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4-Cyclohexyl-1-(2-methoxybenzoyl)thiosemicarbazide with an unknown solvent

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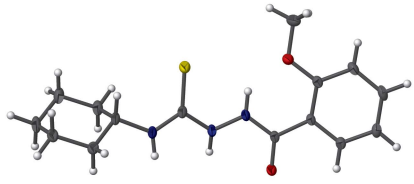
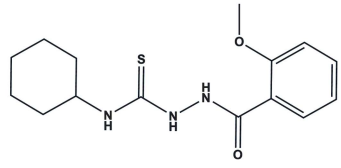
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{21}N_3O_2S$, a short intramolecular N—H···O hydrogen bond generates an $S(6)$ ring. The molecule is twisted with a dihedral angle between the benzene ring and the mean plane of the cyclohexyl ring being 58.90 (6)°. In the crystal, inversion dimers are formed with each molecule linked to the other by two N—H(H)···O hydrogen bonds to the same acceptor, generating $R_2^1(6)$ loops. A region of disordered electron density was corrected for using the SQUEEZE routine in *PLATON* [Spek (2015). *Acta* Cryst. C**71**, 9–18]. The given chemical formula and other crystal data do not take into account the unknown solvent molecule(s).

3D view



Chemical scheme



Structure description

Thiosemicarbazides are a class of organic compounds which are known not only for their various biological activities but also as metal-chelating agents (Siddiqui & Singh, 2003; Castiñeiras *et al.*, 2012; Singh *et al.*, 2014). Thiosemicarbazide is the simplest representative of such ligands having both nitrogen and sulfur atoms as donors. Thiosemicarbazide and substituted thiosemicarbazides are an important class of intermediates used for the synthesis of nitrogen–sulfur or nitrogen–oxygen heterocyclic compounds (Hovsepian *et al.*, 2004; Paswan *et al.*, 2015). Substituted thiosemicarbazides are biologically versatile compounds displaying a variety of biological effects (Bharti *et al.*, 2016; Plech *et al.*, 2011; Siwek *et al.*, 2011). The addition of hydrazides to various isothiocyanates is a convenient method for the synthesis of substituted thiosemicarbazides. As part of our studies in this area, we have synthesized the title compound and herein report on its crystal structure.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	0.88	1.90	2.5907 (12)	135
N2—H2A···O1^{i}	0.88	2.11	2.8523 (12)	142
N3—H3B···O1^{i}	0.88	2.04	2.8607 (12)	155

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

In the title compound, the dihedral angle of 58.90 (6)° between the benzene ring and the mean plane of the cyclohexyl ring indicates that the molecule is twisted (Fig. 1). A short intramolecular N—H···O hydrogen bond generates an $S(6)$ ring (Fig. 1, Table 1). The lengths of the C8═O1 [1.2426 (13) Å] and C9═S1 [1.6737 (11) Å] double bonds are in agreement with bond lengths in related compounds (Nath *et al.*, 2015; Dulare *et al.*, 2011). The C—N bond lengths, N1—C8 1.3321 (13), N2—C9 1.3611 (13) and N3—C10 1.4593 (14) Å, are similar to standard C—N single bonds.

In the crystal, inversion dimers are formed with each molecule linked to the other by two N—H(H)···O hydrogen bonds to the same acceptor, generating $R_2^1(6)$ loops (Fig. 2, Table 1). The overall crystal packing is illustrated in Fig. 3.

A region of disordered electron density was corrected for using the SQUEEZE routine in *PLATON* (Spek, 2015). The given chemical formula and other crystal data do not take into account the unknown solvent molecule(s). The region occupied by the disordered solvent is illustrated in Fig. 4, drawn using *Mercury* (Macrae *et al.*, 2008).

Synthesis and crystallization

The synthesis of the title compound is illustrated in Fig. 5. Methyl-2-methoxy benzoate (1.436 ml, 10 mmol) and hydrazine hydrate (0.485 ml, 10 mmol) were refluxed for 5 h and kept for overnight. The white solid 2-methoxy benzoic acid hydrazide was obtained upon cooling and filtered off, washed with water and and thereafter with ether. A mixture of 2-methoxy benzoic acid hydrazide (1.660 g, 10 mmol) and cyclohexyl isothiocyanate (1.417 ml, 10 mmol) in absolute ethanol (20 ml) was refluxed for 4 h. The solid obtained upon cooling was filtered off and washed with water and thereafter

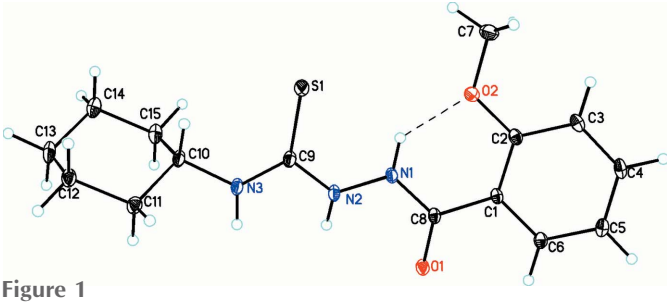


Figure 1
The molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 30% probability level. The intramolecular N—H···O hydrogen bond is shown as a dashed line (see Table 1).

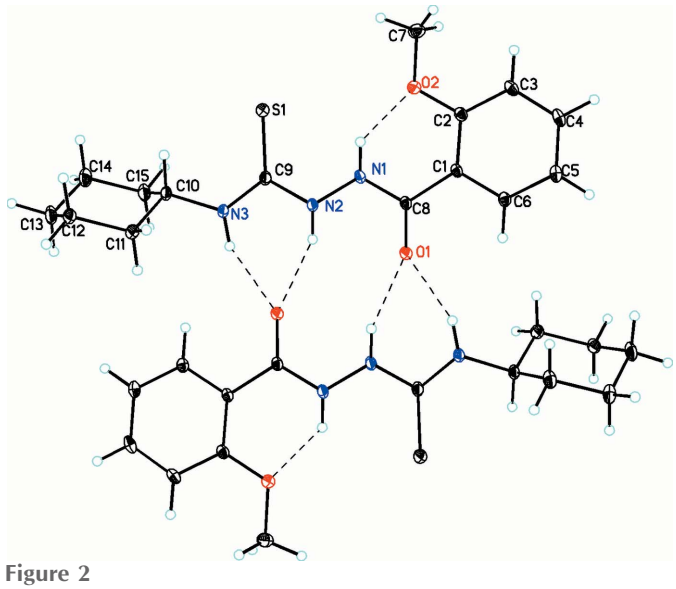


Figure 2
A view of the N—H···O hydrogen-bonding interactions (dashed lines; Table 1) involving the same acceptor atom, forming an inversion dimer. Unlabelled atoms are related to labelled ones by the symmetry operation $-x+1, -y+1, -z+1$.

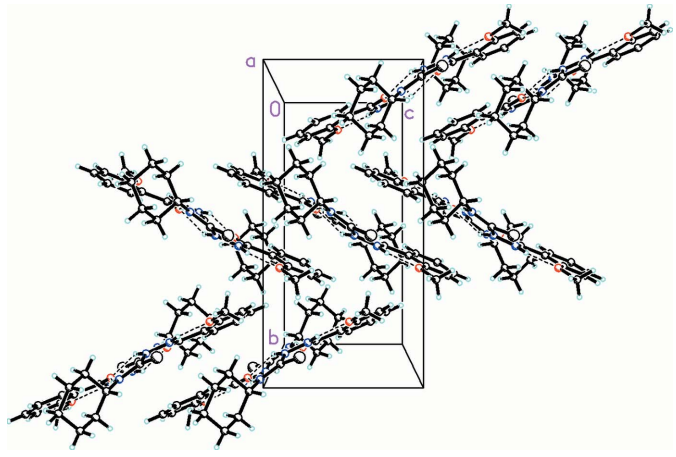


Figure 3
A view along the *a* axis of the crystal packing of the title compound, showing the N—H···O interactions as dashed lines (see Table 1).

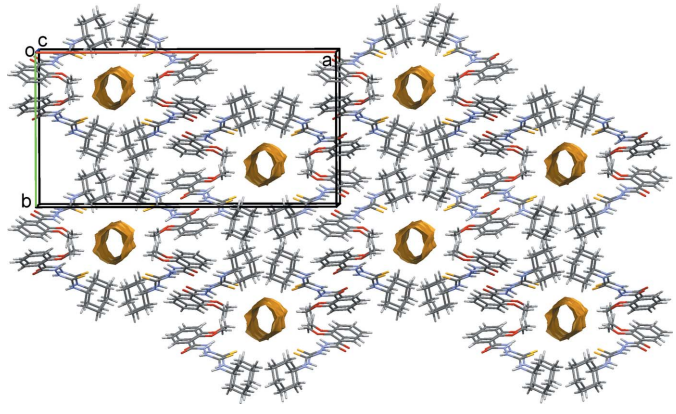


Figure 4
A view along the *c* axis of the crystal packing of the title compound, showing the region occupied by the disordered solvent (*Mercury*; Macrae *et al.*, 2008).

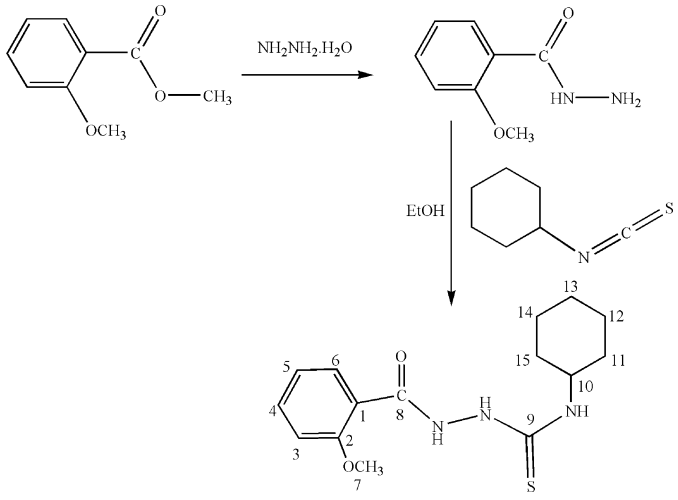


Figure 5
The reaction scheme showing the synthesis of the title compound.

with ether. The above solid was dissolved in methanol and kept for crystallization. Colorless crystals of the title compound were obtained after 10 days (yield: 60%, m.p. 468 K). Analysis calculated for $C_{15}H_{21}N_3O_2S$ (307.41): C, 58.55; H, 6.83; N, 13.66, S, 10.40%; Found. C, 58.05; H, 6.95; N, 13.15, S, 10.60%. IR (Selected, KBr): ν(NH) 3281, 3188; ν(C═O) 1633; ν(N—N) 1046; ν(C═S) 975 cm^{-1}. ^{1}H NMR (DMSO d_6; δ p.p.m.): 10.45, 9.94 (*s*, 2H, NH), 7.48–7.07 (*m*, 4H, aromatic), 3.81 (*s*, 3H, OCH_3), 3.27 (*s*, 1H, NH), 2.46–1.51 (cyclohexyl protons). ^{13}C NMR (DMSO-d_6; δ p.p.m.): 180.1 (C9), 163.0 (C8), 157.7 (C2), 133.9 (C4), 130.8 (C6), 122.3 (C5), 121.1 (C3), 112.9 (C1), 56.5 (C7), 49.0 (C10), 39.6 (C11, C15), 32.8 (C13), 25.1 (C12, C14).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. A region of disordered electron density was corrected for using the SQUEEZE routine in *PLATON* (Spek, 2015). The given chemical formula and other crystal data do not take into account the unknown solvent molecule(s).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{21}N_3O_2S$
M_r	307.41
Crystal system, space group	Orthorhombic, *Pccn*
Temperature (K)	100
a, *b*, *c* (Å)	29.5823 (10), 15.1971 (8), 7.4303 (2)
V (Å^3)	3340.4 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.20
Crystal size (mm)	0.35 × 0.31 × 0.18
Data collection	
Diffractometer	Rigaku OD SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.766, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23969, 8579, 5943
R_{int}	0.038
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.865
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.141, 1.01
No. of reflections	8579
No. of parameters	191
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.57, −0.49

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

IUCrData (2018). **3**, x180383 [https://doi.org/10.1107/S2414314618003838]

4-Cyclohexyl-1-(2-methoxybenzoyl)thiosemicarbazide with an unknown solvent

Rahul Chaurasia, Akhilesh Bharti, Ray J. Butcher, Jan L. Wikaira and Manoj K. Bharty

4-Cyclohexyl-1-(2-methoxybenzoyl)thiosemicarbazide

Crystal data

$C_{15}H_{21}N_3O_2S$
M_r = 307.41
Orthorhombic, *Pccn*
a = 29.5823 (10) Å
b = 15.1971 (8) Å
c = 7.4303 (2) Å
V = 3340.4 (2) Å^3
Z = 8
F(000) = 1312
D_x = 1.223 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5858 reflections
θ = 4.1–36.9°
μ = 0.20 mm^{-1}
T = 100 K
Block, colourless
0.35 × 0.31 × 0.18 mm

Data collection

Rigaku OD SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: micro-focus sealed X-ray tube
Detector resolution: 10.6501 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.766, T_{max} = 1.000
23969 measured reflections
8579 independent reflections
5943 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 38.0°, θ_{min} = 3.4°
h = −51→32
k = −22→25
l = −12→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.141
S = 1.00
8579 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.9308P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were included in calculated positions and refined as riding: (N—H = 0.88 Å, C—H = 0.95–1.00 Å with U_{iso}(H) = 1.5U_{eq}(*C*-methyl) and 1.2Ueq (C, N) for other H atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.35005 (2)	0.45941 (2)	0.30736 (4)	0.02840 (8)
O1	0.51910 (3)	0.44865 (6)	0.30165 (10)	0.02145 (16)
N1	0.44576 (3)	0.42049 (6)	0.25602 (12)	0.01785 (16)
H1A	0.4245	0.3969	0.1885	0.021*
N2	0.43510 (3)	0.45797 (6)	0.41971 (12)	0.01968 (17)
H2A	0.4555	0.4622	0.5055	0.024*
N3	0.38838 (3)	0.54063 (7)	0.58855 (13)	0.02328 (19)
H3B	0.4134	0.5557	0.6450	0.028*
C1	0.49867 (3)	0.38567 (6)	0.01910 (13)	0.01554 (16)
O2	0.42318 (3)	0.34449 (6)	−0.04284 (11)	0.02289 (16)
C2	0.46703 (4)	0.34759 (7)	−0.09866 (13)	0.01771 (18)
C3	0.48083 (4)	0.31472 (7)	−0.26464 (15)	0.0219 (2)
H3A	0.4594	0.2887	−0.3435	0.026*
C4	0.52583 (4)	0.31993 (7)	−0.31475 (14)	0.0236 (2)
H4A	0.5351	0.2970	−0.4278	0.028*
C5	0.55740 (4)	0.35814 (7)	−0.20190 (14)	0.0213 (2)
H5A	0.5882	0.3618	−0.2369	0.026*
C6	0.54345 (4)	0.39094 (7)	−0.03713 (13)	0.01772 (18)
H6A	0.5651	0.4178	0.0397	0.021*
C7	0.39000 (4)	0.30595 (9)	−0.15849 (19)	0.0305 (3)
H7A	0.3601	0.3105	−0.1022	0.046*
H7B	0.3974	0.2439	−0.1786	0.046*
H7C	0.3898	0.3371	−0.2740	0.046*
C8	0.48867 (4)	0.42057 (7)	0.20168 (13)	0.01617 (17)
C9	0.39228 (4)	0.48821 (8)	0.44523 (13)	0.01948 (19)
C10	0.34575 (4)	0.57477 (8)	0.65855 (14)	0.0207 (2)
H10A	0.3240	0.5806	0.5562	0.025*
C11	0.35395 (4)	0.66559 (8)	0.73803 (16)	0.0227 (2)
H11A	0.3648	0.7058	0.6425	0.027*
H11B	0.3777	0.6618	0.8316	0.027*
C12	0.31070 (4)	0.70242 (8)	0.82077 (15)	0.0240 (2)
H12A	0.3173	0.7597	0.8786	0.029*
H12B	0.2881	0.7126	0.7246	0.029*
C13	0.29112 (5)	0.63975 (9)	0.95964 (16)	0.0288 (3)
H13A	0.3124	0.6341	1.0617	0.035*
H13B	0.2624	0.6641	1.0065	0.035*
C14	0.28248 (5)	0.54955 (9)	0.87859 (18)	0.0294 (3)
H14A	0.2589	0.5542	0.7844	0.035*
H14B	0.2712	0.5092	0.9732	0.035*
C15	0.32559 (4)	0.51217 (8)	0.79683 (16)	0.0256 (2)
H15A	0.3479	0.5014	0.8935	0.031*
H15B	0.3188	0.4551	0.7385	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01858 (12)	0.04279 (19)	0.02381 (13)	0.00557 (12)	−0.00243 (10)	−0.01162 (12)
O1	0.0177 (3)	0.0308 (4)	0.0158 (3)	0.0055 (3)	−0.0021 (3)	−0.0051 (3)
N1	0.0182 (4)	0.0209 (4)	0.0145 (3)	0.0035 (3)	0.0016 (3)	−0.0039 (3)
N2	0.0175 (4)	0.0287 (5)	0.0129 (3)	0.0079 (3)	−0.0003 (3)	−0.0034 (3)
N3	0.0160 (4)	0.0367 (5)	0.0172 (4)	0.0089 (4)	−0.0009 (3)	−0.0080 (3)
C1	0.0191 (4)	0.0149 (4)	0.0126 (3)	0.0043 (3)	0.0003 (3)	−0.0005 (3)
O2	0.0183 (3)	0.0273 (4)	0.0230 (4)	0.0018 (3)	−0.0018 (3)	−0.0079 (3)
C2	0.0213 (4)	0.0153 (4)	0.0165 (4)	0.0035 (3)	−0.0003 (4)	−0.0019 (3)
C3	0.0293 (5)	0.0190 (4)	0.0174 (4)	0.0018 (4)	0.0001 (4)	−0.0053 (3)
C4	0.0339 (6)	0.0204 (5)	0.0165 (4)	0.0032 (4)	0.0066 (4)	−0.0036 (3)
C5	0.0237 (5)	0.0208 (5)	0.0194 (4)	0.0036 (4)	0.0066 (4)	0.0004 (4)
C6	0.0200 (4)	0.0171 (4)	0.0161 (4)	0.0036 (3)	0.0015 (3)	0.0005 (3)
C7	0.0238 (5)	0.0315 (6)	0.0363 (6)	0.0025 (5)	−0.0071 (5)	−0.0124 (5)
C8	0.0181 (4)	0.0175 (4)	0.0129 (3)	0.0054 (3)	0.0004 (3)	0.0000 (3)
C9	0.0177 (4)	0.0256 (5)	0.0152 (4)	0.0062 (4)	0.0010 (3)	−0.0006 (3)
C10	0.0171 (4)	0.0297 (5)	0.0154 (4)	0.0082 (4)	0.0012 (3)	−0.0031 (4)
C11	0.0184 (4)	0.0284 (5)	0.0214 (4)	0.0065 (4)	−0.0016 (4)	−0.0027 (4)
C12	0.0215 (5)	0.0292 (5)	0.0213 (4)	0.0098 (4)	−0.0024 (4)	−0.0048 (4)
C13	0.0298 (6)	0.0357 (6)	0.0208 (5)	0.0131 (5)	0.0065 (4)	−0.0040 (4)
C14	0.0274 (5)	0.0351 (6)	0.0258 (5)	0.0049 (5)	0.0102 (5)	−0.0011 (5)
C15	0.0275 (5)	0.0280 (5)	0.0214 (5)	0.0080 (5)	0.0049 (4)	−0.0013 (4)

Geometric parameters (Å, º)

S1—C9	1.6737 (11)	C6—H6A	0.9500
O1—C8	1.2426 (13)	C7—H7A	0.9800
N1—C8	1.3321 (13)	C7—H7B	0.9800
N1—N2	1.3795 (12)	C7—H7C	0.9800
N1—H1A	0.8800	C10—C11	1.5207 (17)
N2—C9	1.3611 (13)	C10—C15	1.5221 (17)
N2—H2A	0.8800	C10—H10A	1.0000
N3—C9	1.3349 (14)	C11—C12	1.5259 (15)
N3—C10	1.4593 (14)	C11—H11A	0.9900
N3—H3B	0.8800	C11—H11B	0.9900
C1—C6	1.3912 (15)	C12—C13	1.5190 (18)
C1—C2	1.4059 (14)	C12—H12A	0.9900
C1—C8	1.4864 (13)	C12—H12B	0.9900
O2—C2	1.3629 (13)	C13—C14	1.5189 (19)
O2—C7	1.4299 (14)	C13—H13A	0.9900
C2—C3	1.3918 (14)	C13—H13B	0.9900
C3—C4	1.3845 (17)	C14—C15	1.5225 (17)
C3—H3A	0.9500	C14—H14A	0.9900
C4—C5	1.3830 (17)	C14—H14B	0.9900
C4—H4A	0.9500	C15—H15A	0.9900
C5—C6	1.3848 (14)	C15—H15B	0.9900

C5—H5A	0.9500

C8—N1—N2	118.99 (9)	N3—C9—S1	125.44 (8)
C8—N1—H1A	120.5	N2—C9—S1	121.41 (8)
N2—N1—H1A	120.5	N3—C10—C11	108.87 (9)
C9—N2—N1	118.36 (9)	N3—C10—C15	110.91 (9)
C9—N2—H2A	120.8	C11—C10—C15	111.57 (9)
N1—N2—H2A	120.8	N3—C10—H10A	108.5
C9—N3—C10	124.83 (10)	C11—C10—H10A	108.5
C9—N3—H3B	117.6	C15—C10—H10A	108.5
C10—N3—H3B	117.6	C10—C11—C12	110.84 (10)
C6—C1—C2	118.08 (9)	C10—C11—H11A	109.5
C6—C1—C8	116.30 (9)	C12—C11—H11A	109.5
C2—C1—C8	125.62 (9)	C10—C11—H11B	109.5
C2—O2—C7	118.99 (9)	C12—C11—H11B	109.5
O2—C2—C3	122.44 (10)	H11A—C11—H11B	108.1
O2—C2—C1	117.30 (9)	C13—C12—C11	111.31 (10)
C3—C2—C1	120.26 (10)	C13—C12—H12A	109.4
C4—C3—C2	119.98 (10)	C11—C12—H12A	109.4
C4—C3—H3A	120.0	C13—C12—H12B	109.4
C2—C3—H3A	120.0	C11—C12—H12B	109.4
C5—C4—C3	120.68 (10)	H12A—C12—H12B	108.0
C5—C4—H4A	119.7	C14—C13—C12	111.14 (10)
C3—C4—H4A	119.7	C14—C13—H13A	109.4
C4—C5—C6	119.07 (11)	C12—C13—H13A	109.4
C4—C5—H5A	120.5	C14—C13—H13B	109.4
C6—C5—H5A	120.5	C12—C13—H13B	109.4
C5—C6—C1	121.91 (10)	H13A—C13—H13B	108.0
C5—C6—H6A	119.0	C13—C14—C15	110.73 (11)
C1—C6—H6A	119.0	C13—C14—H14A	109.5
O2—C7—H7A	109.5	C15—C14—H14A	109.5
O2—C7—H7B	109.5	C13—C14—H14B	109.5
H7A—C7—H7B	109.5	C15—C14—H14B	109.5
O2—C7—H7C	109.5	H14A—C14—H14B	108.1
H7A—C7—H7C	109.5	C10—C15—C14	111.37 (10)
H7B—C7—H7C	109.5	C10—C15—H15A	109.4
O1—C8—N1	120.62 (9)	C14—C15—H15A	109.4
O1—C8—C1	121.60 (9)	C10—C15—H15B	109.4
N1—C8—C1	117.78 (9)	C14—C15—H15B	109.4
N3—C9—N2	113.15 (9)	H15A—C15—H15B	108.0

C8—N1—N2—C9	155.21 (10)	C2—C1—C8—O1	175.77 (10)
C7—O2—C2—C3	0.13 (16)	C6—C1—C8—N1	176.75 (9)
C7—O2—C2—C1	−179.76 (10)	C2—C1—C8—N1	−3.91 (15)
C6—C1—C2—O2	−178.77 (9)	C10—N3—C9—N2	−173.24 (11)
C8—C1—C2—O2	1.90 (15)	C10—N3—C9—S1	6.15 (17)
C6—C1—C2—C3	1.33 (15)	N1—N2—C9—N3	−165.32 (10)
C8—C1—C2—C3	−178.00 (10)	N1—N2—C9—S1	15.26 (14)

O2—C2—C3—C4	179.74 (10)	C9—N3—C10—C11	−147.21 (11)
C1—C2—C3—C4	−0.37 (16)	C9—N3—C10—C15	89.66 (13)
C2—C3—C4—C5	−0.45 (17)	N3—C10—C11—C12	−177.45 (9)
C3—C4—C5—C6	0.27 (17)	C15—C10—C11—C12	−54.71 (12)
C4—C5—C6—C1	0.76 (16)	C10—C11—C12—C13	55.32 (13)
C2—C1—C6—C5	−1.54 (15)	C11—C12—C13—C14	−56.40 (14)
C8—C1—C6—C5	177.85 (9)	C12—C13—C14—C15	56.28 (14)
N2—N1—C8—O1	4.70 (15)	N3—C10—C15—C14	176.78 (10)
N2—N1—C8—C1	−175.62 (9)	C11—C10—C15—C14	55.22 (13)
C6—C1—C8—O1	−3.58 (14)	C13—C14—C15—C10	−55.65 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2	0.88	1.90	2.5907 (12)	135
N2—H2*A*···O1^{i}	0.88	2.11	2.8523 (12)	142
N3—H3*B*···O1^{i}	0.88	2.04	2.8607 (12)	155

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