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Crystal structure of 4-hydroxy-3-methoxybenzaldehyde 4-methylthiosemicarbazone methanol monosolvate

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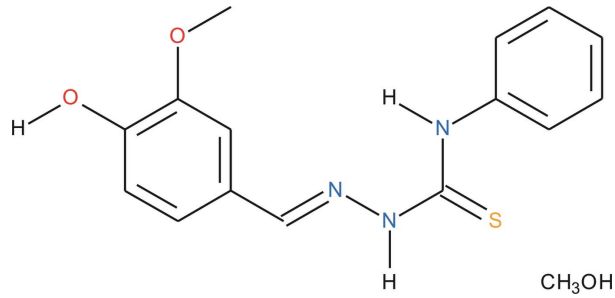
In the title solvate, $C_{15}H_{15}N_3O_2S\cdot CH_3OH$, the thiosemicarbazone molecule is approximately planar; the maximum deviation from the mean plane is 0.4659 (14) Å and the dihedral angle between the aromatic rings is 9.83 (8)°. This conformation is supported by an intramolecular N—H···N hydrogen bond. In the crystal, the thiosemicarbazone molecules are linked into dimers by pairs of N—H···S hydrogen bonds, thereby generating $R_2^2(8)$ loops. The methanol solvent molecule bonds to the thiosemicarbazone molecule through a bifurcated O—H···(O,O) hydrogen bond and also accepts an O—H···O link from the thiosemicarbazone molecule. Together, these links generate a three-dimensional network.

Keywords: crystal structure; bifurcated hydrogen bond; thiosemicarbazone derivative from natural product (vanillin).

CCDC reference: 1059141

1. Related literature

For one of the first reports of thiosemicarbazone derivatives synthesis, see: Freund & Schander (1902). For the report concerning the synthesis and crystal structure of 4-hydroxy-3-methoxybenzaldehyde 4-phenylthiosemicarbazone, see: Oliveira *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{15}H_{15}N_3O_2S\cdot CH_4O$
M_r = 333.40
Monoclinic, $P2_1/n$
a = 11.1833 (2) Å
b = 8.4207 (2) Å
c = 17.2521 (4) Å
β = 95.752 (1)°
V = 1616.47 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 123 K
0.29 × 0.15 × 0.09 mm

2.2. Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.924, T_{max} = 0.983
46461 measured reflections
3692 independent reflections
2926 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.05
3692 reflections
284 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H13···N2	0.866 (18)	2.082 (17)	2.5865 (16)	116.4 (14)
N1—H14···S1^{i}	0.896 (19)	2.530 (19)	3.4033 (13)	165.2 (15)
O1—H15···O3^{ii}	0.86 (2)	1.81 (2)	2.6562 (14)	167.7 (19)
O3—H19···O2	0.83 (2)	2.26 (2)	2.8853 (14)	132.1 (18)
O3—H19···O1	0.83 (2)	2.46 (2)	3.1645 (15)	144.2 (18)

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x-\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *WinGX* (Farrugia, 2012).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7402).

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supporting information

Acta Cryst. (2015). E**71**, o313–o314 [https://doi.org/10.1107/S2056989015007227]

Crystal structure of 4-hydroxy-3-methoxybenzaldehyde 4-methylthiosemicarbazone methanol monosolvate

Adriano Bof de Oliveira, Johannes Beck, Christian Landvogt and Bárbara Regina Santos Feitosa

S1. Structural commentary

Concerning our on-going research on the supramolecular chemistry of thiosemicarbazone derivatives from natural products, we report herein the synthesis and structure of the 4-hydroxy-3-methoxybenzaldehyde 4-phenylthiosemicarbazone methanol monosolvate, a thiosemicarbazone derivative from vanillin. The thiosemicarbazone group of the title compound doesn't match the ideal planarity and the maximum deviation from the mean plane of the non-H atoms concerning the thiosemicarbazone group amounts to 0.4659 (14) Å for C15 and the dihedral angle between the two aromatic rings amounts to 9.83 (8)° (Fig. 1). In the crystal, molecules are linked into dimers *via* pairs of N1—H14···S1 hydrogen bonds. The dimers are linked into a three dimensional hydrogen bonded network through the methanol molecules by the O1—H15···O3, O3—H19···O2 and O3—H19···O1 hydrogen interactions (Fig. 2). In addition, one intermolecular N3—H13···N2 hydrogen interaction is also observed (Table 1).

The crystal structure of the solvate free 4-hydroxy-3-methoxybenzaldehyde 4-phenylthiosemicarbazone is already published (Oliveira *et al.*, 2014) and the molecules are linked by N—H···S hydrogen interactions into dimers. Additionally, the dimers are linked by N—H···O and O—H···S hydrogen interactions building a three-dimensional hydrogen-bonded network.

In the actual structure, the presence of the methanol solvate molecules maintains the dimensionality of the network. As the outstanding feature, a bifurcated hydrogen bond is observed. The atom H19 of the hydroxy group of the methanol solvate builds a bifurcated hydrogen bond with the O1 and O2 atoms of the *ortho*-hydroxy-methoxy entity of the thiosemicarbazone derivative. The H19···O2 and H19···O1 distances amount to 2.26 (2) Å and 2.46 (2) Å (Fig. 1). As the difference between the lengths of the two hydrogen interactions is about 0.2 Å, the bifurcation is considered symmetric. Due to the hydrogen-bond interactions promoted by the solvate molecule, the supramolecularity of the structure modifies the arrangement molecules but the three-dimensional H-bonded network is preserved (Fig. 2).

S2. Synthesis and crystallization

Starting materials were commercially available and were used without further purification. The synthesis of the title compound, 4-hydroxy-3-methoxybenzaldehyde-4-methylthiosemicarbazone, was adapted from a previously procedure (Freund & Schander, 1902 and Oliveira *et al.*, 2014). Colourless blocks were obtained unexpectedly from a mixture containing uranyl acetate dihydrate and the title compound in methanol by the slow evaporation of the solvent.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in the Experimental part. All hydrogen atoms were localized in a difference density Fourier map. Their positions and isotropic displacement parameters were refined.

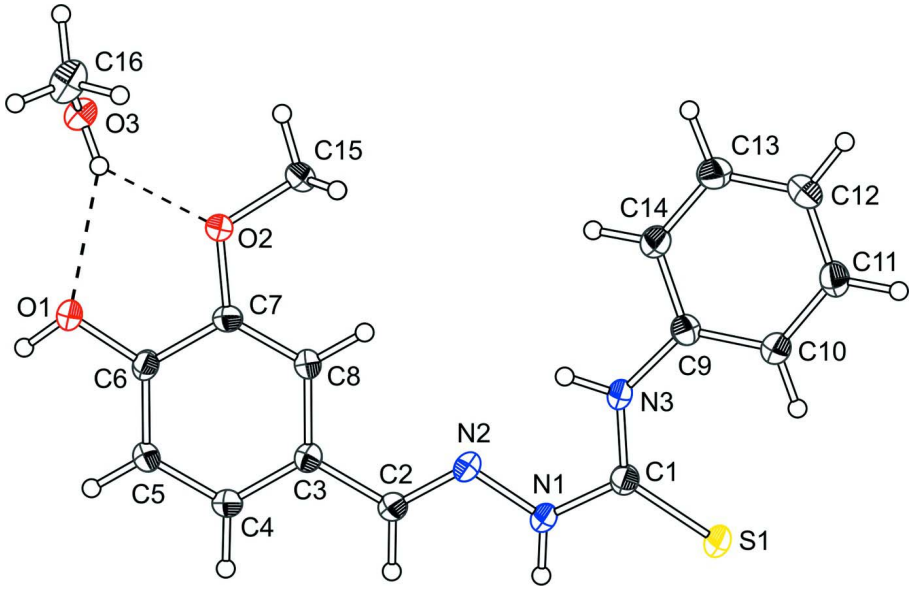

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn isotropically. The bifurcated hydrogen bonds are shown as dashed lines.

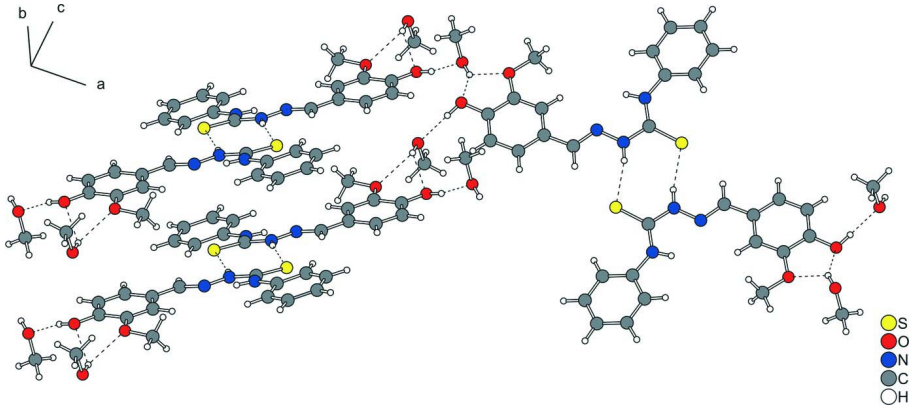

Figure 2

View of the hydrogen bonding in the structure of the title compound showing the three dimensional H-bonded network. Hydrogen bonding is shown as dashed lines.

4-Hydroxy-3-methoxybenzaldehyde 4-methylthiosemicarbazone methanol monosolvate

Crystal data

$C_{15}H_{15}N_3O_2S\cdot CH_4O$
$M_r = 333.40$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.1833\ (2)$ Å
$b = 8.4207\ (2)$ Å
$c = 17.2521\ (4)$ Å
$\beta = 95.752\ (1)°$
$V = 1616.47\ (6)$ Å^3
$Z = 4$

$F(000) = 704$
$D_x = 1.370$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 32712 reflections
$\theta = 2.9–27.5°$
$\mu = 0.22$ mm^{-1}
$T = 123$ K
Block, colorless
0.29 × 0.15 × 0.09 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube, Nonius KappaCCD
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.924, T_{max} = 0.983
46461 measured reflections
3692 independent reflections
2926 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −14→14
k = −10→10
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.05
3692 reflections
284 parameters
0 restraints
Primary atom site location: iterative
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.4527P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-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.

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}
S1	0.61038 (3)	0.16794 (5)	0.57257 (2)	0.03151 (12)
O1	−0.20192 (9)	0.56394 (12)	0.31751 (6)	0.0250 (2)
O2	−0.03515 (8)	0.71233 (11)	0.40440 (6)	0.0228 (2)
O3	−0.17481 (9)	0.93717 (13)	0.30693 (6)	0.0267 (2)
N1	0.39757 (10)	0.21003 (14)	0.49762 (7)	0.0211 (2)
N2	0.29562 (10)	0.29685 (13)	0.47526 (7)	0.0200 (2)
N3	0.44949 (11)	0.40722 (14)	0.58388 (7)	0.0239 (3)
C1	0.48140 (12)	0.26955 (16)	0.55234 (8)	0.0207 (3)
C2	0.21931 (12)	0.23390 (16)	0.42367 (8)	0.0203 (3)
C4	0.02092 (12)	0.24228 (16)	0.34674 (8)	0.0223 (3)
C5	−0.08460 (12)	0.32173 (16)	0.32039 (8)	0.0223 (3)
C3	0.10997 (11)	0.31895 (15)	0.39506 (8)	0.0191 (3)
C6	−0.10163 (11)	0.47803 (16)	0.34134 (8)	0.0197 (3)
C7	−0.01078 (12)	0.55645 (15)	0.38947 (8)	0.0192 (3)
C8	0.09313 (12)	0.47800 (16)	0.41617 (8)	0.0191 (3)
C9	0.51071 (12)	0.51201 (16)	0.63818 (8)	0.0212 (3)
C10	0.61312 (13)	0.47347 (18)	0.68703 (8)	0.0254 (3)

C11	0.66528 (13)	0.58702 (18)	0.73831 (8)	0.0266 (3)
C12	0.61602 (13)	0.73693 (18)	0.74239 (9)	0.0261 (3)
C13	0.51291 (13)	0.77472 (18)	0.69408 (9)	0.0276 (3)
C14	0.46044 (13)	0.66315 (17)	0.64255 (9)	0.0247 (3)
C15	0.05810 (13)	0.80047 (18)	0.44801 (9)	0.0254 (3)
C16	−0.24398 (16)	0.9612 (2)	0.37071 (10)	0.0355 (4)
H1	0.2343 (13)	0.1272 (19)	0.4021 (8)	0.021 (4)*
H2	0.0297 (14)	0.131 (2)	0.3319 (9)	0.028 (4)*
H3	−0.1460 (14)	0.269 (2)	0.2890 (9)	0.028 (4)*
H4	0.1569 (14)	0.5318 (19)	0.4486 (9)	0.026 (4)*
H5	0.6477 (14)	0.370 (2)	0.6856 (9)	0.030 (4)*
H6	0.7391 (15)	0.5623 (19)	0.7720 (9)	0.032 (4)*
H7	0.6504 (15)	0.818 (2)	0.7786 (10)	0.031 (4)*
H8	0.4727 (15)	0.883 (2)	0.6956 (9)	0.032 (4)*
H9	0.3903 (15)	0.688 (2)	0.6076 (10)	0.031 (4)*
H10	0.0725 (14)	0.757 (2)	0.5024 (10)	0.029 (4)*
H11	0.1347 (15)	0.7966 (19)	0.4238 (9)	0.027 (4)*
H12	0.0276 (15)	0.910 (2)	0.4506 (9)	0.033 (4)*
H13	0.3793 (16)	0.438 (2)	0.5638 (9)	0.030 (4)*
H14	0.4096 (15)	0.115 (2)	0.4769 (10)	0.035 (5)*
H15	−0.2449 (17)	0.513 (2)	0.2819 (11)	0.049 (6)*
H16	−0.2038 (19)	0.922 (3)	0.4223 (14)	0.067 (6)*
H17	−0.321 (2)	0.914 (3)	0.3632 (12)	0.064 (7)*
H18	−0.2597 (19)	1.079 (3)	0.3773 (12)	0.061 (6)*
H19	−0.1477 (18)	0.846 (3)	0.3092 (12)	0.052 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02158 (19)	0.0286 (2)	0.0418 (2)	0.00936 (14)	−0.00942 (15)	−0.01015 (16)
O1	0.0196 (5)	0.0227 (5)	0.0306 (6)	0.0036 (4)	−0.0071 (4)	−0.0031 (4)
O2	0.0194 (5)	0.0176 (5)	0.0300 (5)	0.0020 (4)	−0.0043 (4)	−0.0050 (4)
O3	0.0264 (5)	0.0231 (5)	0.0297 (6)	0.0045 (4)	−0.0013 (4)	0.0023 (4)
N1	0.0179 (6)	0.0204 (6)	0.0242 (6)	0.0045 (4)	−0.0020 (4)	−0.0017 (5)
N2	0.0173 (5)	0.0205 (6)	0.0220 (6)	0.0043 (4)	0.0000 (4)	0.0018 (5)
N3	0.0180 (6)	0.0229 (6)	0.0291 (6)	0.0048 (5)	−0.0061 (5)	−0.0042 (5)
C1	0.0190 (6)	0.0214 (7)	0.0214 (7)	0.0008 (5)	0.0008 (5)	0.0011 (5)
C2	0.0209 (7)	0.0190 (7)	0.0208 (7)	0.0014 (5)	0.0006 (5)	−0.0001 (5)
C4	0.0239 (7)	0.0177 (7)	0.0245 (7)	0.0011 (5)	−0.0010 (6)	−0.0016 (5)
C5	0.0212 (7)	0.0202 (7)	0.0240 (7)	−0.0013 (5)	−0.0043 (5)	−0.0015 (5)
C3	0.0184 (6)	0.0199 (7)	0.0187 (6)	0.0013 (5)	0.0005 (5)	0.0011 (5)
C6	0.0171 (6)	0.0210 (7)	0.0206 (7)	0.0019 (5)	−0.0006 (5)	0.0022 (5)
C7	0.0208 (6)	0.0158 (6)	0.0207 (7)	0.0002 (5)	0.0018 (5)	0.0000 (5)
C8	0.0177 (6)	0.0200 (7)	0.0191 (6)	−0.0003 (5)	−0.0005 (5)	−0.0005 (5)
C9	0.0199 (7)	0.0220 (7)	0.0216 (7)	−0.0013 (5)	0.0019 (5)	−0.0001 (5)
C10	0.0244 (7)	0.0237 (7)	0.0271 (7)	0.0033 (6)	−0.0029 (6)	−0.0011 (6)
C11	0.0235 (7)	0.0300 (8)	0.0251 (7)	−0.0009 (6)	−0.0028 (6)	−0.0010 (6)
C12	0.0264 (7)	0.0269 (7)	0.0255 (7)	−0.0059 (6)	0.0042 (6)	−0.0050 (6)

C13	0.0277 (7)	0.0238 (7)	0.0314 (8)	0.0022 (6)	0.0038 (6)	−0.0026 (6)
C14	0.0218 (7)	0.0251 (7)	0.0268 (7)	0.0038 (6)	0.0007 (6)	−0.0019 (6)
C15	0.0228 (7)	0.0218 (7)	0.0305 (8)	−0.0009 (6)	−0.0025 (6)	−0.0068 (6)
C16	0.0295 (9)	0.0420 (10)	0.0355 (9)	0.0070 (7)	0.0059 (7)	0.0097 (7)

Geometric parameters (Å, º)

S1—C1	1.6832 (13)	C3—C8	1.4055 (18)
O1—C6	1.3630 (15)	C6—C7	1.4102 (18)
O1—H15	0.86 (2)	C7—C8	1.3753 (18)
O2—C7	1.3703 (15)	C8—H4	0.973 (16)
O2—C15	1.4308 (16)	C9—C10	1.3906 (19)
O3—C16	1.421 (2)	C9—C14	1.3964 (19)
O3—H19	0.83 (2)	C10—C11	1.391 (2)
N1—C1	1.3579 (17)	C10—H5	0.952 (17)
N1—N2	1.3766 (15)	C11—C12	1.382 (2)
N1—H14	0.896 (19)	C11—H6	0.983 (17)
N2—C2	1.2843 (17)	C12—C13	1.390 (2)
N3—C1	1.3440 (18)	C12—H7	0.975 (17)
N3—C9	1.4130 (17)	C13—C14	1.383 (2)
N3—H13	0.866 (18)	C13—H8	1.020 (17)
C2—C3	1.4597 (18)	C14—H9	0.964 (17)
C2—H1	0.993 (16)	C15—H10	1.004 (17)
C4—C3	1.3918 (18)	C15—H11	0.991 (17)
C4—C5	1.3925 (19)	C15—H12	0.983 (18)
C4—H2	0.975 (17)	C16—H16	1.01 (2)
C5—C6	1.3831 (19)	C16—H17	0.95 (2)
C5—H3	0.941 (16)	C16—H18	1.01 (2)
C6—O1—H15	109.5 (13)	C7—C8—H4	121.0 (9)
C7—O2—C15	116.58 (10)	C3—C8—H4	118.9 (9)
C16—O3—H19	108.7 (15)	C10—C9—C14	119.45 (13)
C1—N1—N2	119.57 (11)	C10—C9—N3	124.81 (13)
C1—N1—H14	119.2 (11)	C14—C9—N3	115.72 (12)
N2—N1—H14	121.2 (11)	C11—C10—C9	119.51 (14)
C2—N2—N1	116.69 (11)	C11—C10—H5	119.9 (10)
C1—N3—C9	132.57 (12)	C9—C10—H5	120.6 (10)
C1—N3—H13	111.3 (11)	C12—C11—C10	121.10 (14)
C9—N3—H13	116.0 (11)	C12—C11—H6	118.6 (10)
N3—C1—N1	114.01 (12)	C10—C11—H6	120.3 (10)
N3—C1—S1	127.69 (10)	C11—C12—C13	119.31 (14)
N1—C1—S1	118.30 (10)	C11—C12—H7	122.3 (10)
N2—C2—C3	120.52 (12)	C13—C12—H7	118.4 (10)
N2—C2—H1	120.5 (8)	C14—C13—C12	120.19 (14)
C3—C2—H1	118.9 (8)	C14—C13—H8	117.8 (9)
C3—C4—C5	120.31 (13)	C12—C13—H8	122.0 (9)
C3—C4—H2	121.1 (9)	C13—C14—C9	120.44 (13)
C5—C4—H2	118.6 (9)	C13—C14—H9	121.3 (10)

C6—C5—C4	120.41 (12)	C9—C14—H9	118.3 (10)
C6—C5—H3	119.0 (10)	O2—C15—H10	110.1 (9)
C4—C5—H3	120.5 (10)	O2—C15—H11	112.1 (9)
C4—C3—C8	119.39 (12)	H10—C15—H11	108.6 (13)
C4—C3—C2	119.98 (12)	O2—C15—H12	105.8 (10)
C8—C3—C2	120.62 (12)	H10—C15—H12	108.6 (14)
O1—C6—C5	123.84 (12)	H11—C15—H12	111.6 (14)
O1—C6—C7	116.85 (12)	O3—C16—H16	114.0 (13)
C5—C6—C7	119.30 (12)	O3—C16—H17	113.2 (13)
O2—C7—C8	125.07 (12)	H16—C16—H17	107.7 (18)
O2—C7—C6	114.46 (11)	O3—C16—H18	109.9 (12)
C8—C7—C6	120.46 (12)	H16—C16—H18	106.6 (18)
C7—C8—C3	120.10 (12)	H17—C16—H18	104.8 (18)

C1—N1—N2—N2	0.00 (10)	O2—O2—C7—C6	0.0 (4)
C1—N1—N2—C2	−179.57 (12)	C15—O2—C7—C6	−175.37 (12)
N2—N1—N2—C2	0 (33)	O1—C6—C7—O2	−1.49 (17)
C9—N3—C1—N1	−175.91 (13)	O1—C6—C7—O2	−1.49 (17)
C9—N3—C1—S1	3.6 (2)	C5—C6—C7—O2	177.86 (12)
N2—N1—C1—N3	4.88 (18)	O1—C6—C7—O2	−1.49 (17)
N2—N1—C1—N3	4.88 (18)	O1—C6—C7—O2	−1.49 (17)
N2—N1—C1—S1	−174.68 (10)	C5—C6—C7—O2	177.86 (12)
N2—N1—C1—S1	−174.68 (10)	O1—C6—C7—C8	179.85 (12)
N1—N2—C2—N2	0 (78)	O1—C6—C7—C8	179.85 (12)
N2—N2—C2—C3	0.00 (13)	C5—C6—C7—C8	−0.8 (2)
N1—N2—C2—C3	−178.52 (11)	O2—C7—C8—C3	−177.97 (12)
C3—C4—C5—C6	0.6 (2)	O2—C7—C8—C3	−177.97 (12)
C5—C4—C3—C8	−0.9 (2)	C6—C7—C8—C3	0.5 (2)
C5—C4—C3—C2	178.81 (13)	C4—C3—C8—C7	0.3 (2)
N2—C2—C3—C4	−171.87 (13)	C2—C3—C8—C7	−179.37 (12)
N2—C2—C3—C4	−171.87 (13)	C1—N3—C9—C10	−17.0 (2)
N2—C2—C3—C8	7.8 (2)	C1—N3—C9—C14	164.40 (15)
N2—C2—C3—C8	7.8 (2)	C14—C9—C10—C11	−1.3 (2)
O1—O1—C6—C5	0.00 (5)	N3—C9—C10—C11	−179.84 (13)
O1—O1—C6—C7	0.00 (7)	C9—C10—C11—C12	1.0 (2)
C4—C5—C6—O1	179.54 (13)	C10—C11—C12—C13	−0.3 (2)
C4—C5—C6—O1	179.54 (13)	C11—C12—C13—C14	0.0 (2)
C4—C5—C6—C7	0.2 (2)	C12—C13—C14—C9	−0.4 (2)
C15—O2—C7—O2	0 (24)	C10—C9—C14—C13	1.0 (2)
O2—O2—C7—C8	0.0 (4)	N3—C9—C14—C13	179.68 (13)
C15—O2—C7—C8	3.22 (19)	C7—O2—C15—O2	0 (76)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H13···N2	0.866 (18)	2.082 (17)	2.5865 (16)	116.4 (14)
N1—H14···S1^{i}	0.896 (19)	2.530 (19)	3.4033 (13)	165.2 (15)
O1—H15···O3^{ii}	0.86 (2)	1.81 (2)	2.6562 (14)	167.7 (19)

O3—H19···O2	0.83 (2)	2.26 (2)	2.8853 (14)	132.1 (18)
O3—H19···O1	0.83 (2)	2.46 (2)	3.1645 (15)	144.2 (18)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*−1/2, *y*−1/2, −*z*+1/2.