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5,5-Diphenyl-2-thioxoimidazolidin-4-one dimethyl sulfoxide monosolvate

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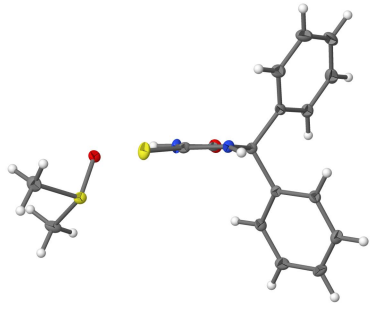
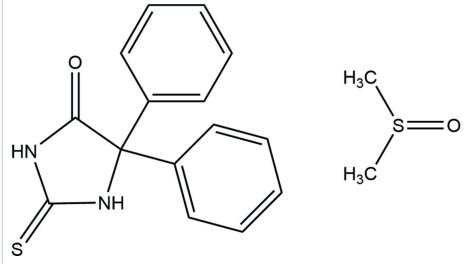
Structural data: full structural data are available from iucrdata.iucr.org

In the title solvate, $C_{15}H_{12}N_2OS{\cdot}C_2H_6OS$, the thioxoimidazolidin-4-one molecule and solvent molecule are linked by an N—H···O hydrogen bond. The planar imidazolidine ring (r.m.s. deviation = 0.022 Å) is inclined to the phenyl substituents in the 5-position by 69.57 (7) and 72.62 (6)°. In the crystal, N—H···O, C—H···O and C—H···S hydrogen bonds, together with C—H···π interactions, generate [100] chains, which stack along the *a*-axis direction.

3D view



Chemical scheme



Structure description

Thiohydantoins (2-thioxoimidazolidin-4-one derivatives) display a broad and potent biological profile and are found in anticonvulsant, antimetastatic, anti-angiogenic (Mudit *et al.*, 2009; Kumar *et al.*, 2009), antimicrobial (Kieć-Kononowicz & Szymańska, 2003; Khodair *et al.*, 2001) and anticancer drugs (Azizmohammadi *et al.*, 2013). As part of our studies in this area, we now present the synthesis and structural analysis of 5,5-diphenyl-2-thioxoimidazolidin-4-one dimethyl sulfoxide monosolvate. A search of the Cambridge Structural Database (Groom *et al.*, 2016) for related structures found 27 hits, including 2-thiohydantoin itself (Walker *et al.*, 1969), the unsolvated stucture of the molecule reported here (Roszak & Weaver, 1998) and the closely related 5-phenyl-2-thioxoimidazolidin-4-one (Ogawa *et al.*, 2007).

The asymmetric unit of the title compound consists of a thiohydantoin molecule with two phenyl substituents at the 5-position and a dimethyl sulfoxide solvent molecule. The molecules are linked by an N3—H3*N*···O1*S* hydrogen bond (Fig. 1 and Table 1). As expected, the imidazolidine ring is almost planar, with an r.m.s. deviation of 0.022 Å from the best-fit plane, with atoms S2 and O4 deviating by 0.138 (3) and −0.021 (3) Å, respectively, from that plane. The C51–C56 and C61–C66 phenyl rings are inclined to the imidazolidine ring plane by 69.57 (7) and 72.62 (6)°, respectively.

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

*Cg*3 is the centroid of the C61–C66 phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O1*S*	0.77 (2)	2.00 (2)	2.766 (2)	171 (2)
N1—H1*N*···O1*S*^{i}	0.86 (2)	1.95 (2)	2.8089 (19)	174 (2)
C66—H66···O4^{i}	0.95	2.62	3.341 (2)	133
C1*S*—H1*S*3···S2^{ii}	0.98	3.17	4.077 (2)	154
C1*S*—H1*S*2···O4^{iii}	0.98	2.58	3.393 (2)	141
C2*S*—H2*S*2···O4^{iii}	0.98	2.43	3.283 (2)	145
C2*S*—H2*S*1···S2^{ii}	0.98	2.93	3.874 (2)	162
C2*S*—HS23···*Cg*3^{ii}	0.98	2.98	3.887 (2)	154

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

In the crystal, N1—H1*N*···O1*S*^{i} and the already-mentioned N3—H3*N*···O1*S* hydrogen bonds (Table 1) link two DMSO solvent molecules to each thiohydantoin ring, with the former contact augmented by a C2*S*—H2*S*1···S2 hydrogen bonds and a weaker C2*S*—H2*S*3···*Cg*3 contact to the C61–C66 phenyl ring. In addition, O4 acts as a triple acceptor with C66—H66···O4^{i} hydrogen bonds linking adjacent main molecules, while C1*S*—H1*S*2···O4^{iii} and C2*S*—H2*S*2···O4^{iii} hydrogen bonds bind a third DMSO molecule to a thiohydantoin unit, enclosing an $R_2^2(6)$ ring (Fig. 2 and Table 1). The net effect of these contacts is to create independent chains of thiohydantoin and solvent molecules along *c* and these independent chains are stacked along the *a*-axis direction (Fig. 3).

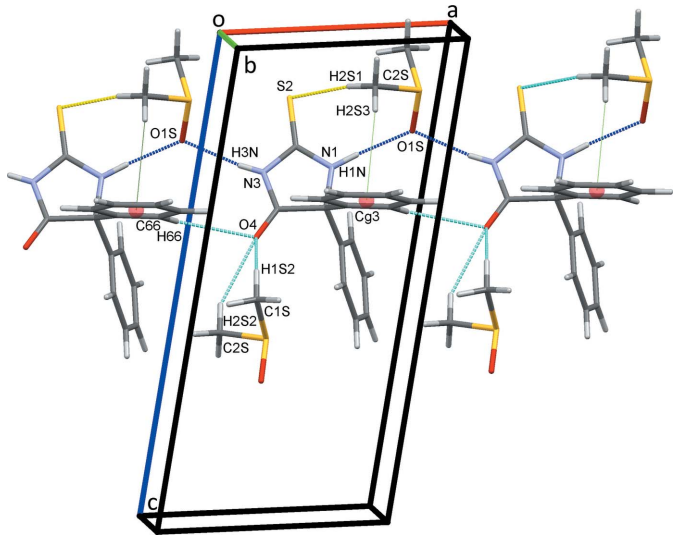


Figure 2
Rows of the thiohydantoin and DMSO solvent molecules formed along *a*. In Figs. 2 and 3, N—H···O, C—H···O and C—H···S hydrogen bonds are shown as dark-blue, light-blue and yellow dashed lines, respectively. C—H···π(ring) contacts are drawn as dotted green lines, with the ring centroids shown as red spheres.

Synthesis and crystallization

The synthesis of 5,5-diphenyl-2-thioxoimidazolidin-4-one was performed by a method reported in the literature (Ghanbari *et al.*, 2014) (Fig. 4). The resulting colourless solid was purified by recrystallization from DMSO solution to give colourless blocks (yield 90%). FT–IR (ATR cm^{-1}): 3251 (N—H amide, CO—NH—CS), 3156 (N—H, amide, CPh_2—NH—CS), 3022 (aromatic C—H stretch), 1746 (*s*, C=O amide), 1584, 1526, 1495 (Ar—C=C), 1226.10 (C=S).

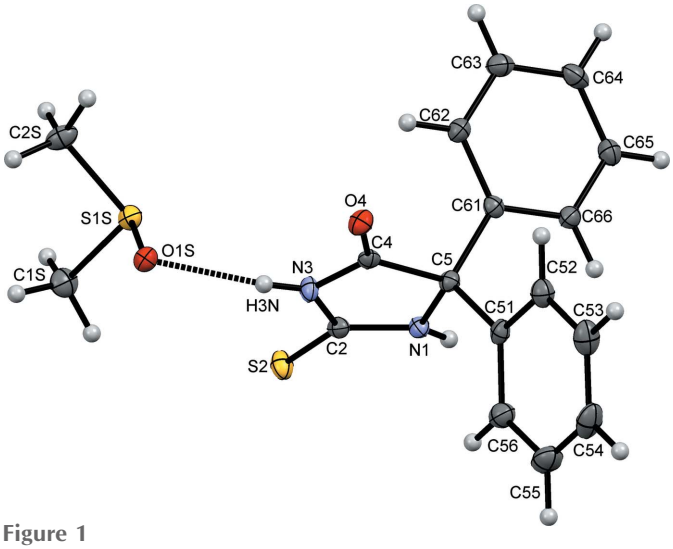


Figure 1
The asymmetric unit, showing 50% probability displacement ellipsoids. The N—H···O hydrogen bond is drawn as a dashed line.

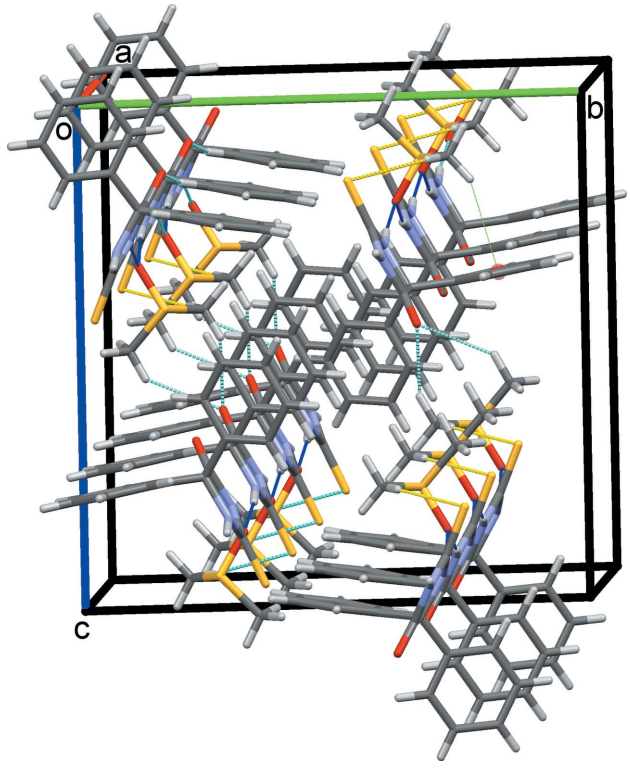


Figure 3
Overall packing, viewed along the *a*-axis direction. For clarity, only a single representative C—H···π(ring) contact is shown.

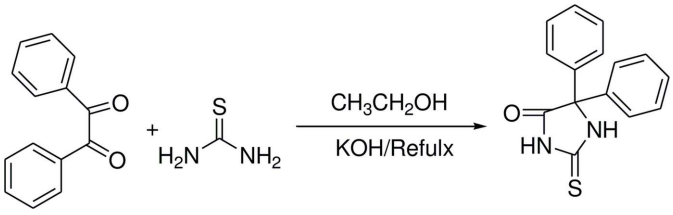


Figure 4
Synthesis of 5,5-diphenyl-2-thioxoimidazolidin-4-one

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{12}N_2OS{\cdot}C_2H_6OS$
M_r	346.45
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	7.3167 (2), 15.5894 (4), 15.5627 (3)
β (°)	101.820 (2)
V (Å^3)	1737.49 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.86
Crystal size (mm)	0.23 × 0.16 × 0.09
Data collection	
Diffractometer	Rigaku SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.932, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6995, 3444, 2966
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.624
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.093, 1.07
No. of reflections	3444
No. of parameters	216
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.29

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *TITAN* (Hunter & Simpson, 1999), *Mercury* (Macrae *et al.*, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009), *publCIF* (Westrip 2010) and *WinGX* (Farrugia, 2012).

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full crystallographic data

IUCrData (2018). **3**, x181010 [https://doi.org/10.1107/S2414314618010106]

5,5-Diphenyl-2-thioxoimidazolidin-4-one dimethyl sulfoxide monosolvate

Hamid Aziz, Aamer Saeed and Jim Simpson

5,5-Diphenyl-2-sulfanylideneimidazolidin-4-one dimethyl sulfoxide monosolvate

Crystal data

$C_{15}H_{12}N_2OS·C_2H_6OS$
M_r = 346.45
Monoclinic, $P2_1/n$
a = 7.3167 (2) Å
b = 15.5894 (4) Å
c = 15.5627 (3) Å
β = 101.820 (2)°
V = 1737.49 (7) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.324 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 4420 reflections
θ = 4.0–74.1°
μ = 2.86 mm^{-1}
T = 100 K
Block, colourless
0.23 × 0.16 × 0.09 mm

Data collection

Rigaku SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Detector resolution: 5.1725 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.932, T_{max} = 1.000
6995 measured reflections
3444 independent reflections
2966 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 74.3°, θ_{min} = 4.1°
h = −5→8
k = −19→17
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.093
S = 1.07
3444 reflections
216 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.8354P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.5315 (2)	0.63041 (10)	0.26867 (10)	0.0130 (3)
H1N	0.629 (3)	0.6288 (14)	0.2450 (14)	0.016*
C2	0.3597 (2)	0.61437 (12)	0.22220 (11)	0.0137 (3)
S2	0.30117 (6)	0.57231 (3)	0.12248 (3)	0.01927 (13)
N3	0.2341 (2)	0.64016 (10)	0.27276 (10)	0.0150 (3)
H3N	0.128 (3)	0.6354 (15)	0.2566 (15)	0.018*
C4	0.3192 (2)	0.67809 (11)	0.34936 (11)	0.0131 (4)
O4	0.24401 (17)	0.70732 (9)	0.40519 (8)	0.0172 (3)
C5	0.5311 (2)	0.67650 (12)	0.35075 (11)	0.0129 (4)
C51	0.6305 (2)	0.62745 (12)	0.43169 (11)	0.0144 (4)
C52	0.6650 (3)	0.66932 (13)	0.51249 (12)	0.0192 (4)
H52	0.631743	0.727945	0.515909	0.023*
C53	0.7479 (3)	0.62539 (15)	0.58791 (13)	0.0248 (4)
H53	0.769965	0.653945	0.642995	0.030*
C54	0.7987 (3)	0.54028 (15)	0.58349 (14)	0.0274 (5)
H54	0.856898	0.510665	0.635227	0.033*
C55	0.7642 (3)	0.49844 (14)	0.50326 (14)	0.0270 (5)
H55	0.798726	0.439965	0.500054	0.032*
C56	0.6792 (3)	0.54174 (13)	0.42730 (13)	0.0205 (4)
H56	0.654498	0.512635	0.372505	0.025*
C61	0.6099 (2)	0.76653 (12)	0.34460 (11)	0.0133 (4)
C62	0.4968 (3)	0.83874 (12)	0.32742 (12)	0.0164 (4)
H62	0.365790	0.833533	0.322920	0.020*
C63	0.5742 (3)	0.91861 (13)	0.31679 (12)	0.0202 (4)
H63	0.496298	0.967842	0.306047	0.024*
C64	0.7652 (3)	0.92639 (13)	0.32186 (13)	0.0211 (4)
H64	0.818091	0.980714	0.313805	0.025*
C65	0.8781 (3)	0.85458 (13)	0.33871 (12)	0.0194 (4)
H65	1.008674	0.859784	0.341895	0.023*
C66	0.8021 (3)	0.77493 (12)	0.35102 (11)	0.0158 (4)
H66	0.880976	0.726208	0.363810	0.019*
S1S	−0.14712 (6)	0.68634 (3)	0.11382 (3)	0.01591 (12)
O1S	−0.14346 (17)	0.63681 (8)	0.19917 (8)	0.0152 (3)
C1S	−0.2253 (3)	0.61145 (14)	0.02807 (12)	0.0223 (4)
H1S1	−0.132121	0.565890	0.030446	0.033*
H1S2	−0.242805	0.640771	−0.028726	0.033*
H1S3	−0.344131	0.586385	0.035218	0.033*
C2S	−0.3494 (3)	0.75316 (14)	0.09889 (13)	0.0223 (4)
H2S1	−0.460088	0.717679	0.098649	0.033*
H2S2	−0.363686	0.783686	0.042859	0.033*
H2S3	−0.335511	0.794801	0.146974	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0117 (7)	0.0138 (7)	0.0142 (7)	−0.0002 (6)	0.0041 (6)	−0.0017 (6)
C2	0.0133 (8)	0.0123 (8)	0.0166 (8)	−0.0001 (7)	0.0055 (7)	0.0008 (7)
S2	0.0159 (2)	0.0247 (3)	0.0172 (2)	−0.00250 (18)	0.00345 (17)	−0.00768 (18)
N3	0.0090 (7)	0.0193 (8)	0.0166 (7)	−0.0012 (6)	0.0027 (6)	−0.0030 (6)
C4	0.0135 (9)	0.0104 (8)	0.0151 (8)	0.0006 (7)	0.0024 (7)	0.0034 (7)
O4	0.0155 (6)	0.0212 (7)	0.0162 (6)	0.0026 (5)	0.0063 (5)	−0.0006 (5)
C5	0.0119 (8)	0.0132 (9)	0.0144 (8)	0.0007 (7)	0.0043 (6)	−0.0003 (7)
C51	0.0101 (8)	0.0181 (9)	0.0152 (8)	−0.0009 (7)	0.0031 (6)	0.0040 (7)
C52	0.0174 (9)	0.0224 (10)	0.0178 (9)	−0.0025 (8)	0.0034 (7)	0.0007 (8)
C53	0.0227 (10)	0.0337 (12)	0.0169 (9)	−0.0062 (9)	0.0019 (8)	0.0051 (8)
C54	0.0199 (10)	0.0348 (12)	0.0257 (10)	−0.0010 (9)	0.0003 (8)	0.0164 (9)
C55	0.0257 (11)	0.0206 (11)	0.0345 (12)	0.0025 (9)	0.0054 (9)	0.0099 (9)
C56	0.0190 (9)	0.0189 (10)	0.0238 (10)	0.0016 (8)	0.0050 (8)	0.0026 (8)
C61	0.0147 (8)	0.0141 (9)	0.0109 (8)	−0.0011 (7)	0.0021 (6)	−0.0012 (7)
C62	0.0144 (9)	0.0174 (9)	0.0171 (8)	0.0023 (7)	0.0026 (7)	0.0006 (7)
C63	0.0233 (10)	0.0154 (9)	0.0215 (9)	0.0029 (8)	0.0039 (8)	0.0005 (8)
C64	0.0267 (10)	0.0145 (9)	0.0214 (9)	−0.0061 (8)	0.0034 (8)	−0.0005 (7)
C65	0.0157 (9)	0.0199 (10)	0.0222 (9)	−0.0034 (8)	0.0033 (7)	−0.0004 (8)
C66	0.0151 (9)	0.0152 (9)	0.0161 (8)	0.0016 (7)	0.0014 (7)	−0.0004 (7)
S1S	0.0153 (2)	0.0171 (2)	0.0165 (2)	−0.00021 (17)	0.00602 (16)	0.00133 (17)
O1S	0.0147 (6)	0.0181 (7)	0.0134 (6)	0.0014 (5)	0.0039 (5)	0.0010 (5)
C1S	0.0261 (10)	0.0249 (10)	0.0153 (9)	0.0010 (9)	0.0027 (7)	−0.0024 (8)
C2S	0.0256 (10)	0.0213 (10)	0.0216 (9)	0.0071 (8)	0.0088 (8)	0.0063 (8)

Geometric parameters (Å, º)

N1—C2	1.339 (2)	C56—H56	0.9500
N1—C5	1.466 (2)	C61—C62	1.390 (3)
N1—H1N	0.86 (2)	C61—C66	1.396 (3)
C2—N3	1.387 (2)	C62—C63	1.392 (3)
C2—S2	1.6581 (18)	C62—H62	0.9500
N3—C4	1.362 (2)	C63—C64	1.389 (3)
N3—H3N	0.77 (2)	C63—H63	0.9500
C4—O4	1.209 (2)	C64—C65	1.384 (3)
C4—C5	1.546 (2)	C64—H64	0.9500
C5—C51	1.525 (2)	C65—C66	1.390 (3)
C5—C61	1.528 (2)	C65—H65	0.9500
C51—C56	1.388 (3)	C66—H66	0.9500
C51—C52	1.393 (3)	S1S—O1S	1.5318 (13)
C52—C53	1.387 (3)	S1S—C1S	1.777 (2)
C52—H52	0.9500	S1S—C2S	1.786 (2)
C53—C54	1.383 (3)	C1S—H1S1	0.9800
C53—H53	0.9500	C1S—H1S2	0.9800
C54—C55	1.385 (3)	C1S—H1S3	0.9800
C54—H54	0.9500	C2S—H2S1	0.9800

C55—C56	1.392 (3)	C2S—H2S2	0.9800
C55—H55	0.9500	C2S—H2S3	0.9800
C2—N1—C5	113.14 (15)	C55—C56—H56	120.0
C2—N1—H1N	121.8 (14)	C62—C61—C66	119.29 (17)
C5—N1—H1N	122.2 (15)	C62—C61—C5	122.59 (16)
N1—C2—N3	107.31 (15)	C66—C61—C5	117.99 (16)
N1—C2—S2	127.83 (14)	C61—C62—C63	120.45 (17)
N3—C2—S2	124.86 (14)	C61—C62—H62	119.8
C4—N3—C2	112.64 (16)	C63—C62—H62	119.8
C4—N3—H3N	125.3 (17)	C64—C63—C62	120.04 (18)
C2—N3—H3N	122.0 (17)	C64—C63—H63	120.0
O4—C4—N3	126.78 (17)	C62—C63—H63	120.0
O4—C4—C5	126.79 (16)	C65—C64—C63	119.65 (18)
N3—C4—C5	106.44 (14)	C65—C64—H64	120.2
N1—C5—C51	112.84 (14)	C63—C64—H64	120.2
N1—C5—C61	109.12 (14)	C64—C65—C66	120.58 (17)
C51—C5—C61	112.97 (15)	C64—C65—H65	119.7
N1—C5—C4	100.17 (14)	C66—C65—H65	119.7
C51—C5—C4	109.08 (14)	C65—C66—C61	119.98 (18)
C61—C5—C4	111.99 (15)	C65—C66—H66	120.0
C56—C51—C52	119.65 (17)	C61—C66—H66	120.0
C56—C51—C5	121.72 (16)	O1S—S1S—C1S	105.41 (9)
C52—C51—C5	118.56 (17)	O1S—S1S—C2S	105.88 (8)
C53—C52—C51	119.96 (19)	C1S—S1S—C2S	98.90 (10)
C53—C52—H52	120.0	S1S—C1S—H1S1	109.5
C51—C52—H52	120.0	S1S—C1S—H1S2	109.5
C54—C53—C52	120.5 (2)	H1S1—C1S—H1S2	109.5
C54—C53—H53	119.8	S1S—C1S—H1S3	109.5
C52—C53—H53	119.8	H1S1—C1S—H1S3	109.5
C53—C54—C55	119.69 (19)	H1S2—C1S—H1S3	109.5
C53—C54—H54	120.2	S1S—C2S—H2S1	109.5
C55—C54—H54	120.2	S1S—C2S—H2S2	109.5
C54—C55—C56	120.3 (2)	H2S1—C2S—H2S2	109.5
C54—C55—H55	119.9	S1S—C2S—H2S3	109.5
C56—C55—H55	119.9	H2S1—C2S—H2S3	109.5
C51—C56—C55	119.95 (19)	H2S2—C2S—H2S3	109.5
C51—C56—H56	120.0		
C5—N1—C2—N3	−5.9 (2)	C5—C51—C52—C53	177.23 (16)
C5—N1—C2—S2	173.76 (14)	C51—C52—C53—C54	0.7 (3)
N1—C2—N3—C4	4.0 (2)	C52—C53—C54—C55	−0.8 (3)
S2—C2—N3—C4	−175.60 (14)	C53—C54—C55—C56	0.1 (3)
C2—N3—C4—O4	179.16 (18)	C52—C51—C56—C55	−0.8 (3)
C2—N3—C4—C5	−0.7 (2)	C5—C51—C56—C55	−177.83 (17)
C2—N1—C5—C51	120.98 (16)	C54—C55—C56—C51	0.7 (3)
C2—N1—C5—C61	−112.57 (17)	N1—C5—C61—C62	101.61 (19)
C2—N1—C5—C4	5.14 (18)	C51—C5—C61—C62	−132.01 (17)

O4—C4—C5—N1	177.63 (18)	C4—C5—C61—C62	−8.4 (2)
N3—C4—C5—N1	−2.46 (17)	N1—C5—C61—C66	−74.20 (19)
O4—C4—C5—C51	59.0 (2)	C51—C5—C61—C66	52.2 (2)
N3—C4—C5—C51	−121.10 (16)	C4—C5—C61—C66	175.82 (15)
O4—C4—C5—C61	−66.8 (2)	C66—C61—C62—C63	0.0 (3)
N3—C4—C5—C61	113.10 (16)	C5—C61—C62—C63	−175.80 (16)
N1—C5—C51—C56	−12.6 (2)	C61—C62—C63—C64	1.1 (3)
C61—C5—C51—C56	−136.94 (17)	C62—C63—C64—C65	−0.9 (3)
C4—C5—C51—C56	97.82 (19)	C63—C64—C65—C66	−0.3 (3)
N1—C5—C51—C52	170.41 (15)	C64—C65—C66—C61	1.4 (3)
C61—C5—C51—C52	46.0 (2)	C62—C61—C66—C65	−1.2 (3)
C4—C5—C51—C52	−79.2 (2)	C5—C61—C66—C65	174.80 (16)
C56—C51—C52—C53	0.1 (3)		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C61–C66 phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O1*S*	0.77 (2)	2.00 (2)	2.766 (2)	171 (2)
N1—H1*N*···O1*S*^{i}	0.86 (2)	1.95 (2)	2.8089 (19)	174 (2)
C66—H66···O4^{i}	0.95	2.62	3.341 (2)	133
C1*S*—H1*S*3···S2^{ii}	0.98	3.17	4.077 (2)	154
C1*S*—H1*S*2···O4^{iii}	0.98	2.58	3.393 (2)	141
C2*S*—H2*S*2···O4^{iii}	0.98	2.43	3.283 (2)	145
C2*S*—H2*S*1···S2^{ii}	0.98	2.93	3.874 (2)	162
C2*S*—HS23···*Cg*3^{ii}	0.98	2.98	3.887 (2)	154

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