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The crystal structure of 1-(2-hydroxy-5-methoxyphenyl)ethanone 4,4-dimethylthiosemicarbazone

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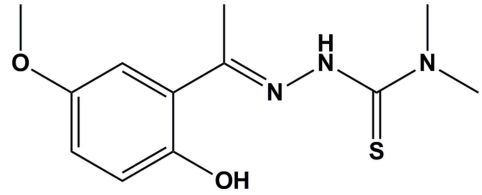
The asymmetric unit of the title compound, $C_{12}H_{17}N_3O_2S$, contains two independent molecules, *A* and *B*. Both molecules are nearly planar with the dihedral angle between the mean planes of the thioamide group and benzene ring being 7.5 (1)° in *A* and 4.3 (2)° in *B*. In each molecule, the hydroxy group participates in intramolecular O—H···N hydrogen bonding, while the amino H atom is not involved in hydrogen bonding because of the steric hinderence caused by two neighboring methyl groups. In the crystal, the individual molecules are linked by weak C—H···O hydrogen bonds, forming *A*–*A* and *B*–*B* inversion dimers. The dimers are linked *via* C—H···π interactions which help stabilize the packing.

Keywords: crystal structure; thiosemicarbazone; weak intermolecular interactions; C—H···π interactions.

CCDC reference: 1428535

1. Related literature

For thiosemicarbazone ligands and metal complexes, see: Lobana *et al.* (2009, 2012). For biological and antitumor and antifungal activity of palladium complexes with thiosemicarbazone ligands, see: Chellan *et al.* (2010). For biological activity of a thiosemicarbazone ligand with a terminal dimethyl substitution, see: Kowol *et al.* (2009). For related structures, see: Anderson *et al.* (2012, 2013); Kovala-Demertzi *et al.* (2000).



2. Experimental

2.1. Crystal data

$C_{12}H_{17}N_3O_2S$
M_r = 267.34
Monoclinic $P2_1/a$
a = 15.7097 (12) Å
b = 7.8300 (5) Å
c = 21.2351 (19) Å
β = 92.635 (8)°
V = 2609.3 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 173 K
0.54 × 0.35 × 0.05 mm

2.2. Data collection

Agilent, Eos, Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.803, T_{max} = 1.000
33509 measured reflections
8982 independent reflections
6065 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.224
S = 1.06
8982 reflections
334 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the C3···C8 and C3*A*···C8*A* rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.84	1.84	2.563 (2)	143
O1*A*—H1*A*···N3*A*	0.84	1.86	2.565 (3)	141
C11—H11*A*···O1^{i}	0.98	2.51	3.315 (3)	139
C11*A*—H11*E*···O1*A*^{ii}	0.98	2.68	3.305 (4)	122
C11*A*—H11*E*···*Cg*2^{iii}	0.98	2.73	3.590 (3)	147
C12—H12*B*···*Cg*1^{i}	0.98	2.82	3.530 (3)	130

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: CV5496).

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supporting information

Acta Cryst. (2015). E**71**, o811–o812 [https://doi.org/10.1107/S2056989015018228]

The crystal structure of 1-(2-hydroxy-5-methoxyphenyl)ethanone 4,4-dimethyl-thiosemicarbazone

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S1. Comment

The asymmetric unit of the title compound, $C_{12}H_{17}N_3O_2S$, contains two independent molecules *A* and *B*, respectively (Fig. 1). Both molecules are nearly planar with the dihedral angle between the mean planes of the thioamide group and benzene ring being 7.5 (1)° in *A* and 4.3 (2)° in *B*. In each molecule, the hydroxy group participates in intramolecular O—H···N hydrogen bonding, while the amino H atom is not involved in hydrogen bonding because of the steric hinderence caused by two neighboring methyl groups. In the crystal, weak intermolecular C—H···O and C—H···π (Table 1) interactions are observed which help stabilize the packing (Fig. 2). No π—π stacking interactions are present.

S2. Experimental

A 25 mL round bottom flask charged with 2.5 mL of H_2O, 2.5 mL ethanol and 0.1499 g (1.26 mmol) of 4,4-dimethyl-3-thiosemicarbazide was dissolved in a water/ethanol mixture and heated. Once the mixture was completely dissolved, 0.2225 g (1.34 mmol) of 2′-hydroxy-5′-methoxyacetophenone was added, and the solution was refluxed for 18 hours resulting in the formation of a yellow solid. After reflux, the slurry was allowed to cool to room temperature, transferred to a separatory funnel and water (15 mL) and dichloromethane (15 mL) was added. The organic layer was separated and the aqueous layer was extracted with an additional 15 mL of DCM. The organic layers were then combined and washed with brine (20 mL), and then dried with magnesium sulfate. The solvent was removed by rotary evaporation. The resulting solid was crystallized from acetonitrile to give 67 mg (18% yield) of yellow crystals. The crystals were observed to decompose above 460 K.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were located in difference maps. The C–H and N–H atoms were treated as riding atoms in geometrically idealized positions with C—H, N—H distances of 0.95 Å, 0.88 Å and refined with U_{iso}(H) = 1.2U_{eq}(C, N). The CH_3 and O–H atoms were also treated as riding atoms in geometrically idealized positions with the CH_3, O—H distances of 0.98 Å, 0.84 Å and refined with U_{iso}(H) = 1.5U_{eq}(C, O).

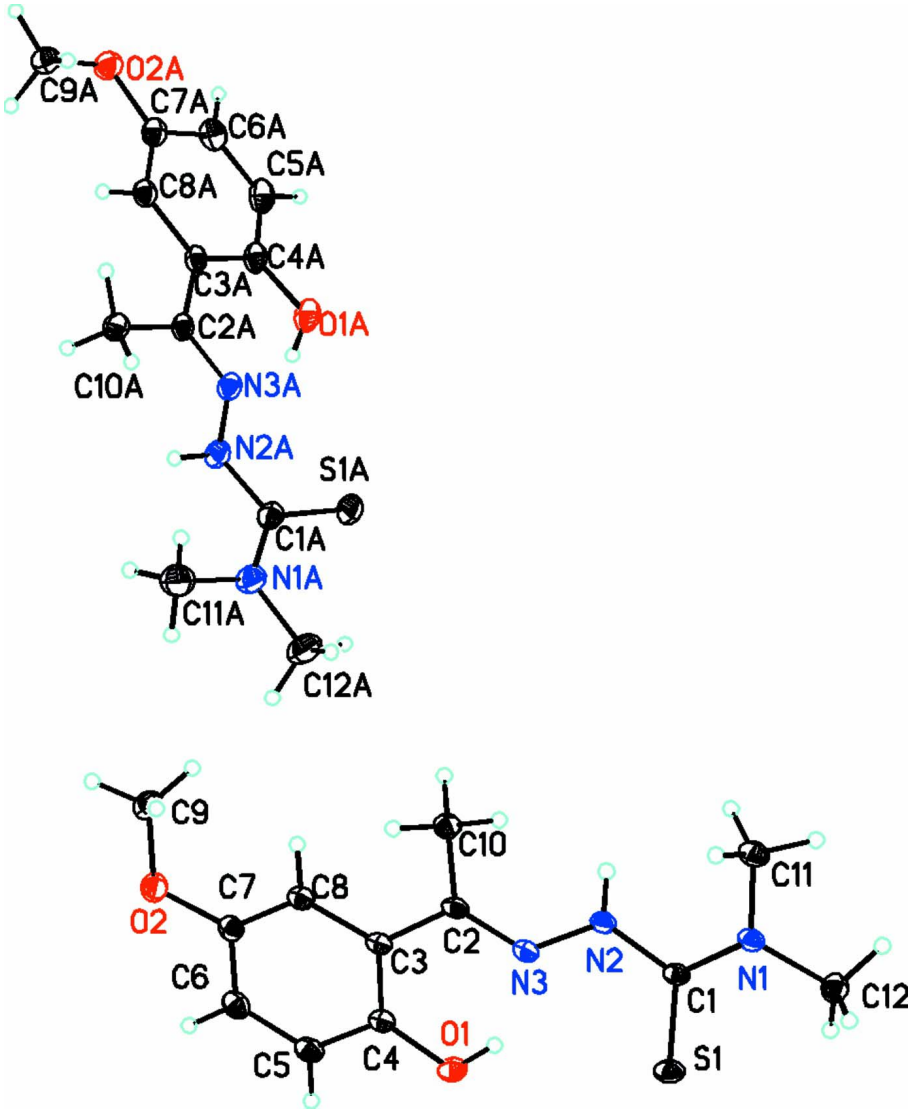

Figure 1

Two independent molecules of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

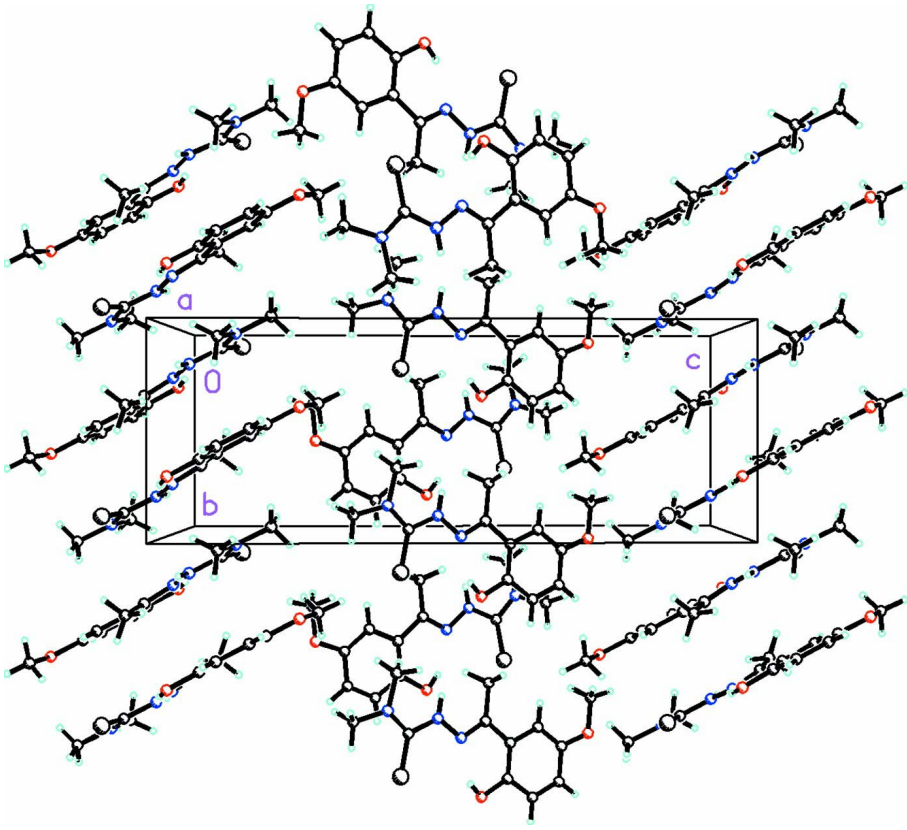

Figure 2

A portion of the crystal packing viewed approximately along the *a* axis.

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Crystal data

$C_{12}H_{17}N_3O_2S$
$M_r = 267.34$
Monoclinic, $P2_1/a$
$a = 15.7097$ (12) Å
$b = 7.8300$ (5) Å
$c = 21.2351$ (19) Å
$\beta = 92.635$ (8)°
$V = 2609.3$ (3) Å^3
$Z = 8$
$F(000) = 1136$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7195 reflections
$\theta = 3.0$–$32.8°$
$\mu = 0.25$ mm^{-1}
$T = 173$ K
Prism, colourless
0.54 × 0.35 × 0.05 mm

Data collection

Agilent, Eos, Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.803$, $T_{max} = 1.000$
33509 measured reflections
8982 independent reflections
6065 reflections with $I > 2\sigma(I)$
$R_{int} = 0.081$
$\theta_{max} = 32.9°$, $\theta_{min} = 3.0°$
$h = -23 \rightarrow 23$
$k = -11 \rightarrow 11$
$l = -30 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.224$
$S = 1.06$
8982 reflections
334 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1106P)^2 + 0.7177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.08$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro (Agilent, 2014). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.44809 (3)	0.09151 (7)	1.11976 (3)	0.03013 (15)
O1	0.32782 (10)	0.2857 (2)	0.99601 (7)	0.0293 (3)
H1	0.3742	0.2469	1.0112	0.044*
O2	0.35386 (11)	0.6134 (2)	0.76763 (7)	0.0336 (4)
N1	0.61624 (11)	0.0389 (2)	1.11969 (8)	0.0248 (3)
N2	0.55831 (11)	0.1799 (2)	1.03407 (8)	0.0246 (3)
H2	0.6099	0.1881	1.0199	0.030*
N3	0.48981 (11)	0.2446 (2)	1.00018 (8)	0.0233 (3)
C1	0.54532 (13)	0.1017 (2)	1.09064 (9)	0.0215 (4)
C2	0.50099 (12)	0.3329 (2)	0.94986 (9)	0.0220 (4)
C3	0.42248 (12)	0.3932 (2)	0.91666 (9)	0.0211 (4)
C4	0.34139 (13)	0.3677 (3)	0.94099 (9)	0.0236 (4)
C5	0.26923 (14)	0.4287 (3)	0.90750 (11)	0.0295 (4)
H5	0.2148	0.4145	0.9246	0.035*
C6	0.27513 (14)	0.5088 (3)	0.85053 (11)	0.0299 (4)
H6	0.2251	0.5482	0.8282	0.036*
C7	0.35447 (13)	0.5322 (3)	0.82530 (10)	0.0258 (4)
C8	0.42704 (13)	0.4770 (3)	0.85839 (9)	0.0247 (4)
H8	0.4811	0.4960	0.8414	0.030*
C9	0.43341 (17)	0.6297 (4)	0.73913 (11)	0.0405 (6)
H9A	0.4248	0.6843	0.6978	0.061*
H9B	0.4585	0.5162	0.7339	0.061*
H9C	0.4719	0.6997	0.7660	0.061*
C10	0.58601 (13)	0.3769 (3)	0.92477 (10)	0.0267 (4)
H10A	0.6306	0.3603	0.9580	0.040*
H10B	0.5858	0.4963	0.9110	0.040*
H10C	0.5971	0.3026	0.8889	0.040*

C11	0.70021 (13)	0.0579 (3)	1.09373 (10)	0.0292 (4)
H11A	0.7003	0.0029	1.0523	0.044*
H11B	0.7432	0.0041	1.1221	0.044*
H11C	0.7134	0.1795	1.0894	0.044*
C12	0.61403 (15)	−0.0437 (3)	1.18084 (10)	0.0302 (4)
H12A	0.6294	0.0392	1.2140	0.045*
H12B	0.6547	−0.1386	1.1828	0.045*
H12C	0.5565	−0.0873	1.1869	0.045*
S1A	0.56414 (5)	−0.19363 (10)	0.59613 (3)	0.0475 (2)
O1A	0.66714 (13)	−0.3267 (2)	0.44906 (9)	0.0443 (5)
H1A	0.6392	−0.2552	0.4692	0.066*
O2A	0.82584 (12)	−0.0500 (2)	0.24624 (8)	0.0411 (4)
N1A	0.53156 (14)	0.1371 (3)	0.61861 (10)	0.0413 (5)
N2A	0.59826 (13)	0.0826 (3)	0.52876 (9)	0.0357 (4)
H2A	0.5970	0.1935	0.5220	0.043*
N3A	0.63480 (12)	−0.0228 (3)	0.48627 (9)	0.0319 (4)
C1A	0.56361 (15)	0.0174 (4)	0.58155 (11)	0.0355 (5)
C2A	0.67559 (14)	0.0493 (3)	0.44166 (10)	0.0287 (4)
C3A	0.71116 (14)	−0.0682 (3)	0.39541 (10)	0.0280 (4)
C4A	0.70414 (16)	−0.2470 (3)	0.40023 (11)	0.0349 (5)
C5A	0.73558 (19)	−0.3507 (3)	0.35358 (12)	0.0421 (6)
H5A	0.7299	−0.4711	0.3568	0.050*
C6A	0.77470 (18)	−0.2826 (3)	0.30294 (12)	0.0406 (6)
H6A	0.7957	−0.3555	0.2714	0.049*
C7A	0.78354 (16)	−0.1063 (3)	0.29783 (11)	0.0334 (5)
C8A	0.75226 (15)	−0.0007 (3)	0.34303 (11)	0.0311 (4)
H8A	0.7584	0.1195	0.3390	0.037*
C9A	0.83626 (17)	0.1293 (4)	0.24074 (12)	0.0396 (5)
H9AA	0.8701	0.1720	0.2773	0.059*
H9AB	0.7802	0.1845	0.2389	0.059*
H9AC	0.8656	0.1550	0.2021	0.059*
C10A	0.68571 (17)	0.2386 (3)	0.43561 (12)	0.0357 (5)
H10D	0.6873	0.2908	0.4776	0.054*
H10E	0.6375	0.2851	0.4101	0.054*
H10F	0.7389	0.2637	0.4151	0.054*
C11A	0.5344 (2)	0.3188 (4)	0.60277 (14)	0.0511 (7)
H11D	0.5078	0.3852	0.6357	0.077*
H11E	0.5035	0.3382	0.5623	0.077*
H11F	0.5939	0.3547	0.5998	0.077*
C12A	0.4973 (2)	0.0922 (5)	0.67918 (14)	0.0571 (8)
H12D	0.5362	0.1325	0.7134	0.086*
H12E	0.4912	−0.0321	0.6820	0.086*
H12F	0.4414	0.1462	0.6828	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0221 (3)	0.0347 (3)	0.0344 (3)	−0.0020 (2)	0.01022 (19)	0.00048 (19)

O1	0.0209 (7)	0.0346 (9)	0.0330 (8)	−0.0050 (6)	0.0082 (6)	0.0030 (6)
O2	0.0299 (8)	0.0352 (9)	0.0358 (8)	−0.0002 (7)	0.0011 (6)	0.0072 (6)
N1	0.0191 (8)	0.0243 (8)	0.0313 (9)	0.0007 (6)	0.0058 (6)	0.0012 (6)
N2	0.0204 (8)	0.0245 (8)	0.0295 (8)	0.0024 (6)	0.0079 (6)	0.0015 (6)
N3	0.0206 (8)	0.0212 (8)	0.0284 (8)	0.0020 (6)	0.0043 (6)	−0.0025 (6)
C1	0.0209 (9)	0.0178 (8)	0.0263 (9)	−0.0008 (6)	0.0050 (7)	−0.0035 (6)
C2	0.0208 (9)	0.0179 (8)	0.0277 (9)	−0.0001 (7)	0.0067 (7)	−0.0049 (6)
C3	0.0178 (8)	0.0168 (8)	0.0290 (9)	−0.0024 (6)	0.0047 (7)	−0.0040 (6)
C4	0.0214 (9)	0.0196 (8)	0.0303 (10)	−0.0028 (7)	0.0053 (7)	−0.0035 (7)
C5	0.0184 (9)	0.0295 (10)	0.0410 (12)	−0.0021 (8)	0.0067 (8)	−0.0009 (8)
C6	0.0220 (10)	0.0278 (10)	0.0396 (12)	−0.0004 (8)	−0.0011 (8)	−0.0004 (8)
C7	0.0261 (10)	0.0195 (9)	0.0317 (10)	−0.0008 (7)	0.0020 (7)	−0.0004 (7)
C8	0.0219 (9)	0.0212 (9)	0.0314 (10)	−0.0013 (7)	0.0049 (7)	−0.0028 (7)
C9	0.0376 (13)	0.0515 (16)	0.0330 (12)	0.0026 (11)	0.0066 (9)	0.0089 (10)
C10	0.0200 (9)	0.0272 (10)	0.0333 (10)	−0.0021 (7)	0.0050 (7)	0.0037 (7)
C11	0.0197 (9)	0.0314 (11)	0.0370 (11)	−0.0006 (8)	0.0051 (8)	−0.0012 (8)
C12	0.0307 (11)	0.0297 (11)	0.0300 (10)	−0.0010 (9)	0.0009 (8)	0.0034 (8)
S1A	0.0435 (4)	0.0464 (4)	0.0531 (4)	−0.0001 (3)	0.0089 (3)	0.0232 (3)
O1A	0.0528 (12)	0.0284 (9)	0.0515 (11)	−0.0099 (8)	0.0010 (8)	0.0119 (7)
O2A	0.0469 (11)	0.0383 (10)	0.0388 (9)	0.0026 (8)	0.0079 (7)	0.0008 (7)
N1A	0.0350 (12)	0.0492 (13)	0.0402 (11)	−0.0032 (10)	0.0064 (9)	0.0068 (9)
N2A	0.0370 (11)	0.0329 (10)	0.0375 (11)	−0.0036 (8)	0.0050 (8)	0.0086 (7)
N3A	0.0310 (10)	0.0310 (10)	0.0336 (10)	−0.0049 (8)	0.0004 (7)	0.0070 (7)
C1A	0.0237 (11)	0.0454 (14)	0.0373 (12)	−0.0057 (9)	0.0001 (8)	0.0117 (9)
C2A	0.0254 (10)	0.0248 (10)	0.0352 (11)	−0.0042 (8)	−0.0036 (8)	0.0084 (8)
C3A	0.0257 (10)	0.0233 (9)	0.0343 (11)	−0.0039 (8)	−0.0053 (8)	0.0055 (7)
C4A	0.0356 (12)	0.0273 (11)	0.0408 (12)	−0.0071 (9)	−0.0075 (9)	0.0079 (9)
C5A	0.0529 (16)	0.0243 (11)	0.0480 (14)	−0.0046 (10)	−0.0079 (11)	0.0022 (9)
C6A	0.0466 (15)	0.0311 (12)	0.0432 (13)	0.0005 (10)	−0.0060 (11)	−0.0047 (9)
C7A	0.0342 (12)	0.0317 (12)	0.0338 (11)	−0.0026 (9)	−0.0048 (9)	0.0020 (8)
C8A	0.0297 (11)	0.0268 (10)	0.0364 (11)	−0.0018 (8)	−0.0026 (8)	0.0046 (8)
C9A	0.0350 (13)	0.0426 (14)	0.0416 (13)	−0.0040 (11)	0.0070 (10)	0.0037 (10)
C10A	0.0385 (13)	0.0264 (11)	0.0428 (13)	−0.0029 (9)	0.0083 (10)	0.0061 (9)
C11A	0.0501 (18)	0.0504 (18)	0.0536 (17)	−0.0002 (13)	0.0090 (13)	−0.0007 (12)
C12A	0.0508 (18)	0.079 (2)	0.0424 (16)	−0.0007 (16)	0.0133 (13)	0.0123 (14)

Geometric parameters (Å, º)

S1—C1	1.676 (2)	S1A—C1A	1.681 (3)
O1—H1	0.8400	O1A—H1A	0.8400
O1—C4	1.359 (2)	O1A—C4A	1.363 (3)
O2—C7	1.380 (3)	O2A—C7A	1.379 (3)
O2—C9	1.419 (3)	O2A—C9A	1.419 (3)
N1—C1	1.342 (3)	N1A—C1A	1.337 (4)
N1—C11	1.461 (3)	N1A—C11A	1.463 (4)
N1—C12	1.452 (3)	N1A—C12A	1.460 (3)
N2—H2	0.8800	N2A—H2A	0.8800
N2—N3	1.364 (2)	N2A—N3A	1.368 (3)

N2—C1	1.372 (2)	N2A—C1A	1.367 (3)
N3—C2	1.292 (3)	N3A—C2A	1.297 (3)
C2—C3	1.470 (3)	C2A—C3A	1.474 (3)
C2—C10	1.500 (3)	C2A—C10A	1.496 (3)
C3—C4	1.411 (3)	C3A—C4A	1.408 (3)
C3—C8	1.405 (3)	C3A—C8A	1.414 (3)
C4—C5	1.395 (3)	C4A—C5A	1.389 (4)
C5—H5	0.9500	C5A—H5A	0.9500
C5—C6	1.369 (3)	C5A—C6A	1.371 (4)
C6—H6	0.9500	C6A—H6A	0.9500
C6—C7	1.391 (3)	C6A—C7A	1.392 (3)
C7—C8	1.381 (3)	C7A—C8A	1.375 (3)
C8—H8	0.9500	C8A—H8A	0.9500
C9—H9A	0.9800	C9A—H9AA	0.9800
C9—H9B	0.9800	C9A—H9AB	0.9800
C9—H9C	0.9800	C9A—H9AC	0.9800
C10—H10A	0.9800	C10A—H10D	0.9800
C10—H10B	0.9800	C10A—H10E	0.9800
C10—H10C	0.9800	C10A—H10F	0.9800
C11—H11A	0.9800	C11A—H11D	0.9800
C11—H11B	0.9800	C11A—H11E	0.9800
C11—H11C	0.9800	C11A—H11F	0.9800
C12—H12A	0.9800	C12A—H12D	0.9800
C12—H12B	0.9800	C12A—H12E	0.9800
C12—H12C	0.9800	C12A—H12F	0.9800
C4—O1—H1	109.5	C4A—O1A—H1A	109.5
C7—O2—C9	116.72 (17)	C7A—O2A—C9A	116.31 (19)
C1—N1—C11	122.29 (17)	C1A—N1A—C11A	122.0 (2)
C1—N1—C12	121.38 (17)	C1A—N1A—C12A	121.0 (3)
C12—N1—C11	116.23 (17)	C12A—N1A—C11A	116.9 (3)
N3—N2—H2	120.6	N3A—N2A—H2A	119.6
N3—N2—C1	118.84 (17)	C1A—N2A—H2A	119.6
C1—N2—H2	120.6	C1A—N2A—N3A	120.7 (2)
C2—N3—N2	120.09 (17)	C2A—N3A—N2A	117.1 (2)
N1—C1—S1	124.37 (15)	N1A—C1A—S1A	125.40 (19)
N1—C1—N2	114.36 (17)	N1A—C1A—N2A	113.3 (2)
N2—C1—S1	121.27 (15)	N2A—C1A—S1A	121.3 (2)
N3—C2—C3	115.20 (17)	N3A—C2A—C3A	115.5 (2)
N3—C2—C10	125.01 (18)	N3A—C2A—C10A	123.5 (2)
C3—C2—C10	119.79 (17)	C3A—C2A—C10A	121.05 (19)
C4—C3—C2	122.05 (18)	C4A—C3A—C2A	122.6 (2)
C8—C3—C2	119.78 (17)	C4A—C3A—C8A	118.0 (2)
C8—C3—C4	118.16 (18)	C8A—C3A—C2A	119.43 (19)
O1—C4—C3	124.19 (18)	O1A—C4A—C3A	123.2 (2)
O1—C4—C5	116.38 (18)	O1A—C4A—C5A	116.9 (2)
C5—C4—C3	119.43 (19)	C5A—C4A—C3A	119.8 (2)
C4—C5—H5	119.3	C4A—C5A—H5A	119.4

C6—C5—C4	121.38 (19)	C6A—C5A—C4A	121.3 (2)
C6—C5—H5	119.3	C6A—C5A—H5A	119.4
C5—C6—H6	120.0	C5A—C6A—H6A	120.1
C5—C6—C7	119.9 (2)	C5A—C6A—C7A	119.8 (2)
C7—C6—H6	120.0	C7A—C6A—H6A	120.1
O2—C7—C6	115.65 (18)	O2A—C7A—C6A	115.6 (2)
O2—C7—C8	124.61 (19)	C8A—C7A—O2A	124.3 (2)
C8—C7—C6	119.74 (19)	C8A—C7A—C6A	120.1 (2)
C3—C8—H8	119.3	C3A—C8A—H8A	119.5
C7—C8—C3	121.33 (19)	C7A—C8A—C3A	121.0 (2)
C7—C8—H8	119.3	C7A—C8A—H8A	119.5
O2—C9—H9A	109.5	O2A—C9A—H9AA	109.5
O2—C9—H9B	109.5	O2A—C9A—H9AB	109.5
O2—C9—H9C	109.5	O2A—C9A—H9AC	109.5
H9A—C9—H9B	109.5	H9AA—C9A—H9AB	109.5
H9A—C9—H9C	109.5	H9AA—C9A—H9AC	109.5
H9B—C9—H9C	109.5	H9AB—C9A—H9AC	109.5
C2—C10—H10A	109.5	C2A—C10A—H10D	109.5
C2—C10—H10B	109.5	C2A—C10A—H10E	109.5
C2—C10—H10C	109.5	C2A—C10A—H10F	109.5
H10A—C10—H10B	109.5	H10D—C10A—H10E	109.5
H10A—C10—H10C	109.5	H10D—C10A—H10F	109.5
H10B—C10—H10C	109.5	H10E—C10A—H10F	109.5
N1—C11—H11A	109.5	N1A—C11A—H11D	109.5
N1—C11—H11B	109.5	N1A—C11A—H11E	109.5
N1—C11—H11C	109.5	N1A—C11A—H11F	109.5
H11A—C11—H11B	109.5	H11D—C11A—H11E	109.5
H11A—C11—H11C	109.5	H11D—C11A—H11F	109.5
H11B—C11—H11C	109.5	H11E—C11A—H11F	109.5
N1—C12—H12A	109.5	N1A—C12A—H12D	109.5
N1—C12—H12B	109.5	N1A—C12A—H12E	109.5
N1—C12—H12C	109.5	N1A—C12A—H12F	109.5
H12A—C12—H12B	109.5	H12D—C12A—H12E	109.5
H12A—C12—H12C	109.5	H12D—C12A—H12F	109.5
H12B—C12—H12C	109.5	H12E—C12A—H12F	109.5

O1—C4—C5—C6	−178.0 (2)	O1A—C4A—C5A—C6A	−179.3 (2)
O2—C7—C8—C3	−179.22 (18)	O2A—C7A—C8A—C3A	−178.6 (2)
N2—N3—C2—C3	178.91 (16)	N2A—N3A—C2A—C3A	−177.70 (18)
N2—N3—C2—C10	−1.7 (3)	N2A—N3A—C2A—C10A	1.3 (3)
N3—N2—C1—S1	−2.4 (2)	N3A—N2A—C1A—S1A	−0.3 (3)
N3—N2—C1—N1	178.56 (17)	N3A—N2A—C1A—N1A	179.2 (2)
N3—C2—C3—C4	4.8 (3)	N3A—C2A—C3A—C4A	−2.1 (3)
N3—C2—C3—C8	−174.23 (17)	N3A—C2A—C3A—C8A	176.3 (2)
C1—N2—N3—C2	173.41 (17)	C1A—N2A—N3A—C2A	−171.6 (2)
C2—C3—C4—O1	−0.5 (3)	C2A—C3A—C4A—O1A	−2.7 (3)
C2—C3—C4—C5	179.65 (18)	C2A—C3A—C4A—C5A	177.1 (2)
C2—C3—C8—C7	178.66 (18)	C2A—C3A—C8A—C7A	−177.7 (2)

C3—C4—C5—C6	1.9 (3)	C3A—C4A—C5A—C6A	0.9 (4)
C4—C3—C8—C7	−0.4 (3)	C4A—C3A—C8A—C7A	0.7 (3)
C4—C5—C6—C7	−0.7 (3)	C4A—C5A—C6A—C7A	0.1 (4)
C5—C6—C7—O2	179.7 (2)	C5A—C6A—C7A—O2A	178.3 (2)
C5—C6—C7—C8	−1.0 (3)	C5A—C6A—C7A—C8A	−0.8 (4)
C6—C7—C8—C3	1.6 (3)	C6A—C7A—C8A—C3A	0.3 (3)
C8—C3—C4—O1	178.59 (18)	C8A—C3A—C4A—O1A	178.9 (2)
C8—C3—C4—C5	−1.3 (3)	C8A—C3A—C4A—C5A	−1.3 (3)
C9—O2—C7—C6	−176.3 (2)	C9A—O2A—C7A—C6A	−179.3 (2)
C9—O2—C7—C8	4.5 (3)	C9A—O2A—C7A—C8A	−0.3 (3)
C10—C2—C3—C4	−174.64 (18)	C10A—C2A—C3A—C4A	178.9 (2)
C10—C2—C3—C8	6.3 (3)	C10A—C2A—C3A—C8A	−2.8 (3)
C11—N1—C1—S1	−177.10 (15)	C11A—N1A—C1A—S1A	179.4 (2)
C11—N1—C1—N2	1.9 (3)	C11A—N1A—C1A—N2A	0.0 (3)
C12—N1—C1—S1	−0.7 (3)	C12A—N1A—C1A—S1A	3.7 (4)
C12—N1—C1—N2	178.29 (18)	C12A—N1A—C1A—N2A	−175.7 (2)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C3···C8 and C3A···C8A rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.84	1.84	2.563 (2)	143
O1*A*—H1*A*···N3*A*	0.84	1.86	2.565 (3)	141
C11—H11*A*···O1^{i}	0.98	2.51	3.315 (3)	139
C11*A*—H11*E*···O1*A*^{ii}	0.98	2.68	3.305 (4)	122
C11*A*—H11*E*···*Cg*2^{iii}	0.98	2.73	3.590 (3)	147
C12—H12*B*···*Cg*1^{i}	0.98	2.82	3.530 (3)	130

Symmetry codes: (i) −*x*+1, −*y*, −*z*+2; (ii) −*x*+1, −*y*, −*z*+1; (iii) −*x*+3/2, *y*+1/2, −*z*+1.