data reports

Crystal structure of N′-[(E)-(1S,3R)-(3-isopropyl-1-methyl-2-oxocyclopentyl)-methylidene]-4-methylbenzenesulfono-hydrazide

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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 C atom adjacent to the quaternary C atom being the flap, and the methyl and isopropyl substituents lying to the same side of the ring. The dihedral angles between the four nearly coplanar atoms of the five-membered ring and the flap and the aromatic ring are 35.74 (15) and 55.72 (9)°, respectively. The bond angles around the S atom are in the range from 103.26 (12) to 120.65 (14)°. In the crystal, molecules are linked via N—H⋯O hydrogen bonds, forming a chain along the a axis.

1. Related literature

For the synthesis of terpenoid-related buildings blocks, in particular cyclopentanoids, see: Helmboldt et al. (2006); Gille et al. (2011); Becker et al. (2013); Tymann et al. (2014). For the crystal structure of the corresponding trans-diastereomer, see: Tymann et al. (2015). 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).
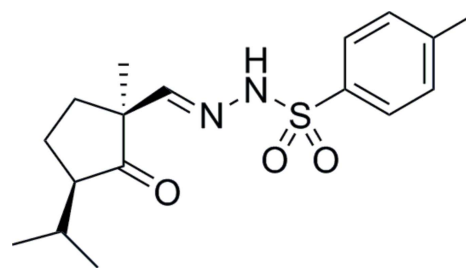
2. Experimental

2.1. Crystal data

$C_{17}H_{24}N_2O_3S$	$V = 1842.8$ (3) Å^3
$M_r = 336.44$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 9.4918$ (7) Å	$\mu = 0.19$ mm^{-1}
$b = 13.2348$ (12) Å	$T = 173$ K
$c = 14.6691$ (12) Å	$0.31 \times 0.25 \times 0.23$ mm

2.2. Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer	15093 measured reflections
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)	4215 independent reflections
	3526 reflections with $I > 2\sigma(I)$
$T_{min} = 0.98$, $T_{max} = 1.00$	$R_{int} = 0.046$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$wR(F^2) = 0.093$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
$S = 1.05$	Absolute structure: Flack x determined using 1277 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
4215 reflections	
216 parameters	
H atoms treated by a mixture of independent and constrained refinement	Absolute structure parameter: −0.02 (4)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1$N\cdots$O3^i	0.86 (3)	2.00 (4)	2.836 (3)	164 (3)

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z$.

Data collection: CrysAlis CCD (Oxford Diffraction, 2008); cell refinement: CrysAlis CCD; data reduction: CrysAlis CCD; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL2013 (Sheldrick, 2015); molecular graphics: SHELXTL-Plus (Sheldrick, 2008); software used to prepare material for publication: SHELXL97 (Sheldrick, 2008) and PLATON (Spek, 2009).

Acknowledgements

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supporting information

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

David Tymann, Dina Christina Dragon, Christopher Golz, Hans Preut, Carsten Strohmann and Martin Hiersemann

S1. Comment

Prompted by our efforts in natural product synthesis, we seek access to cyclopentyl units. Herein, we chose a ring contraction strategy of a cyclic epoxy ketone. A Brønsted-acid promoted semi-pinacol rearrangement of *trans*-piperitone oxide afforded *cis*-3-isopropyl-1-methyl-2-oxocyclopentane-1-carbaldehyde (II) along with 2-hydroxypiperitone. A 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 *cis*-3-isopropyl-1-methyl-2-oxocyclopentane-1-carbaldehyde (II) ($C_{10}H_{16}O_2$, M = 168.23 g/mol, $[\alpha]_D^{20}$ = -107.4 (c 0.059, CHCl$_3$), 40 mg, 0.238 mmol, 1.0 eq) and *p*-toluenesulfonyl hydrazide ($C_7H_{10}N_2O_2S$, M = 186.23 g/mol, 62 mg, 0.333 mmol, 1.4 eq) in methanol (3 ml, 12.6 ml/mmol). The tube was sealed with a Teflon screw cap and stirred for 75 min at ambient temperature. Next, the reaction mixture was concentrated *in vacuo* and loaded onto silica gel with CH_2Cl_2 for flash chromatography (cyclohexane/ethyl acetate 10/1) to deliver the title compound (I) ($C_{17}H_{24}N_2O_3S$, M = 336.45 g/mol, 56 mg, 0.166 mmol, 70%) as a white solid and as an apparent mixture of double bond isomers (ratio = 95:5). Subsequent recrystallization of (I) from *n*-pentane/Et$_2$O provided colourless crystals of the *E*-configured double bond isomer of (I). The ratio of isomers was determined by integration of the ^{1}H NMR signals at 0.35 p.p.m. (d, J = 6.9 Hz, 3H) and 0.47 p.p.m. (d, J = 6.9 Hz, 3H). Characterization data are reported for the major compound from the mixture of double bond isomers. R_f 0.41 (cyclo-hexane/ethyl acetate 2/1); m.p. 388–391 K; ^{1}H NMR (CDCl$_3$, 500 MHz) δ 0.47 (d, J = 6.9 Hz, 3H), 0.85 (d, J = 6.9 Hz, 3H), 1.16 (s, 3H), 1.48–1.59 (m, 2H), 1.82–1.95 (m, 2H), 2.11 (td, J = 9.4 Hz, J = 5.0 Hz, 1H), 2.42 (s, 3H), 2.49–2.53 (m, 1H), 6.88 (s, 1H), 7.30 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.0 Hz, 2H), 7.89 (s, 1H). ^{13}C NMR (CDCl$_3$, 126 MHz) δ 18.4 (CH$_3$), 20.8 (CH$_3$), 21.2 (CH$_2$), 21.5 (CH$_3$), 21.9 (CH$_3$), 27.8 (CH), 31.6 (CH$_2$), 53.8 (C), 54.7 (CH), 128.0 (2x CH), 129.6 (2x CH), 135.1 (C), 144.2 (C), 150.6 (CH), 219.1 (C). IR v 3415 (*w*), 3126 (*s*), 2960 (*s*), 22870 (*s*), 1725 (*s*), 1600 (*m*), 1455 (*s*), 1360 (*s*), 1330 (*m*), 1170 (*s*), 1095 (*m*), 1050 (*s*), 940 (*m*) 820 (*s*); Anal. Calcd. for $C_{17}H_{24}N_2O_3S$: C, 60.7, H, 7.2, N, 8.3. Found: C, 60.6, H, 7.3, N, 8.3.

S3. Refinement

H-atoms attached to C, except those in CH$_3$, were placed in calculated positions (C—H = 0.95 - 1.00 Å and U_{iso}(H) = 1.2 U_{eq}(C)). All CH$_3$ hydrogen atoms, which were taken from a Fourier map (AFIX 137), were allowed to rotate but not to tip (C—H = 0.98 Å and U_{iso}(H) = 1.5 U_{eq}(C)). The H-atom at N was taken from a Fourier map and refined isotropically.
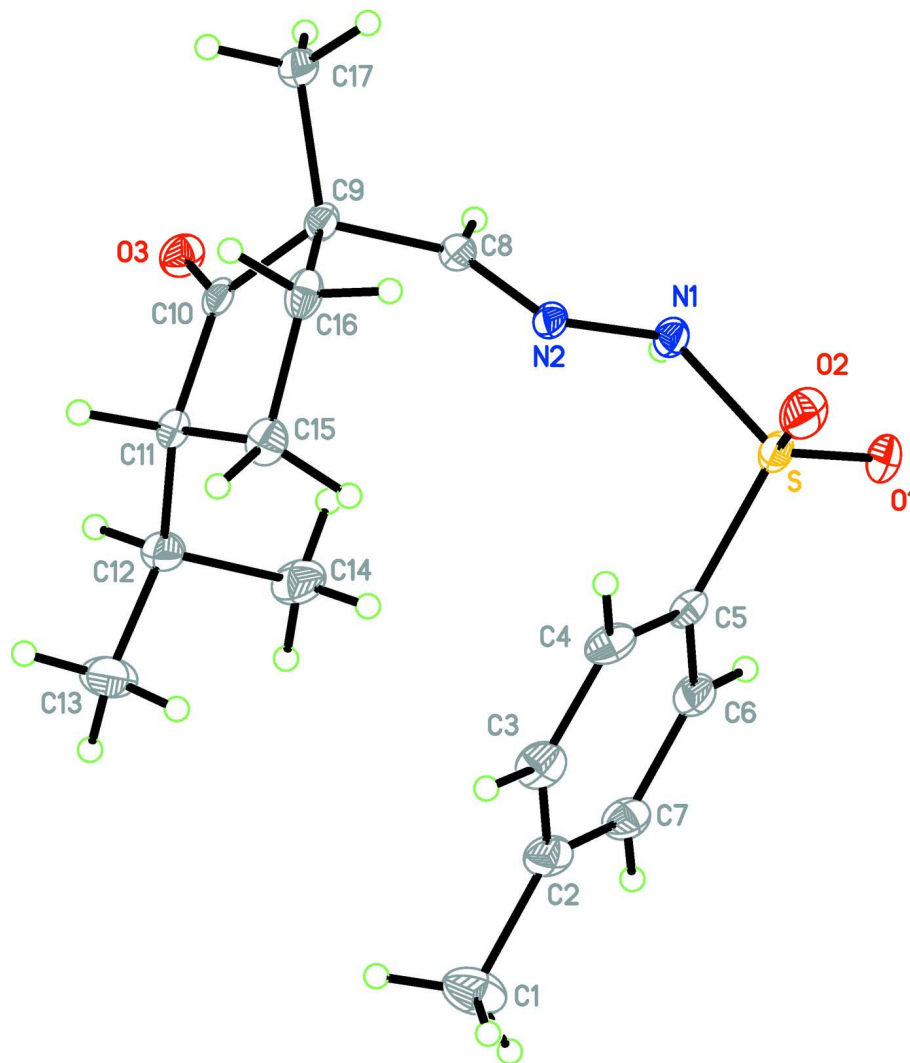
Figure 1

The molecular structure of the title compound, showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

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

Crystal data

$C_{17}H_{24}N_2O_3S$	$D_x = 1.213$ Mg m^{-3}
$M_r = 336.44$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 3585 reflections
$a = 9.4918$ (7) Å	$\theta = 2.6–27.1°$
$b = 13.2348$ (12) Å	$\mu = 0.19$ mm^{-1}
$c = 14.6691$ (12) Å	$T = 173$ K
$V = 1842.8$ (3) Å^3	Block, colourless
$Z = 4$	$0.31 \times 0.25 \times 0.23$ mm
$F(000) = 720$	

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer	15093 measured reflections
Radiation source: Enhance (Mo) X-ray Source	4215 independent reflections
Detector resolution: 16.0560 pixels mm^{-1}	3526 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.046$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)	$\theta_{max} = 28.0°$, $\theta_{min} = 2.6°$
$T_{min} = 0.98$, $T_{max} = 1.00$	$h = -12 \rightarrow 12$
	$k = -15 \rightarrow 16$
	$l = -19 \rightarrow 18$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.042$	
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.1494P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	
4215 reflections	$(\Delta/\sigma)_{max} < 0.001$
216 parameters	$\Delta\rho_{max} = 0.22$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.33$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack x determined using 1277 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
Secondary atom site location: difference Fourier map	Absolute structure parameter: -0.02 (4)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S	0.57466 (7)	0.53116 (6)	0.08855 (5)	0.02777 (17)
O1	0.45052 (19)	0.57619 (18)	0.05053 (14)	0.0389 (6)
O2	0.5837 (2)	0.42449 (16)	0.10068 (15)	0.0411 (5)
O3	1.1776 (2)	0.72234 (15)	−0.00066 (13)	0.0309 (5)
N1	0.6994 (2)	0.5647 (2)	0.01656 (15)	0.0241 (5)
N2	0.8352 (2)	0.53514 (19)	0.04375 (14)	0.0226 (5)
C1	0.7064 (4)	0.7294 (3)	0.4507 (2)	0.0510 (10)
H1A	0.6958	0.6789	0.4990	0.077*
H1B	0.6409	0.7854	0.4614	0.077*
H1C	0.8033	0.7550	0.4506	0.077*
C2	0.6745 (3)	0.6814 (2)	0.35949 (19)	0.0333 (7)
C3	0.7286 (3)	0.5861 (3)	0.3378 (2)	0.0358 (8)
H3	0.7880	0.5526	0.3803	0.043*
C4	0.6978 (3)	0.5398 (2)	0.25650 (18)	0.0308 (6)
H4	0.7341	0.4745	0.2433	0.037*

C5	0.6127 (3)	0.5896 (2)	0.19358 (18)	0.0259 (6)
C6	0.5606 (3)	0.6853 (2)	0.21212 (19)	0.0303 (7)
H6	0.5052	0.7199	0.1681	0.036*
C7	0.5902 (3)	0.7302 (2)	0.2954 (2)	0.0329 (7)
H7	0.5526	0.7950	0.3089	0.039*
C8	0.9350 (3)	0.5757 (2)	−0.00108 (18)	0.0240 (6)
H8	0.9128	0.6228	−0.0478	0.029*
C9	1.0880 (3)	0.5513 (2)	0.01786 (18)	0.0251 (6)
C10	1.1497 (3)	0.6526 (2)	0.05022 (18)	0.0227 (6)
C11	1.1652 (3)	0.6526 (2)	0.15308 (17)	0.0247 (6)
H11	1.2680	0.6440	0.1659	0.030*
C12	1.1208 (3)	0.7517 (2)	0.1995 (2)	0.0320 (7)
H12	1.1814	0.8067	0.1739	0.038*
C13	1.1488 (4)	0.7473 (3)	0.3020 (2)	0.0475 (9)
H13A	1.0847	0.6984	0.3302	0.071*
H13B	1.1332	0.8142	0.3288	0.071*
H13C	1.2464	0.7264	0.3127	0.071*
C14	0.9673 (3)	0.7800 (3)	0.1793 (2)	0.0416 (8)
H14A	0.9047	0.7275	0.2034	0.062*
H14B	0.9538	0.7858	0.1133	0.062*
H14C	0.9451	0.8448	0.2084	0.062*
C15	1.0931 (3)	0.5542 (2)	0.18366 (19)	0.0306 (7)
H15A	1.1406	0.5254	0.2379	0.037*
H15B	0.9927	0.5661	0.1984	0.037*
C16	1.1078 (3)	0.4835 (2)	0.1014 (2)	0.0304 (7)
H16A	1.2019	0.4512	0.1006	0.036*
H16B	1.0349	0.4300	0.1030	0.036*
C17	1.1573 (3)	0.5148 (2)	−0.0706 (2)	0.0359 (7)
H17A	1.2582	0.5039	−0.0602	0.054*
H17B	1.1447	0.5660	−0.1183	0.054*
H17C	1.1135	0.4513	−0.0900	0.054*
H1N	0.690 (3)	0.627 (3)	0.001 (2)	0.040 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0189 (3)	0.0290 (4)	0.0354 (4)	−0.0034 (3)	0.0053 (3)	−0.0060 (3)
O1	0.0171 (10)	0.0542 (15)	0.0455 (12)	0.0008 (10)	−0.0003 (9)	−0.0093 (11)
O2	0.0389 (11)	0.0293 (12)	0.0549 (14)	−0.0106 (10)	0.0121 (11)	−0.0049 (10)
O3	0.0323 (10)	0.0271 (12)	0.0334 (11)	−0.0043 (9)	0.0072 (9)	0.0008 (10)
N1	0.0182 (11)	0.0256 (14)	0.0286 (13)	0.0020 (10)	0.0006 (9)	−0.0015 (10)
N2	0.0177 (10)	0.0213 (12)	0.0286 (11)	0.0023 (10)	0.0013 (9)	−0.0062 (10)
C1	0.077 (3)	0.047 (2)	0.0290 (17)	−0.009 (2)	0.0018 (17)	0.0017 (16)
C2	0.0404 (17)	0.0341 (19)	0.0255 (15)	−0.0055 (15)	0.0097 (13)	0.0052 (12)
C3	0.0395 (17)	0.039 (2)	0.0291 (16)	0.0024 (15)	0.0036 (13)	0.0138 (14)
C4	0.0358 (15)	0.0263 (16)	0.0302 (15)	0.0065 (14)	0.0113 (12)	0.0071 (13)
C5	0.0204 (13)	0.0296 (17)	0.0278 (14)	−0.0006 (11)	0.0080 (11)	0.0012 (12)
C6	0.0249 (15)	0.0327 (17)	0.0334 (16)	0.0049 (14)	0.0025 (12)	−0.0005 (12)

C7	0.0352 (16)	0.0260 (17)	0.0375 (16)	0.0031 (14)	0.0084 (14)	−0.0025 (13)
C8	0.0219 (13)	0.0253 (15)	0.0247 (13)	−0.0015 (12)	0.0019 (11)	−0.0030 (11)
C9	0.0180 (12)	0.0243 (16)	0.0329 (14)	−0.0006 (12)	0.0038 (11)	−0.0059 (11)
C10	0.0126 (12)	0.0245 (16)	0.0310 (14)	0.0022 (11)	0.0036 (11)	−0.0035 (12)
C11	0.0159 (12)	0.0303 (17)	0.0279 (14)	0.0000 (12)	0.0005 (11)	−0.0016 (12)
C12	0.0323 (16)	0.0319 (18)	0.0319 (16)	−0.0016 (13)	0.0042 (12)	−0.0074 (13)
C13	0.054 (2)	0.057 (3)	0.0313 (17)	0.0063 (18)	0.0011 (16)	−0.0120 (16)
C14	0.0392 (18)	0.042 (2)	0.0434 (19)	0.0141 (16)	0.0068 (15)	−0.0046 (15)
C15	0.0272 (14)	0.0316 (17)	0.0330 (15)	0.0023 (13)	−0.0008 (12)	0.0076 (12)
C16	0.0210 (13)	0.0226 (16)	0.0475 (18)	0.0017 (11)	0.0010 (12)	0.0028 (13)
C17	0.0256 (14)	0.041 (2)	0.0417 (17)	−0.0039 (14)	0.0063 (13)	−0.0178 (14)

Geometric parameters (Å, º)

S—O2	1.426 (2)	C9—C16	1.531 (4)
S—O1	1.433 (2)	C9—C17	1.534 (4)
S—N1	1.648 (2)	C9—C10	1.538 (4)
S—C5	1.761 (3)	C10—C11	1.516 (4)
O3—C10	1.216 (3)	C11—C12	1.537 (4)
N1—N2	1.405 (3)	C11—C15	1.539 (4)
N1—H1N	0.86 (3)	C11—H11	1.0000
N2—C8	1.272 (3)	C12—C13	1.528 (4)
C1—C2	1.511 (4)	C12—C14	1.533 (4)
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—C7	1.393 (4)	C13—H13C	0.9800
C2—C3	1.399 (5)	C14—H14A	0.9800
C3—C4	1.372 (4)	C14—H14B	0.9800
C3—H3	0.9500	C14—H14C	0.9800
C4—C5	1.392 (4)	C15—C16	1.533 (4)
C4—H4	0.9500	C15—H15A	0.9900
C5—C6	1.387 (4)	C15—H15B	0.9900
C6—C7	1.387 (4)	C16—H16A	0.9900
C6—H6	0.9500	C16—H16B	0.9900
C7—H7	0.9500	C17—H17A	0.9800
C8—C9	1.514 (3)	C17—H17B	0.9800
C8—H8	0.9500	C17—H17C	0.9800
O2—S—O1	120.65 (14)	O3—C10—C9	123.7 (2)
O2—S—N1	107.67 (13)	C11—C10—C9	110.1 (2)
O1—S—N1	103.26 (12)	C10—C11—C12	114.4 (2)
O2—S—C5	108.25 (14)	C10—C11—C15	104.3 (2)
O1—S—C5	109.04 (13)	C12—C11—C15	118.2 (2)
N1—S—C5	107.17 (12)	C10—C11—H11	106.4
N2—N1—S	113.73 (18)	C12—C11—H11	106.4
N2—N1—H1N	116 (2)	C15—C11—H11	106.4
S—N1—H1N	111 (2)	C13—C12—C14	111.3 (3)

C8—N2—N1	114.7 (2)	C13—C12—C11	110.8 (3)
C2—C1—H1A	109.5	C14—C12—C11	112.6 (3)
C2—C1—H1B	109.5	C13—C12—H12	107.3
H1A—C1—H1B	109.5	C14—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
C7—C2—C3	118.4 (3)	H13A—C13—H13B	109.5
C7—C2—C1	121.2 (3)	C12—C13—H13C	109.5
C3—C2—C1	120.5 (3)	H13A—C13—H13C	109.5
C4—C3—C2	121.5 (3)	H13B—C13—H13C	109.5
C4—C3—H3	119.3	C12—C14—H14A	109.5
C2—C3—H3	119.3	C12—C14—H14B	109.5
C3—C4—C5	119.2 (3)	H14A—C14—H14B	109.5
C3—C4—H4	120.4	C12—C14—H14C	109.5
C5—C4—H4	120.4	H14A—C14—H14C	109.5
C6—C5—C4	120.6 (3)	H14B—C14—H14C	109.5
C6—C5—S	120.0 (2)	C16—C15—C11	104.3 (2)
C4—C5—S	119.4 (2)	C16—C15—H15A	110.9
C5—C6—C7	119.5 (3)	C11—C15—H15A	110.9
C5—C6—H6	120.3	C16—C15—H15B	110.9
C7—C6—H6	120.3	C11—C15—H15B	110.9
C6—C7—C2	120.8 (3)	H15A—C15—H15B	108.9
C6—C7—H7	119.6	C9—C16—C15	105.1 (2)
C2—C7—H7	119.6	C9—C16—H16A	110.7
N2—C8—C9	122.0 (3)	C15—C16—H16A	110.7
N2—C8—H8	119.0	C9—C16—H16B	110.7
C9—C8—H8	119.0	C15—C16—H16B	110.7
C8—C9—C16	112.9 (2)	H16A—C16—H16B	108.8
C8—C9—C17	108.9 (2)	C9—C17—H17A	109.5
C16—C9—C17	116.1 (2)	C9—C17—H17B	109.5
C8—C9—C10	103.6 (2)	H17A—C17—H17B	109.5
C16—C9—C10	102.6 (2)	C9—C17—H17C	109.5
C17—C9—C10	111.9 (2)	H17A—C17—H17C	109.5
O3—C10—C11	126.2 (3)	H17B—C17—H17C	109.5
O2—S—N1—N2	−55.9 (2)	N2—C8—C9—C17	124.3 (3)
O1—S—N1—N2	175.41 (19)	N2—C8—C9—C10	−116.4 (3)
C5—S—N1—N2	60.3 (2)	C8—C9—C10—O3	−76.0 (3)
S—N1—N2—C8	−167.6 (2)	C16—C9—C10—O3	166.3 (2)
C7—C2—C3—C4	−1.5 (4)	C17—C9—C10—O3	41.2 (4)
C1—C2—C3—C4	178.1 (3)	C8—C9—C10—C11	102.3 (2)
C2—C3—C4—C5	1.1 (4)	C16—C9—C10—C11	−15.4 (3)
C3—C4—C5—C6	0.7 (4)	C17—C9—C10—C11	−140.5 (2)
C3—C4—C5—S	179.2 (2)	O3—C10—C11—C12	40.3 (4)
O2—S—C5—C6	−155.0 (2)	C9—C10—C11—C12	−137.9 (2)
O1—S—C5—C6	−22.0 (3)	O3—C10—C11—C15	171.0 (3)
N1—S—C5—C6	89.2 (2)	C9—C10—C11—C15	−7.3 (3)

O2—S—C5—C4	26.5 (2)	C10—C11—C12—C13	−176.4 (2)
O1—S—C5—C4	159.5 (2)	C15—C11—C12—C13	60.2 (3)
N1—S—C5—C4	−89.3 (2)	C10—C11—C12—C14	58.2 (3)
C4—C5—C6—C7	−2.1 (4)	C15—C11—C12—C14	−65.2 (3)
S—C5—C6—C7	179.4 (2)	C10—C11—C15—C16	27.2 (3)
C5—C6—C7—C2	1.7 (4)	C12—C11—C15—C16	155.5 (2)
C3—C2—C7—C6	0.0 (4)	C8—C9—C16—C15	−78.7 (3)
C1—C2—C7—C6	−179.6 (3)	C17—C9—C16—C15	154.5 (2)
N1—N2—C8—C9	−179.0 (2)	C10—C9—C16—C15	32.2 (2)
N2—C8—C9—C16	−6.2 (4)	C11—C15—C16—C9	−37.7 (3)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯O3[i]	0.86 (3)	2.00 (4)	2.836 (3)	164 (3)

Symmetry code: (i) $x-1/2, -y+3/2, -z$.