) for methyl H atoms.

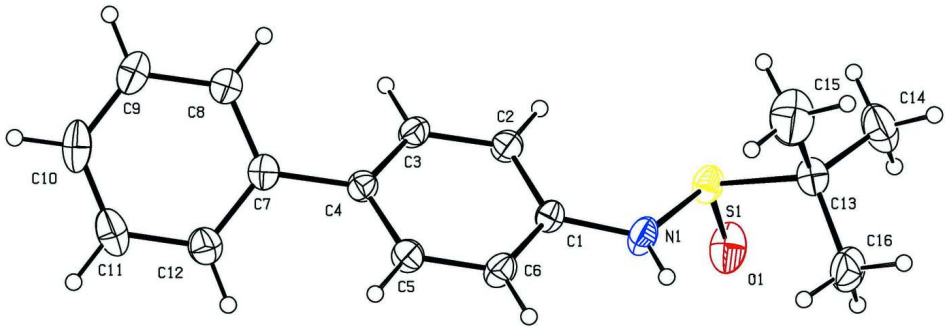


**Figure 1**

Molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

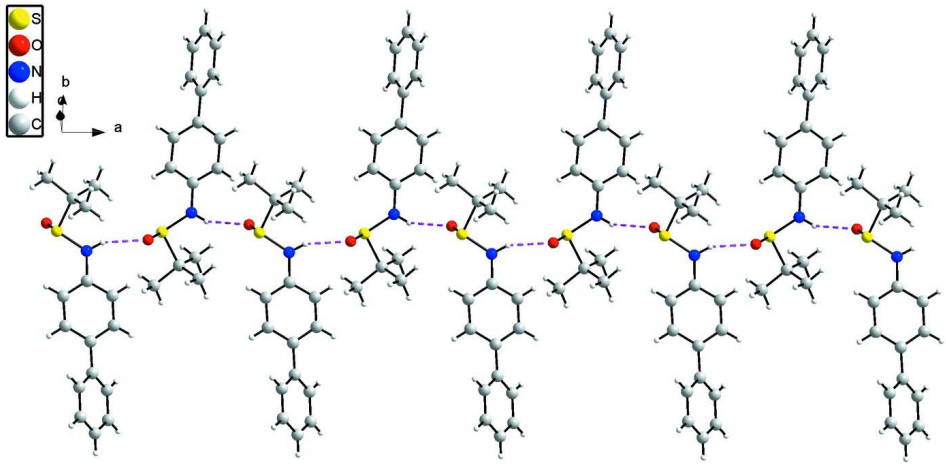


**Figure 2**

The one-dimensional structure of (I) in the crystal packing, showing intermolecular hydrogen bonding as dashed lines.

### (*R*)-*N*-(Biphenyl-4-yl)-*tert*-butanesulfinamide

*Crystal data*

$C_{16}H_{19}NOS$
$M_r = 273.38$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 9.3588\ (5)$ Å
$b = 11.9452\ (5)$ Å
$c = 13.3136\ (7)$ Å
$V = 1488.36\ (12)$ Å$^3$
$Z = 4$
$F(000) = 584$
$D_x = 1.220$ Mg m$^{-3}$
Melting point: 427 K
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 1425 reflections
$\theta = 3.1–28.9°$
$\mu = 0.21$ mm$^{-1}$
$T = 293$ K
Block, colourless
0.43 × 0.41 × 0.40 mm

*Data collection*

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0874 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.988$, $T_{max} = 1.000$
3983 measured reflections
2766 independent reflections
2325 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$

$\theta_{max}$ = 26.4°, $\theta_{min}$ = 3.1°
$h$ = −11→5
$k$ = −7→14
$l$ = −16→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.097
$S$ = 1.06
2766 reflections
175 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 0.0448P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$
Absolute structure: assigned from the known absolute structure of the (*R*)-tert-butanesulfinamide starting material; the Flack (1983) parameter is consistent with this assignment, 1017 Friedel pairs
Absolute structure parameter: 0.03 (10)

*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**. 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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | −0.15256 (8) | −0.31264 (5) | −0.40407 (5) | 0.04256 (19) |
| O1 | −0.2124 (3) | −0.22979 (16) | −0.47627 (16) | 0.0639 (7) |
| N1 | −0.0065 (3) | −0.36901 (17) | −0.45167 (18) | 0.0488 (7) |
| H1 | 0.0702 | −0.3294 | −0.4536 | 0.059* |
| C1 | −0.0021 (3) | −0.4790 (2) | −0.48927 (19) | 0.0355 (6) |
| C2 | −0.1153 (3) | −0.5532 (2) | −0.4810 (2) | 0.0415 (7) |
| H2 | −0.1987 | −0.5318 | −0.4482 | 0.050* |
| C3 | −0.1032 (3) | −0.6600 (2) | −0.5220 (2) | 0.0400 (7) |
| H3 | −0.1801 | −0.7090 | −0.5166 | 0.048* |
| C4 | 0.0197 (3) | −0.6959 (2) | −0.57082 (17) | 0.0357 (6) |
| C5 | 0.1315 (3) | −0.6196 (2) | −0.5777 (2) | 0.0395 (6) |
| H5 | 0.2149 | −0.6404 | −0.6107 | 0.047* |
| C6 | 0.1217 (3) | −0.5131 (2) | −0.5366 (2) | 0.0423 (7) |
| H6 | 0.1990 | −0.4644 | −0.5410 | 0.051* |
| C7 | 0.0310 (3) | −0.8089 (2) | −0.61585 (18) | 0.0377 (6) |
| C8 | −0.0253 (3) | −0.9024 (2) | −0.5693 (2) | 0.0476 (8) |
| H8 | −0.0694 | −0.8944 | −0.5071 | 0.057* |
| C9 | −0.0181 (4) | −1.0075 (2) | −0.6126 (2) | 0.0558 (8) |
| H9 | −0.0581 | −1.0688 | −0.5801 | 0.067* |

| | | | | |
|---|---|---|---|---|
| C10 | 0.0480 (4) | −1.0208 (3) | −0.7031 (3) | 0.0600 (9) |
| H10 | 0.0535 | −1.0914 | −0.7324 | 0.072* |
| C11 | 0.1060 (3) | −0.9301 (3) | −0.7507 (2) | 0.0586 (9) |
| H11 | 0.1514 | −0.9395 | −0.8122 | 0.070* |
| C12 | 0.0980 (3) | −0.8240 (2) | −0.7081 (2) | 0.0471 (7) |
| H12 | 0.1375 | −0.7630 | −0.7414 | 0.057* |
| C13 | −0.0712 (3) | −0.2310 (2) | −0.3024 (2) | 0.0461 (7) |
| C14 | −0.1958 (4) | −0.1746 (3) | −0.2498 (2) | 0.0769 (12) |
| H14A | −0.2385 | −0.1207 | −0.2941 | 0.115* |
| H14B | −0.1622 | −0.1375 | −0.1903 | 0.115* |
| H14C | −0.2656 | −0.2299 | −0.2316 | 0.115* |
| C15 | −0.0002 (6) | −0.3136 (3) | −0.2314 (3) | 0.0911 (14) |
| H15A | −0.0670 | −0.3713 | −0.2138 | 0.137* |
| H15B | 0.0301 | −0.2752 | −0.1718 | 0.137* |
| H15C | 0.0812 | −0.3465 | −0.2639 | 0.137* |
| C16 | 0.0316 (4) | −0.1448 (2) | −0.3427 (3) | 0.0670 (10) |
| H16A | 0.1088 | −0.1818 | −0.3767 | 0.100* |
| H16B | 0.0690 | −0.1013 | −0.2880 | 0.100* |
| H16C | −0.0173 | −0.0965 | −0.3889 | 0.100* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0340 (4) | 0.0355 (3) | 0.0581 (4) | 0.0019 (3) | −0.0013 (4) | −0.0073 (4) |
| O1 | 0.0696 (17) | 0.0521 (12) | 0.0701 (14) | 0.0165 (12) | −0.0276 (13) | −0.0088 (11) |
| N1 | 0.0381 (15) | 0.0330 (11) | 0.0754 (16) | −0.0062 (11) | 0.0102 (13) | −0.0137 (11) |
| C1 | 0.0374 (16) | 0.0289 (13) | 0.0403 (14) | 0.0022 (12) | −0.0009 (13) | −0.0009 (11) |
| C2 | 0.0343 (16) | 0.0364 (14) | 0.0538 (16) | 0.0000 (13) | 0.0130 (14) | −0.0031 (13) |
| C3 | 0.0377 (16) | 0.0319 (14) | 0.0505 (16) | −0.0043 (12) | 0.0086 (14) | −0.0013 (12) |
| C4 | 0.0386 (15) | 0.0326 (12) | 0.0360 (13) | 0.0026 (13) | −0.0008 (12) | 0.0012 (12) |
| C5 | 0.0306 (15) | 0.0389 (13) | 0.0488 (16) | 0.0043 (12) | 0.0058 (14) | −0.0017 (13) |
| C6 | 0.0363 (17) | 0.0353 (14) | 0.0552 (17) | −0.0038 (13) | 0.0004 (14) | −0.0009 (13) |
| C7 | 0.0333 (14) | 0.0373 (13) | 0.0423 (14) | 0.0061 (13) | −0.0057 (12) | −0.0024 (13) |
| C8 | 0.0546 (19) | 0.0393 (14) | 0.0488 (17) | −0.0007 (15) | 0.0038 (16) | −0.0023 (13) |
| C9 | 0.063 (2) | 0.0360 (14) | 0.068 (2) | −0.0014 (16) | −0.0094 (19) | −0.0060 (15) |
| C10 | 0.054 (2) | 0.0503 (18) | 0.075 (2) | 0.0092 (17) | −0.021 (2) | −0.0296 (18) |
| C11 | 0.046 (2) | 0.073 (2) | 0.0567 (19) | 0.0086 (19) | −0.0018 (16) | −0.0280 (18) |
| C12 | 0.0446 (18) | 0.0491 (16) | 0.0477 (16) | 0.0017 (15) | −0.0003 (15) | −0.0044 (15) |
| C13 | 0.0484 (19) | 0.0452 (15) | 0.0447 (16) | 0.0038 (14) | −0.0030 (15) | −0.0074 (14) |
| C14 | 0.075 (3) | 0.085 (3) | 0.070 (2) | 0.006 (2) | 0.014 (2) | −0.030 (2) |
| C15 | 0.125 (4) | 0.076 (2) | 0.073 (2) | 0.018 (3) | −0.036 (3) | −0.002 (2) |
| C16 | 0.065 (2) | 0.0593 (19) | 0.077 (2) | −0.0194 (18) | 0.001 (2) | −0.0224 (18) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O1 | 1.489 (2) | C9—H9 | 0.9300 |
| S1—N1 | 1.650 (2) | C9—C10 | 1.364 (4) |
| S1—C13 | 1.833 (3) | C10—H10 | 0.9300 |

| | | | |
|---|---|---|---|
| N1—H1 | 0.8600 | C10—C11 | 1.367 (4) |
| N1—C1 | 1.407 (3) | C11—H11 | 0.9300 |
| C1—C2 | 1.385 (3) | C11—C12 | 1.391 (4) |
| C1—C6 | 1.381 (4) | C12—H12 | 0.9300 |
| C2—H2 | 0.9300 | C13—C14 | 1.519 (4) |
| C2—C3 | 1.393 (3) | C13—C15 | 1.519 (4) |
| C3—H3 | 0.9300 | C13—C16 | 1.508 (4) |
| C3—C4 | 1.389 (4) | C14—H14A | 0.9600 |
| C4—C5 | 1.390 (4) | C14—H14B | 0.9600 |
| C4—C7 | 1.481 (3) | C14—H14C | 0.9600 |
| C5—H5 | 0.9300 | C15—H15A | 0.9600 |
| C5—C6 | 1.388 (3) | C15—H15B | 0.9600 |
| C6—H6 | 0.9300 | C15—H15C | 0.9600 |
| C7—C8 | 1.381 (4) | C16—H16A | 0.9600 |
| C7—C12 | 1.390 (4) | C16—H16B | 0.9600 |
| C8—H8 | 0.9300 | C16—H16C | 0.9600 |
| C8—C9 | 1.383 (4) | | |

| | | | |
|---|---|---|---|
| O1—S1—N1 | 109.58 (13) | C9—C10—H10 | 120.1 |
| O1—S1—C13 | 106.21 (12) | C9—C10—C11 | 119.8 (3) |
| N1—S1—C13 | 99.00 (13) | C11—C10—H10 | 120.1 |
| S1—N1—H1 | 118.6 | C10—C11—H11 | 119.6 |
| C1—N1—S1 | 122.8 (2) | C10—C11—C12 | 120.8 (3) |
| C1—N1—H1 | 118.6 | C12—C11—H11 | 119.6 |
| C2—C1—N1 | 123.1 (3) | C7—C12—C11 | 120.2 (3) |
| C6—C1—N1 | 117.5 (2) | C7—C12—H12 | 119.9 |
| C6—C1—C2 | 119.3 (2) | C11—C12—H12 | 119.9 |
| C1—C2—H2 | 120.2 | C14—C13—S1 | 105.0 (2) |
| C1—C2—C3 | 119.5 (3) | C14—C13—C15 | 109.7 (3) |
| C3—C2—H2 | 120.2 | C15—C13—S1 | 107.2 (2) |
| C2—C3—H3 | 118.9 | C16—C13—S1 | 111.5 (2) |
| C4—C3—C2 | 122.2 (3) | C16—C13—C14 | 110.6 (3) |
| C4—C3—H3 | 118.9 | C16—C13—C15 | 112.6 (3) |
| C3—C4—C5 | 116.8 (2) | C13—C14—H14A | 109.5 |
| C3—C4—C7 | 122.0 (2) | C13—C14—H14B | 109.5 |
| C5—C4—C7 | 121.2 (2) | C13—C14—H14C | 109.5 |
| C4—C5—H5 | 119.1 | H14A—C14—H14B | 109.5 |
| C6—C5—C4 | 121.7 (3) | H14A—C14—H14C | 109.5 |
| C6—C5—H5 | 119.1 | H14B—C14—H14C | 109.5 |
| C1—C6—C5 | 120.4 (3) | C13—C15—H15A | 109.5 |
| C1—C6—H6 | 119.8 | C13—C15—H15B | 109.5 |
| C5—C6—H6 | 119.8 | C13—C15—H15C | 109.5 |
| C8—C7—C4 | 121.9 (2) | H15A—C15—H15B | 109.5 |
| C8—C7—C12 | 117.6 (2) | H15A—C15—H15C | 109.5 |
| C12—C7—C4 | 120.5 (2) | H15B—C15—H15C | 109.5 |
| C7—C8—H8 | 119.1 | C13—C16—H16A | 109.5 |
| C7—C8—C9 | 121.9 (3) | C13—C16—H16B | 109.5 |
| C9—C8—H8 | 119.1 | C13—C16—H16C | 109.5 |

| | | | |
|---|---|---|---|
| C8—C9—H9 | 120.1 | H16A—C16—H16B | 109.5 |
| C10—C9—C8 | 119.8 (3) | H16A—C16—H16C | 109.5 |
| C10—C9—H9 | 120.1 | H16B—C16—H16C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.86 | 2.35 | 3.144 (3) | 154 |

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