



Crystal structure of cafenstrole

Gihaeng Kang, Jineun Kim,* Hyunjin Park and Tae Ho Kim*

Department of Chemistry and Research Institute of Natural Sciences, Gyeongsang National University, Jinju 660-701, Republic of Korea. *Correspondence e-mail: thkim@gnu.ac.kr, jekim@gnu.ac.kr

Received 14 July 2015; accepted 22 July 2015

Edited by P. C. Healy, Griffith University, Australia

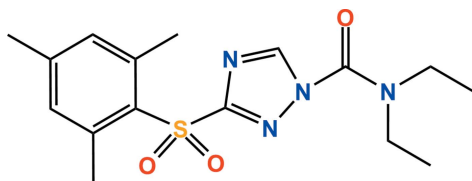
The title compound (systematic name: *N,N*-diethyl-3-mesityl-sulfonyl-1*H*-1,2,4-triazole-1-carboxamide), C₁₆H₂₂N₄O₃S, is a triazole herbicide. The dihedral angle between the planes of the triazole and benzene ring planes is 88.14 (10)°. In the crystal, C—H···O hydrogen bonds and weak C—H···π interactions link adjacent molecules, forming one-dimensional chains along the *a* axis.

Keywords: crystal structure; cafenstrole; triazole; herbicide.

CCDC reference: 1414616

1. Related literature

For information on the herbicidal properties of the title compound, see: Takahashi *et al.* (2001). For related crystal structure, see: Ohkata *et al.* (2002).



2. Experimental

2.1. Crystal data

C₁₆H₂₂N₄O₃S
M_r = 350.43
 Monoclinic, *P*2₁/*c*

a = 7.2800 (3) Å
b = 8.0410 (4) Å
c = 30.1792 (13) Å

β = 95.290 (3)°
V = 1759.12 (14) Å³
Z = 4
 Mo *K*α radiation

μ = 0.21 mm⁻¹
T = 173 K
 0.36 × 0.30 × 0.02 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2013)
*T*_{min} = 0.929, *T*_{max} = 0.996

11871 measured reflections
 3385 independent reflections
 2760 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.033

2.3. Refinement

R[*F*² > 2σ(*F*²)] = 0.051
wR(*F*²) = 0.118
S = 1.12
 3385 reflections

222 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max}$ = 0.32 e Å⁻³
 $\Delta\rho_{\min}$ = -0.35 e Å⁻³

Table 1

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>
C11—H11···O2 ⁱ	0.95	2.38	3.136 (3)	136
C7—H7C···Cg1 ⁱⁱ	0.98	2.80	3.561 (3)	135

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) -*x* + 1, -*y* + 1, -*z*.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (No. 2015R1D1A4A01020317).

Supporting information for this paper is available from the IUCr electronic archives (Reference: HG5453).

References

- Brandenburg, K. (2010). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Ohkata, K., Yano, T., Kojima, S., Hiraga, Y., Yoshii, T. & Hori, M. (2002). *Bull. Chem. Soc. Jpn.*, **75**, 567–574.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
 Takahashi, H., Ohki, A., Kanzaki, M., Tanaka, A., Sato, Y., Matthes, B., Böger, P. & Wakabayashi, K. (2001). *J. Biosci.* **56**, 781–786.

supporting information

Acta Cryst. (2015). E71, o614 [https://doi.org/10.1107/S2056989015013869]

Crystal structure of cafenstrole

Gihaeng Kang, Jineun Kim, Hyunjin Park and Tae Ho Kim

S1. Comment

Cafenstrole, or *N,N*-diethyl-3-mesitylsulfonyl-1*H*-1,2,4-triazole-1-carboxamide, is a triazole herbicide and has been used for rice cultivation which especially inhibits the germination of grass weeds (Takahashi *et al.*, 2001). However, until now its crystal structure has not been reported. In the title compound (Fig. 1), the dihedral angle between the planes of the triazole ring and the phenyl ring planes is 88.14 (10)°. All bond lengths and bond angles are normal and comparable to those observed in similar crystal structure (Ohkata *et al.*, 2002).

In the crystal structure (Fig. 2, Table 1), C11—H11···O2 hydrogen bonds and weak intermolecular C7—H7C···Cg1 (Cg1 is the centroid of the C1—C6 ring) interactions link adjacent molecules, forming one-dimensional chains along to *a*-axis.

S2. Experimental

The title compound was purchased from the Dr. Ehrenstorfer GmbH Company. Slow evaporation of a solution in CH₃CN gave single crystals suitable for X-ray analysis.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with $d(\text{C—H}) = 0.98 \text{ \AA}$, $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$ for methyl group, $d(\text{C—H}) = 0.99 \text{ \AA}$, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for $\text{C}_{\text{sp}^3}\text{—H}$, $d(\text{C—H}) = 0.95 \text{ \AA}$, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for aromatic C—H.

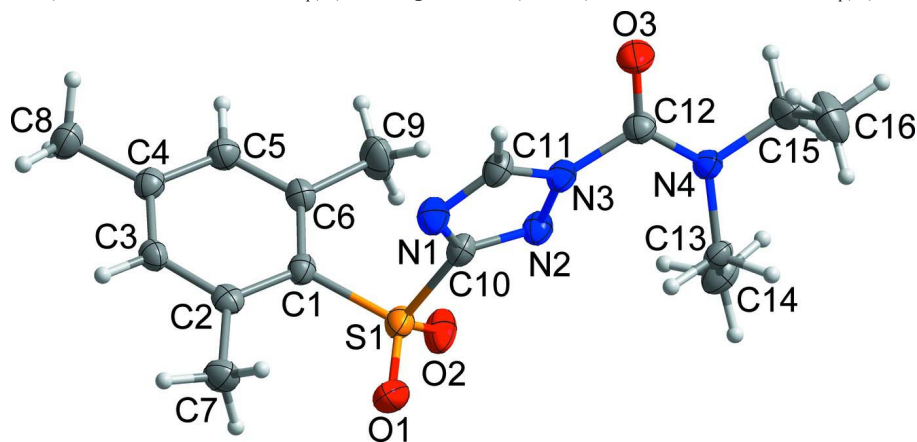


Figure 1

The asymmetric unit of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius.

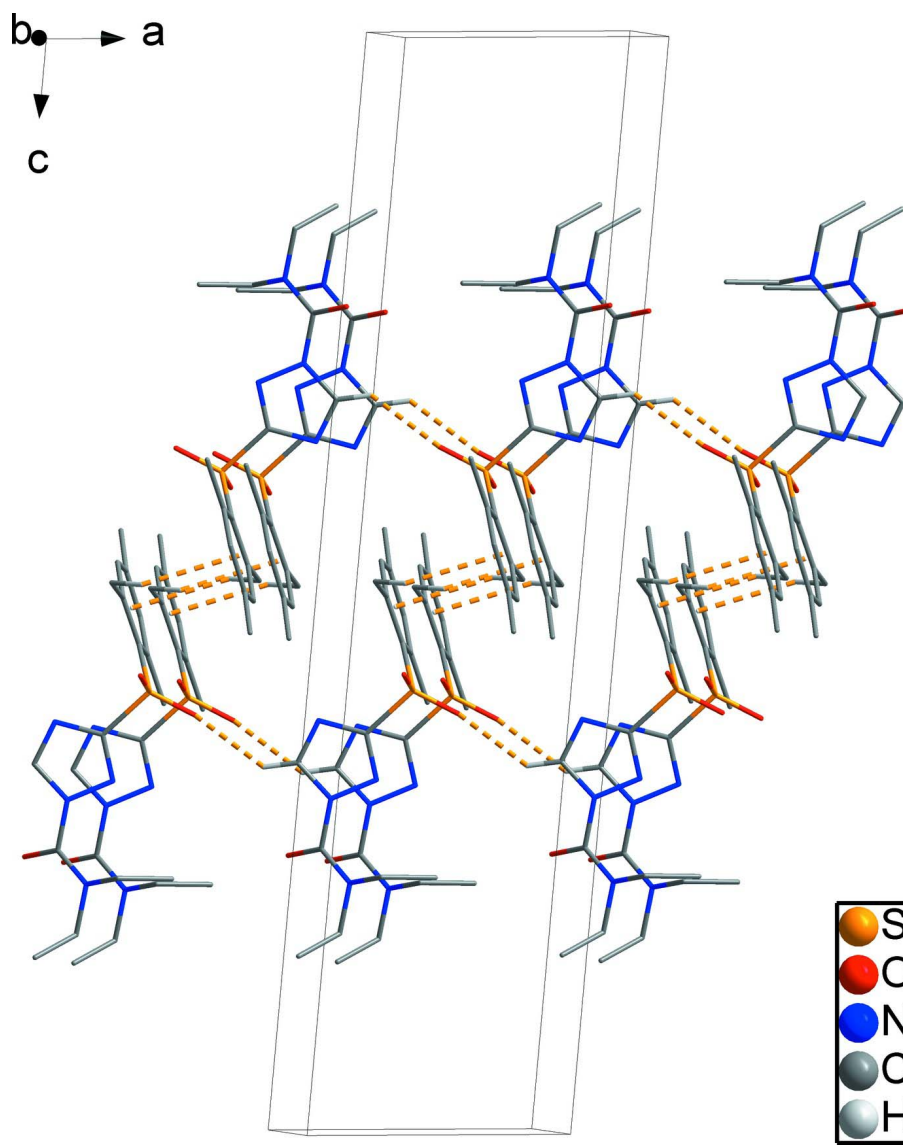


Figure 2

Crystal packing viewed along the b axis. The intermolecular interactions are shown as dashed lines.

N,N-Diethyl-3-mesitylsulfonyl-1*H*-1,2,4-triazole-1-carboxamide

Crystal data

$C_{16}H_{22}N_4O_3S$

$M_r = 350.43$

Monoclinic, $P2_1/c$

$a = 7.2800$ (3) Å

$b = 8.0410$ (4) Å

$c = 30.1792$ (13) Å

$\beta = 95.290$ (3)°

$V = 1759.12$ (14) Å³

$Z = 4$

$F(000) = 744$

$D_x = 1.323$ Mg m⁻³

Melting point: 390 K

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

Cell parameters from 3608 reflections

$\theta = 2.6$ – 25.8 °

$\mu = 0.21$ mm⁻¹

$T = 173$ K

Plate, colourless

$0.36 \times 0.30 \times 0.02$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2013)

$T_{\min} = 0.929$, $T_{\max} = 0.996$

11871 measured reflections

3385 independent reflections

2760 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 1.4^\circ$

$h = -8 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -37 \rightarrow 37$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.118$

$S = 1.12$

3385 reflections

222 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 1.5378P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.41370 (9)	0.41398 (9)	0.10060 (2)	0.03098 (19)
O1	0.3991 (3)	0.2440 (2)	0.08694 (6)	0.0440 (5)
O2	0.5862 (2)	0.4698 (3)	0.12191 (6)	0.0426 (5)
O3	-0.0331 (3)	0.6067 (3)	0.24940 (6)	0.0450 (5)
N1	0.0629 (3)	0.4287 (3)	0.12755 (6)	0.0321 (5)
N2	0.2960 (3)	0.4724 (3)	0.18146 (6)	0.0280 (5)
N3	0.1290 (3)	0.4783 (3)	0.19847 (6)	0.0262 (5)
N4	0.2305 (3)	0.4731 (3)	0.27538 (6)	0.0265 (5)
C1	0.3372 (3)	0.5499 (3)	0.05661 (7)	0.0243 (5)
C2	0.2701 (3)	0.4890 (3)	0.01447 (7)	0.0250 (5)
C3	0.2198 (3)	0.6048 (3)	-0.01847 (7)	0.0262 (5)
H3	0.1760	0.5657	-0.0472	0.031*
C4	0.2303 (3)	0.7740 (3)	-0.01148 (8)	0.0275 (6)
C5	0.2917 (3)	0.8308 (3)	0.03064 (8)	0.0293 (6)
H5	0.2964	0.9472	0.0360	0.035*
C6	0.3468 (3)	0.7227 (3)	0.06518 (7)	0.0272 (6)
C7	0.2494 (4)	0.3081 (3)	0.00170 (8)	0.0348 (6)
H7A	0.1922	0.2994	-0.0289	0.052*
H7B	0.1713	0.2521	0.0219	0.052*
H7C	0.3711	0.2552	0.0038	0.052*
C8	0.1789 (4)	0.8928 (4)	-0.04883 (8)	0.0389 (7)
H8A	0.2902	0.9482	-0.0574	0.058*

H8B	0.0931	0.9763	-0.0391	0.058*
H8C	0.1199	0.8317	-0.0744	0.058*
C9	0.4143 (4)	0.7991 (4)	0.10936 (8)	0.0456 (8)
H9A	0.5462	0.7758	0.1159	0.068*
H9B	0.3459	0.7514	0.1328	0.068*
H9C	0.3947	0.9196	0.1081	0.068*
C10	0.2465 (3)	0.4418 (3)	0.13928 (7)	0.0263 (5)
C11	-0.0062 (3)	0.4550 (3)	0.16559 (8)	0.0302 (6)
H11	-0.1343	0.4573	0.1694	0.036*
C12	0.1025 (3)	0.5235 (3)	0.24394 (8)	0.0293 (6)
C13	0.3683 (3)	0.3424 (3)	0.27089 (8)	0.0253 (5)
H13A	0.3367	0.2817	0.2427	0.030*
H13B	0.3635	0.2621	0.2956	0.030*
C14	0.5623 (3)	0.4096 (4)	0.27125 (9)	0.0364 (6)
H14A	0.5731	0.4752	0.2443	0.055*
H14B	0.6499	0.3169	0.2723	0.055*
H14C	0.5896	0.4803	0.2975	0.055*
C15	0.2014 (4)	0.5244 (4)	0.32107 (8)	0.0371 (7)
H15A	0.1512	0.6389	0.3205	0.045*
H15B	0.3214	0.5253	0.3394	0.045*
C16	0.0700 (4)	0.4095 (5)	0.34227 (9)	0.0528 (9)
H16A	-0.0498	0.4096	0.3245	0.079*
H16B	0.0542	0.4480	0.3725	0.079*
H16C	0.1204	0.2964	0.3435	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0289 (4)	0.0379 (4)	0.0261 (3)	0.0080 (3)	0.0024 (2)	0.0056 (3)
O1	0.0604 (13)	0.0328 (11)	0.0394 (10)	0.0158 (10)	0.0082 (9)	0.0063 (9)
O2	0.0237 (10)	0.0704 (15)	0.0327 (9)	0.0075 (10)	-0.0024 (7)	0.0098 (10)
O3	0.0377 (11)	0.0587 (14)	0.0388 (10)	0.0233 (10)	0.0043 (8)	0.0019 (10)
N1	0.0265 (11)	0.0399 (14)	0.0290 (10)	-0.0012 (10)	-0.0020 (9)	0.0017 (10)
N2	0.0220 (11)	0.0345 (12)	0.0275 (10)	0.0021 (9)	0.0020 (8)	0.0046 (10)
N3	0.0213 (11)	0.0306 (12)	0.0264 (10)	0.0019 (9)	0.0000 (8)	0.0035 (9)
N4	0.0290 (11)	0.0244 (11)	0.0251 (10)	0.0033 (9)	-0.0031 (8)	-0.0011 (9)
C1	0.0214 (12)	0.0284 (13)	0.0230 (11)	0.0013 (11)	0.0018 (9)	0.0029 (11)
C2	0.0173 (12)	0.0319 (14)	0.0261 (11)	-0.0003 (10)	0.0037 (9)	-0.0022 (11)
C3	0.0241 (12)	0.0326 (14)	0.0215 (11)	0.0007 (11)	0.0003 (9)	-0.0030 (11)
C4	0.0233 (13)	0.0316 (14)	0.0276 (12)	0.0018 (11)	0.0026 (10)	0.0034 (11)
C5	0.0321 (14)	0.0252 (13)	0.0309 (12)	-0.0004 (11)	0.0043 (11)	-0.0005 (11)
C6	0.0272 (13)	0.0318 (14)	0.0226 (11)	-0.0017 (11)	0.0016 (10)	-0.0004 (11)
C7	0.0390 (16)	0.0295 (15)	0.0353 (13)	0.0013 (13)	0.0002 (11)	-0.0050 (12)
C8	0.0479 (17)	0.0350 (16)	0.0328 (13)	0.0050 (14)	-0.0023 (12)	0.0057 (13)
C9	0.066 (2)	0.0384 (17)	0.0306 (14)	-0.0064 (16)	-0.0051 (13)	-0.0052 (13)
C10	0.0267 (13)	0.0270 (13)	0.0246 (11)	0.0018 (11)	-0.0011 (10)	0.0046 (11)
C11	0.0256 (13)	0.0338 (15)	0.0299 (12)	0.0007 (11)	-0.0034 (10)	0.0067 (12)
C12	0.0253 (14)	0.0319 (14)	0.0306 (13)	0.0029 (12)	0.0022 (10)	0.0014 (11)

C13	0.0276 (13)	0.0210 (12)	0.0265 (11)	0.0040 (10)	-0.0014 (10)	0.0047 (10)
C14	0.0278 (14)	0.0360 (15)	0.0441 (15)	-0.0003 (12)	-0.0026 (11)	0.0088 (13)
C15	0.0461 (17)	0.0354 (15)	0.0281 (13)	0.0066 (13)	-0.0057 (12)	-0.0099 (12)
C16	0.0481 (18)	0.083 (3)	0.0285 (13)	-0.0110 (18)	0.0087 (13)	-0.0122 (16)

Geometric parameters (Å, °)

S1—O1	1.429 (2)	C6—C9	1.509 (3)
S1—O2	1.4293 (19)	C7—H7A	0.9800
S1—C1	1.769 (2)	C7—H7B	0.9800
S1—C10	1.777 (2)	C7—H7C	0.9800
O3—C12	1.216 (3)	C8—H8A	0.9800
N1—C11	1.313 (3)	C8—H8B	0.9800
N1—C10	1.355 (3)	C8—H8C	0.9800
N2—C10	1.314 (3)	C9—H9A	0.9800
N2—N3	1.364 (3)	C9—H9B	0.9800
N3—C11	1.345 (3)	C9—H9C	0.9800
N3—C12	1.450 (3)	C11—H11	0.9500
N4—C12	1.331 (3)	C13—C14	1.511 (3)
N4—C13	1.468 (3)	C13—H13A	0.9900
N4—C15	1.473 (3)	C13—H13B	0.9900
C1—C2	1.408 (3)	C14—H14A	0.9800
C1—C6	1.414 (4)	C14—H14B	0.9800
C2—C3	1.387 (3)	C14—H14C	0.9800
C2—C7	1.509 (3)	C15—C16	1.513 (4)
C3—C4	1.378 (4)	C15—H15A	0.9900
C3—H3	0.9500	C15—H15B	0.9900
C4—C5	1.385 (3)	C16—H16A	0.9800
C4—C8	1.499 (3)	C16—H16B	0.9800
C5—C6	1.387 (3)	C16—H16C	0.9800
C5—H5	0.9500		
O1—S1—O2	118.01 (13)	H8A—C8—H8C	109.5
O1—S1—C1	111.37 (11)	H8B—C8—H8C	109.5
O2—S1—C1	110.25 (12)	C6—C9—H9A	109.5
O1—S1—C10	105.83 (12)	C6—C9—H9B	109.5
O2—S1—C10	106.94 (11)	H9A—C9—H9B	109.5
C1—S1—C10	103.17 (11)	C6—C9—H9C	109.5
C11—N1—C10	101.98 (19)	H9A—C9—H9C	109.5
C10—N2—N3	101.33 (18)	H9B—C9—H9C	109.5
C11—N3—N2	109.55 (19)	N2—C10—N1	116.4 (2)
C11—N3—C12	125.5 (2)	N2—C10—S1	121.10 (18)
N2—N3—C12	124.48 (19)	N1—C10—S1	122.51 (17)
C12—N4—C13	126.3 (2)	N1—C11—N3	110.7 (2)
C12—N4—C15	115.5 (2)	N1—C11—H11	124.6
C13—N4—C15	116.46 (19)	N3—C11—H11	124.6
C2—C1—C6	120.9 (2)	O3—C12—N4	126.5 (2)
C2—C1—S1	121.47 (19)	O3—C12—N3	116.5 (2)

C6—C1—S1	117.58 (17)	N4—C12—N3	116.9 (2)
C3—C2—C1	117.4 (2)	N4—C13—C14	112.9 (2)
C3—C2—C7	116.8 (2)	N4—C13—H13A	109.0
C1—C2—C7	125.8 (2)	C14—C13—H13A	109.0
C4—C3—C2	123.1 (2)	N4—C13—H13B	109.0
C4—C3—H3	118.4	C14—C13—H13B	109.0
C2—C3—H3	118.4	H13A—C13—H13B	107.8
C3—C4—C5	118.3 (2)	C13—C14—H14A	109.5
C3—C4—C8	120.5 (2)	C13—C14—H14B	109.5
C5—C4—C8	121.1 (2)	H14A—C14—H14B	109.5
C4—C5—C6	122.0 (2)	C13—C14—H14C	109.5
C4—C5—H5	119.0	H14A—C14—H14C	109.5
C6—C5—H5	119.0	H14B—C14—H14C	109.5
C5—C6—C1	118.2 (2)	N4—C15—C16	112.1 (2)
C5—C6—C9	117.2 (2)	N4—C15—H15A	109.2
C1—C6—C9	124.6 (2)	C16—C15—H15A	109.2
C2—C7—H7A	109.5	N4—C15—H15B	109.2
C2—C7—H7B	109.5	C16—C15—H15B	109.2
H7A—C7—H7B	109.5	H15A—C15—H15B	107.9
C2—C7—H7C	109.5	C15—C16—H16A	109.5
H7A—C7—H7C	109.5	C15—C16—H16B	109.5
H7B—C7—H7C	109.5	H16A—C16—H16B	109.5
C4—C8—H8A	109.5	C15—C16—H16C	109.5
C4—C8—H8B	109.5	H16A—C16—H16C	109.5
H8A—C8—H8B	109.5	H16B—C16—H16C	109.5
C4—C8—H8C	109.5		
C10—N2—N3—C11	-1.4 (3)	N3—N2—C10—S1	-177.87 (17)
C10—N2—N3—C12	-174.3 (2)	C11—N1—C10—N2	0.7 (3)
O1—S1—C1—C2	0.7 (2)	C11—N1—C10—S1	178.95 (19)
O2—S1—C1—C2	133.7 (2)	O1—S1—C10—N2	113.2 (2)
C10—S1—C1—C2	-112.4 (2)	O2—S1—C10—N2	-13.4 (2)
O1—S1—C1—C6	-178.97 (18)	C1—S1—C10—N2	-129.7 (2)
O2—S1—C1—C6	-46.0 (2)	O1—S1—C10—N1	-65.0 (2)
C10—S1—C1—C6	67.9 (2)	O2—S1—C10—N1	168.3 (2)
C6—C1—C2—C3	2.1 (3)	C1—S1—C10—N1	52.1 (2)
S1—C1—C2—C3	-177.58 (17)	C10—N1—C11—N3	-1.5 (3)
C6—C1—C2—C7	-178.5 (2)	N2—N3—C11—N1	2.0 (3)
S1—C1—C2—C7	1.8 (3)	C12—N3—C11—N1	174.8 (2)
C1—C2—C3—C4	-1.0 (4)	C13—N4—C12—O3	164.5 (3)
C7—C2—C3—C4	179.6 (2)	C15—N4—C12—O3	0.2 (4)
C2—C3—C4—C5	-0.9 (4)	C13—N4—C12—N3	-17.0 (4)
C2—C3—C4—C8	178.2 (2)	C15—N4—C12—N3	178.7 (2)
C3—C4—C5—C6	1.7 (4)	C11—N3—C12—O3	-29.9 (4)
C8—C4—C5—C6	-177.4 (2)	N2—N3—C12—O3	141.9 (3)
C4—C5—C6—C1	-0.6 (4)	C11—N3—C12—N4	151.5 (2)
C4—C5—C6—C9	178.9 (2)	N2—N3—C12—N4	-36.8 (3)
C2—C1—C6—C5	-1.3 (4)	C12—N4—C13—C14	110.4 (3)

S1—C1—C6—C5	178.35 (18)	C15—N4—C13—C14	-85.5 (3)
C2—C1—C6—C9	179.2 (2)	C12—N4—C15—C16	82.9 (3)
S1—C1—C6—C9	-1.2 (3)	C13—N4—C15—C16	-83.0 (3)
N3—N2—C10—N1	0.4 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C11—H11 \cdots O2 ⁱ	0.95	2.38	3.136 (3)	136
C7—H7C \cdots Cg1 ⁱⁱ	0.98	2.80	3.561 (3)	135

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z$.