



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

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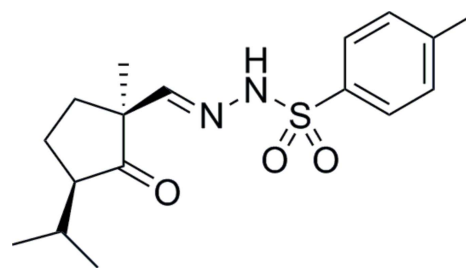
The title compound, $C_{17}H_{24}N_2O_3S$, was synthesized in order to determine the relative configuration of the corresponding β -keto aldehyde. In the U-shaped molecule, the five-membered ring approximates an envelope, with the methylene C atom adjacent to the quaternary C atom being the flap, and 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.

Keywords: crystal structure; hydrogen bonding; terpenoid synthesis.

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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

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

2.2. Data collection

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

2.3. Refinement

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

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O3^i$	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 for this paper is available from the IUCr electronic archives (Reference: TK5399).

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supporting information

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

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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*-toluenesulfonyl 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) (C₁₀H₁₆O₂, *M* = 168.23 g/mol, [α]_D²⁰ = -107.4 (c 0.059, CHCl₃), 40 mg, 0.238 mmol, 1.0 eq) and *p*-toluenesulfonyl hydrazide (C₇H₁₀N₂O₂S, *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 CH₂Cl₂ for flash chromatography (cyclohexane/ethyl acetate 10/1) to deliver the title compound (I) (C₁₇H₂₄N₂O₃S, *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/Et₂O provided colourless crystals of the *E*-configured double bond isomer of (I). The ratio of isomers was determined by integration of the ¹H 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 (cyclohexane/ethyl acetate 2/1); m.p. 388–391 K; ¹H NMR (CDCl₃, 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). ¹³C NMR (CDCl₃, 126 MHz) δ 18.4 (CH₃), 20.8 (CH₃), 21.2 (CH₂), 21.5 (CH₃), 21.9 (CH₃), 27.8 (CH), 31.6 (CH₂), 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 ν 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 C₁₇H₂₄N₂O₃S: 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 CH₃, were placed in calculated positions (C—H = 0.95 - 1.00 Å and *U*_{iso}(H) = 1.2 *U*_{eq}(C)). All CH₃ 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.

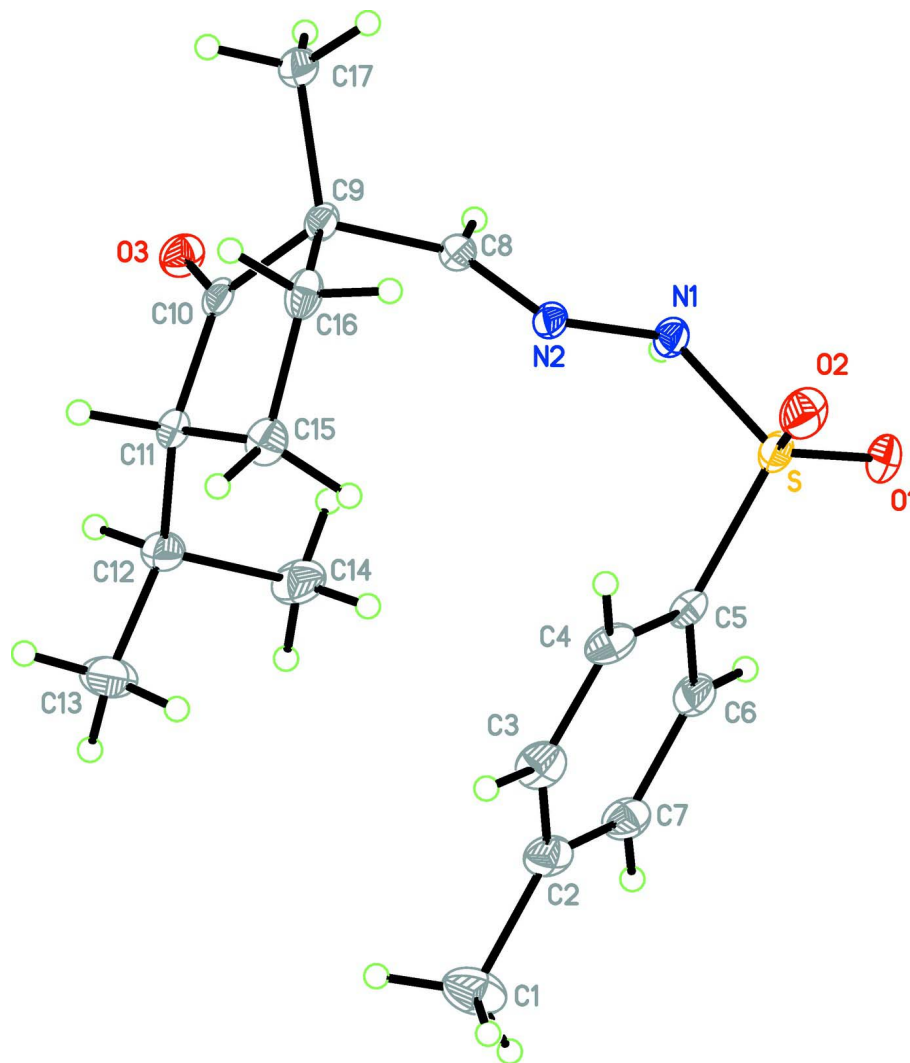


Figure 1

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-methylbenzenesulfonylhydrazide

Crystal data

$C_{17}H_{24}N_2O_3S$

$M_r = 336.44$

Orthorhombic, $P2_12_12_1$

$a = 9.4918$ (7) Å

$b = 13.2348$ (12) Å

$c = 14.6691$ (12) Å

$V = 1842.8$ (3) Å³

$Z = 4$

$F(000) = 720$

$D_x = 1.213$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3585 reflections

$\theta = 2.6$ – 27.1°

$\mu = 0.19$ mm⁻¹

$T = 173$ K

Block, colourless

$0.31 \times 0.25 \times 0.23$ mm

*Data collection*Oxford Diffraction Xcalibur Sapphire3
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Detector resolution: 16.0560 pixels mm⁻¹ φ and ω scans

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2008)

 $T_{\min} = 0.98$, $T_{\max} = 1.00$

15093 measured reflections

4215 independent reflections

3526 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.046$ $\theta_{\max} = 28.0^\circ$, $\theta_{\min} = 2.6^\circ$ $h = -12 \rightarrow 12$ $k = -15 \rightarrow 16$ $l = -19 \rightarrow 18$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.093$ $S = 1.05$

4215 reflections

216 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.1494P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$ Absolute structure: Flack x determined using
1277 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons &
Flack, 2004)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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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, °)

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, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O3 ⁱ	0.86 (3)	2.00 (4)	2.836 (3)	164 (3)

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