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Crystal structure of (*E*)-*N*′-{[(1*R*,3*R*)-3-isopropyl-1-methyl-2-oxocyclopentyl]-methylidene}-4-methylbenzenesulfonohydrazide

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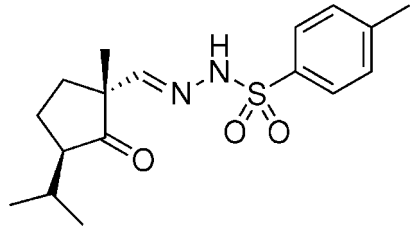
The title compound, $C_{17}H_{24}N_2O_3S$, was synthesized in order to determine the relative configuration of the corresponding β-keto aldehyde. In the U-shaped molecule, the five-membered ring approximates an envelope with the methylene atom adjacent to the quaternary C atom being the flap. The dihedral angles between the four nearly coplanar atoms of the five-membered ring and the flap and the aromatic ring are 38.8 (4) and 22.9 (2)°, respectively. The bond angles around the S atom are in the range 104.11 (16)–119.95 (16)°. In the crystal, molecules are linked *via* N—H···O by hydrogen bonds, forming a chain along the *a*-axis direction.

Keywords: crystal structure; benzenesulfonohydrazide; terpenoid-related building blocks; hydrogen bonding; cyclopentanoids.

CCDC reference: 1037859

1. Related literature

For the synthesis of terpenoid-related building blocks, in particular cyclopentanoids, see: Becker *et al.* (2013); Gille *et al.* (2011); Helmboldt *et al.* (2006); Nelson *et al.* (2011); Tymann *et al.*(2014). For a review on cyclopentanoids by ring contraction see: Silva (2002). For a solid–acid catalysed rearrangement of cyclic α,β-epoxy ketones see: Elings *et al.* (2000).



2. Experimental

2.1. Crystal data

$C_{17}H_{24}N_2O_3S$
M_r = 336.44
Monoclinic, $P2_1$
a = 6.6198 (8) Å
b = 16.8318 (18) Å
c = 7.9506 (9) Å
β = 97.141 (11)°
V = 879.00 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 173 K
0.23 × 0.10 × 0.03 mm

2.2. Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Absorption correction: multi-scan (*CrysAlis CCD*; Oxford Diffraction, 2008)
T_{min} = 0.98, T_{max} = 1.00
6620 measured reflections
3684 independent reflections
3185 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.091
S = 1.01
3684 reflections
216 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack *x* determined using 1307 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack,2004)
Absolute structure parameter: 0.02 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O3^{i}	0.91 (4)	2.03 (4)	2.889 (4)	158 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED* (Oxford Diffraction, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Acknowledgements

Financial support obtained from the Beilstein Institut is gratefully acknowledged

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5349).

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supporting information

Acta Cryst. (2015). E**71**, o99–o100 [doi:10.1107/S2056989014026747]

Crystal structure of (*E*)-*N*′-{[(1*R*,3*R*)-3-isopropyl-1-methyl-2-oxocyclopentyl]-methylidene}-4-methylbenzenesulfonohydrazide

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S1. Comment

Prompted by our efforts in natural product synthesis, we seek access to cyclopentyl units. Herein, we chosed a ring contraction strategy of a cyclic epoxy ketone. A Brønsted-acid promoted [1,2]-sigmatropic rearrangement of *cis*-piperitone oxide delivered *trans*-3-Isopropyl-1-methyl-2-oxocyclopentane-1-carbaldehyde (II). A subsequent condensation of (II) with *p*-toulenesulfonyl hydrazide afforded the title compound (I).

S2. Experimental

A sealable glass pressure tube was charged with a solution of *trans*-3-Isopropyl-1-methyl-2-oxocyclopentane-1-carbaldehyde (II) ($C_{10}H_{16}O_2$, M = 168.23 g/mol, $[\alpha]_D^{20}$ = +248.3 (c 0.059 mol/*L*, $CHCl_3$), 150 mg, 0.89 mmol, 1.0 eq) and *p*-toluenesulfonyl hydrazide ($C_7H_{10}N_2O_2S$, M = 186.23 g/mol, 232 mg, 1.24 mmol, 1.4 eq) in methanol (9 ml, 10.1 ml/mmol). The tube was sealed with a Teflon screw cap and placed in a pre-heated oil bath (353 K). After being stirred for 2.5 h at 353 K, the reaction mixture was cooled to ambient temperature and stirred for additional 16 h at room temperature. Next, the volatiles were removed under reduced pressure. Purification of the residue by flash chromatography (cyclohexane/ethyl acetate 50/1 to 5/1) delivered the title compound (I) ($C_{17}H_{24}N_2O_3S$, M = 336.45 g/mol, 161 mg, 0.48 mmol, 54%) as a white solid and as an apparent mixture of double bond isomers (ratio = 68:32). Subsequent recrystallization of (I) from *n*-pentane provided colourless plates of the *E*-configured double bond isomer of (I). The ratio of isomers was determined by integration of the ^{1}H NMR signals at 6.66 p.p.m. (s, 1H) and 7.12 p.p.m. (s, 1H). Characterization data are reported for the mixture of isomers. R_f 0.44 (cyclohexane/ethyl acetate 2/1); m.p. 361–363 K; ^{1}H NMR ($CDCl_3$, 500 MHz, ratio of double bond isomers = 68:32) δ 0.81 (d, J = 6.7 Hz, 3H^{major}), 0.93 (d, J = 6.9 Hz, 3H^{minor}), 0.97 (d, J = 6.7 Hz, 3H^{major}), 1.08–1.09 (m, 3H^{major}+3H^{minor}), 1.12 (s, 3H^{minor}), 1.52–1.59 (m, 1H^{minor}), 1.62–1.81 (m, 2H^{major}+3H^{minor}), 1.93–2.02 (m, 1H^{major}), 2.05–2.23 (m, 3H^{major}+1H^{minor}), 2.42 (s, 3H^{minor}), 2.43 (s, 3H^{major}), 2.53–2.56 (m, 1H^{minor}), 6.66 (s, 1H^{minor}), 7.12 (s 1H^{minor}), 7.29–7.32 (m, 2H^{major}+2H^{minor}), 7.58 (br. s, 1H), 7.70 (br. s, 1H), 7.79 (d, J = 8.2 Hz, 2H^{major}), 7.84 (d, J = 8.2 Hz, 2H^{minor}); ^{13}C NMR ($CDCl_3$, 126 MHz) δ 16.9 (CH_3^{minor}), 18.5 (CH_3^{major}), 19.3 (CH_3^{minor}), 20.1 (CH_3^{major}), 20.5 (CH_2^{major}), 20.9 (CH_3^{major}), 21.57 (CH_3^{minor}), 21.59 (CH_3^{major}), 23.7 (CH_3^{minor}), 24.3 (CH_2^{minor}), 26.2 (CH^{major}), 27.3 (CH^{minor}), 31.4 (CH_2^{major}), 35.8 (CH_2^{minor}), 53.1 (C^{major}), 55.2 (CH^{minor}), 55.3 (CH^{major}), 61.9 (C^{minor}), 127.8 (CH^{minor}), 127.9 (CH^{major}), 129.4 (CH^{minor}), 129.5 (CH^{major}), 135.1 (C^{major}), 136.6 (C^{minor}), 134.8 (C^{minor}), 144.2 (C^{major}), 152.8 (CH^{major}), 154.0 (CH^{minor}), 218.9 (C^{major}+C^{minor}); IR ν 3445 (*m*), 3175 (*m*), 2960 (*m*), 2360 (w), 1730 (*s*), 1595 (*m*), 1470 (*m*), 1355 (*s*), 1320 (*m*), 1185 (*s*), 1165 (*s*), 1093 (*m*), 815 (*s*); Anal. Calcd. for $C_{17}H_{24}N_2O_3S$: C, 60.7; H, 7.2; N, 8.3; Found: C, 60.9; H, 7.2; N, 8.1.

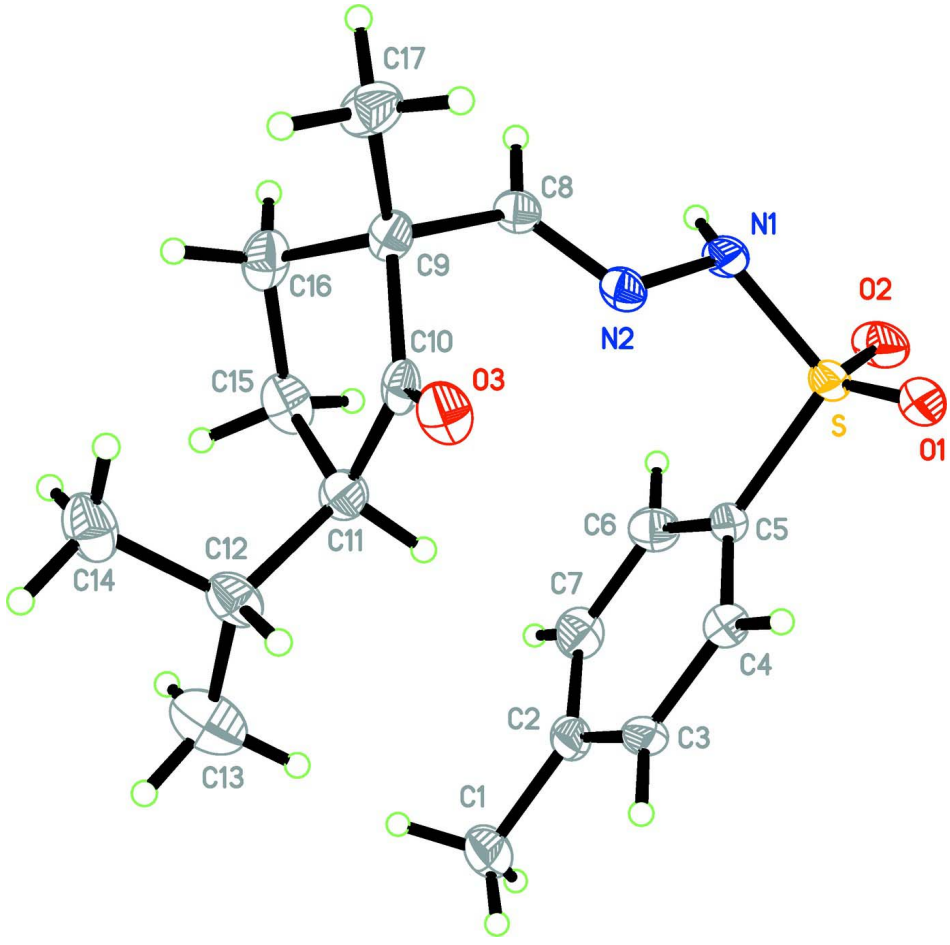

Figure 1

The molecular structure of the title compound, showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 30% probability level.

(*E*)-*N*′-{[(1*R*,3*R*)-3-Isopropyl-1-methyl-2-oxocyclopentyl]methylidene}-4-methylbenzenesulfonohydrazide

Crystal data

$C_{17}H_{24}N_2O_3S$
$M_r = 336.44$
Monoclinic, $P2_1$
$a = 6.6198\ (8)$ Å
$b = 16.8318\ (18)$ Å
$c = 7.9506\ (9)$ Å
$\beta = 97.141\ (11)°$
$V = 879.00\ (17)$ Å^3
$Z = 2$
$F(000) = 360$
$D_x = 1.271$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\mu = 0.20$ mm^{-1}
$T = 173$ K
Plate, colourless
0.23 × 0.10 × 0.03 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Detector resolution: 16.0560 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*CrysAlis CCD*; Oxford Diffraction, 2008)
$T_{min} = 0.98$, $T_{max} = 1.00$
6620 measured reflections
3684 independent reflections
3185 reflections with $I > 2\sigma(I)$

R_{int} = 0.032
θ_{max} = 27.0°, θ_{min} = 2.4°
h = −7→8
k = −21→21
l = −10→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.091
S = 1.01
3684 reflections
216 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack x determined using 1307 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack,2004)
Absolute structure parameter: 0.02 (5)

Special details

Experimental. Absorption correction: *CrysAlis PRO*, Agilent Technologies, Version 1.171.36.24 (release 03–12-2012 CrysAlis171. NET) (compiled Dec 3 2012,18:21:49) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}
S	0.09218 (13)	0.45682 (4)	0.12604 (10)	0.0211 (2)
O1	0.2056 (4)	0.41202 (15)	0.0179 (3)	0.0301 (7)
O2	−0.1089 (4)	0.43231 (14)	0.1507 (3)	0.0328 (7)
O3	0.7126 (4)	0.64917 (14)	0.0645 (3)	0.0263 (6)
N1	0.0612 (5)	0.54744 (16)	0.0481 (4)	0.0201 (7)
N2	0.2410 (4)	0.59285 (16)	0.0665 (4)	0.0190 (6)
C1	0.6011 (7)	0.4966 (3)	0.8060 (5)	0.0363 (10)
H1A	0.6121	0.5531	0.8347	0.054*
H1B	0.7374	0.4746	0.8012	0.054*
H1C	0.5358	0.4684	0.8926	0.054*
C2	0.4754 (6)	0.4867 (2)	0.6364 (5)	0.0233 (8)
C4	0.4451 (5)	0.4458 (2)	0.3415 (4)	0.0209 (8)
H4	0.5048	0.4249	0.2483	0.025*
C3	0.5595 (5)	0.4551 (2)	0.4991 (4)	0.0233 (7)
H3	0.6986	0.4396	0.5133	0.028*

C5	0.2427 (5)	0.46758 (19)	0.3241 (4)	0.0185 (7)
C6	0.1547 (6)	0.4995 (2)	0.4585 (5)	0.0251 (8)
H6	0.0157	0.5151	0.4435	0.030*
C7	0.2701 (6)	0.5084 (2)	0.6137 (5)	0.0277 (9)
H7	0.2095	0.5295	0.7063	0.033*
C8	0.2181 (5)	0.66745 (19)	0.0519 (4)	0.0190 (7)
H8	0.0848	0.6889	0.0286	0.023*
C9	0.3979 (6)	0.7225 (2)	0.0708 (5)	0.0206 (8)
C10	0.5880 (5)	0.67705 (19)	0.1474 (5)	0.0199 (8)
C11	0.6049 (6)	0.6771 (2)	0.3399 (5)	0.0229 (8)
H11	0.5766	0.6223	0.3794	0.027*
C12	0.8215 (6)	0.7019 (2)	0.4184 (5)	0.0260 (9)
H12	0.9204	0.6689	0.3633	0.031*
C13	0.8553 (7)	0.6835 (3)	0.6095 (5)	0.0462 (12)
H13A	0.9966	0.6954	0.6544	0.069*
H13B	0.8275	0.6271	0.6276	0.069*
H13C	0.7633	0.7161	0.6681	0.069*
C14	0.8694 (6)	0.7886 (2)	0.3864 (5)	0.0331 (10)
H14A	0.7745	0.8227	0.4385	0.050*
H14B	0.8555	0.7986	0.2641	0.050*
H14C	1.0091	0.8005	0.4361	0.050*
C15	0.4298 (6)	0.7325 (2)	0.3774 (5)	0.0283 (9)
H15A	0.3113	0.7012	0.4040	0.034*
H15B	0.4747	0.7677	0.4747	0.034*
C16	0.3749 (6)	0.7811 (2)	0.2166 (5)	0.0282 (9)
H16A	0.4683	0.8268	0.2132	0.034*
H16B	0.2334	0.8011	0.2095	0.034*
C17	0.4217 (6)	0.7612 (2)	−0.0986 (5)	0.0296 (10)
H17A	0.4403	0.7200	−0.1823	0.044*
H17B	0.5406	0.7964	−0.0853	0.044*
H17C	0.2994	0.7923	−0.1372	0.044*
H1N	−0.051 (6)	0.572 (2)	0.079 (5)	0.027 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0204 (5)	0.0192 (4)	0.0219 (4)	−0.0035 (4)	−0.0047 (3)	0.0012 (4)
O1	0.0387 (18)	0.0257 (13)	0.0233 (14)	0.0052 (12)	−0.0065 (13)	−0.0048 (11)
O2	0.0232 (16)	0.0337 (15)	0.0382 (16)	−0.0125 (11)	−0.0090 (12)	0.0108 (12)
O3	0.0152 (14)	0.0305 (14)	0.0335 (15)	0.0027 (11)	0.0039 (12)	−0.0092 (12)
N1	0.0120 (16)	0.0218 (16)	0.0252 (17)	−0.0016 (12)	−0.0024 (13)	0.0021 (13)
N2	0.0129 (16)	0.0229 (15)	0.0209 (16)	−0.0022 (12)	0.0005 (12)	0.0003 (12)
C1	0.046 (3)	0.032 (2)	0.027 (2)	−0.004 (2)	−0.009 (2)	−0.0023 (18)
C2	0.029 (2)	0.0184 (18)	0.021 (2)	−0.0061 (15)	−0.0019 (17)	0.0027 (14)
C4	0.0215 (19)	0.0194 (19)	0.0217 (17)	0.0011 (16)	0.0023 (14)	0.0007 (15)
C3	0.0167 (17)	0.0217 (16)	0.0296 (18)	0.0004 (17)	−0.0053 (14)	0.0021 (19)
C5	0.0200 (18)	0.0157 (18)	0.0188 (16)	−0.0015 (14)	−0.0018 (14)	0.0005 (14)
C6	0.020 (2)	0.0289 (19)	0.026 (2)	0.0013 (16)	0.0034 (17)	0.0025 (16)

C7	0.034 (2)	0.028 (2)	0.021 (2)	0.0013 (17)	0.0071 (18)	−0.0025 (16)
C8	0.0119 (18)	0.0218 (18)	0.0229 (18)	0.0016 (14)	0.0008 (14)	−0.0003 (15)
C9	0.0154 (19)	0.0183 (18)	0.029 (2)	−0.0004 (14)	0.0039 (16)	0.0001 (15)
C10	0.014 (2)	0.0154 (17)	0.031 (2)	−0.0055 (14)	0.0051 (16)	−0.0030 (15)
C11	0.019 (2)	0.0229 (19)	0.027 (2)	−0.0016 (15)	0.0032 (16)	0.0008 (16)
C12	0.017 (2)	0.035 (2)	0.026 (2)	0.0014 (15)	0.0024 (17)	−0.0061 (17)
C13	0.037 (3)	0.067 (3)	0.034 (3)	−0.001 (2)	0.000 (2)	−0.002 (2)
C14	0.021 (2)	0.038 (2)	0.041 (2)	−0.0060 (17)	0.0044 (19)	−0.0082 (19)
C15	0.021 (2)	0.035 (2)	0.030 (2)	−0.0016 (17)	0.0063 (17)	−0.0097 (18)
C16	0.020 (2)	0.0221 (18)	0.043 (2)	0.0017 (15)	0.0068 (18)	−0.0071 (17)
C17	0.024 (2)	0.028 (2)	0.037 (2)	0.0000 (16)	0.0047 (19)	0.0094 (17)

Geometric parameters (Å, º)

S—O1	1.426 (3)	C9—C17	1.522 (5)
S—O2	1.431 (3)	C9—C10	1.533 (5)
S—N1	1.649 (3)	C9—C16	1.544 (5)
S—C5	1.765 (3)	C10—C11	1.520 (5)
O3—C10	1.213 (4)	C11—C15	1.546 (5)
N1—N2	1.407 (4)	C11—C12	1.548 (5)
N1—H1N	0.91 (4)	C11—H11	1.0000
N2—C8	1.268 (4)	C12—C14	1.522 (6)
C1—C2	1.503 (5)	C12—C13	1.539 (5)
C1—H1A	0.9800	C12—H12	1.0000
C1—H1B	0.9800	C13—H13A	0.9800
C1—H1C	0.9800	C13—H13B	0.9800
C2—C3	1.391 (5)	C13—H13C	0.9800
C2—C7	1.398 (5)	C14—H14A	0.9800
C4—C5	1.380 (5)	C14—H14B	0.9800
C4—C3	1.390 (5)	C14—H14C	0.9800
C4—H4	0.9500	C15—C16	1.523 (6)
C3—H3	0.9500	C15—H15A	0.9900
C5—C6	1.387 (5)	C15—H15B	0.9900
C6—C7	1.376 (5)	C16—H16A	0.9900
C6—H6	0.9500	C16—H16B	0.9900
C7—H7	0.9500	C17—H17A	0.9800
C8—C9	1.501 (5)	C17—H17B	0.9800
C8—H8	0.9500	C17—H17C	0.9800
O1—S—O2	119.95 (16)	O3—C10—C9	123.9 (3)
O1—S—N1	108.22 (16)	C11—C10—C9	110.7 (3)
O2—S—N1	104.11 (16)	C10—C11—C15	103.4 (3)
O1—S—C5	108.13 (16)	C10—C11—C12	110.8 (3)
O2—S—C5	109.82 (16)	C15—C11—C12	116.0 (3)
N1—S—C5	105.67 (15)	C10—C11—H11	108.8
N2—N1—S	113.5 (2)	C15—C11—H11	108.8
N2—N1—H1N	116 (2)	C12—C11—H11	108.8
S—N1—H1N	113 (2)	C14—C12—C13	110.5 (3)

C8—N2—N1	116.0 (3)	C14—C12—C11	113.1 (3)
C2—C1—H1A	109.5	C13—C12—C11	110.9 (3)
C2—C1—H1B	109.5	C14—C12—H12	107.3
H1A—C1—H1B	109.5	C13—C12—H12	107.3
C2—C1—H1C	109.5	C11—C12—H12	107.3
H1A—C1—H1C	109.5	C12—C13—H13A	109.5
H1B—C1—H1C	109.5	C12—C13—H13B	109.5
C3—C2—C7	118.4 (3)	H13A—C13—H13B	109.5
C3—C2—C1	121.0 (4)	C12—C13—H13C	109.5
C7—C2—C1	120.6 (4)	H13A—C13—H13C	109.5
C5—C4—C3	118.3 (3)	H13B—C13—H13C	109.5
C5—C4—H4	120.8	C12—C14—H14A	109.5
C3—C4—H4	120.8	C12—C14—H14B	109.5
C4—C3—C2	121.6 (3)	H14A—C14—H14B	109.5
C4—C3—H3	119.2	C12—C14—H14C	109.5
C2—C3—H3	119.2	H14A—C14—H14C	109.5
C4—C5—C6	121.4 (3)	H14B—C14—H14C	109.5
C4—C5—S	119.7 (3)	C16—C15—C11	105.5 (3)
C6—C5—S	118.8 (3)	C16—C15—H15A	110.6
C7—C6—C5	119.5 (3)	C11—C15—H15A	110.6
C7—C6—H6	120.2	C16—C15—H15B	110.6
C5—C6—H6	120.2	C11—C15—H15B	110.6
C6—C7—C2	120.7 (3)	H15A—C15—H15B	108.8
C6—C7—H7	119.7	C15—C16—C9	104.6 (3)
C2—C7—H7	119.7	C15—C16—H16A	110.8
N2—C8—C9	121.2 (3)	C9—C16—H16A	110.8
N2—C8—H8	119.4	C15—C16—H16B	110.8
C9—C8—H8	119.4	C9—C16—H16B	110.8
C8—C9—C17	110.1 (3)	H16A—C16—H16B	108.9
C8—C9—C10	109.5 (3)	C9—C17—H17A	109.5
C17—C9—C10	113.3 (3)	C9—C17—H17B	109.5
C8—C9—C16	108.5 (3)	H17A—C17—H17B	109.5
C17—C9—C16	114.9 (3)	C9—C17—H17C	109.5
C10—C9—C16	100.0 (3)	H17A—C17—H17C	109.5
O3—C10—C11	125.3 (3)	H17B—C17—H17C	109.5

O1—S—N1—N2	−71.4 (3)	N2—C8—C9—C10	−13.1 (5)
O2—S—N1—N2	159.9 (2)	N2—C8—C9—C16	−121.3 (3)
C5—S—N1—N2	44.2 (3)	C8—C9—C10—O3	97.8 (4)
S—N1—N2—C8	−161.8 (3)	C17—C9—C10—O3	−25.6 (5)
C5—C4—C3—C2	−0.9 (5)	C16—C9—C10—O3	−148.4 (3)
C7—C2—C3—C4	0.7 (5)	C8—C9—C10—C11	−86.2 (4)
C1—C2—C3—C4	−179.9 (3)	C17—C9—C10—C11	150.5 (3)
C3—C4—C5—C6	1.2 (5)	C16—C9—C10—C11	27.7 (4)
C3—C4—C5—S	179.9 (3)	O3—C10—C11—C15	169.9 (3)
O1—S—C5—C4	10.2 (3)	C9—C10—C11—C15	−6.1 (4)
O2—S—C5—C4	142.7 (3)	O3—C10—C11—C12	45.0 (5)
N1—S—C5—C4	−105.5 (3)	C9—C10—C11—C12	−131.0 (3)

O1—S—C5—C6	−171.1 (3)	C10—C11—C12—C14	68.4 (4)
O2—S—C5—C6	−38.5 (3)	C15—C11—C12—C14	−49.0 (4)
N1—S—C5—C6	73.2 (3)	C10—C11—C12—C13	−166.7 (3)
C4—C5—C6—C7	−1.2 (5)	C15—C11—C12—C13	75.8 (4)
S—C5—C6—C7	−179.9 (3)	C10—C11—C15—C16	−18.9 (4)
C5—C6—C7—C2	0.9 (6)	C12—C11—C15—C16	102.6 (4)
C3—C2—C7—C6	−0.7 (5)	C11—C15—C16—C9	37.0 (4)
C1—C2—C7—C6	179.9 (3)	C8—C9—C16—C15	75.9 (4)
N1—N2—C8—C9	179.3 (3)	C17—C9—C16—C15	−160.4 (3)
N2—C8—C9—C17	112.1 (4)	C10—C9—C16—C15	−38.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O3^{i}	0.91 (4)	2.03 (4)	2.889 (4)	158 (3)

Symmetry code: (i) *x*−1, *y*, *z*.