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5,7,12,14-Tetrahydro-5,14:7,12-bis([1,2]benzeno)-pentacene-6,13-diol dimethylformamide disolvate

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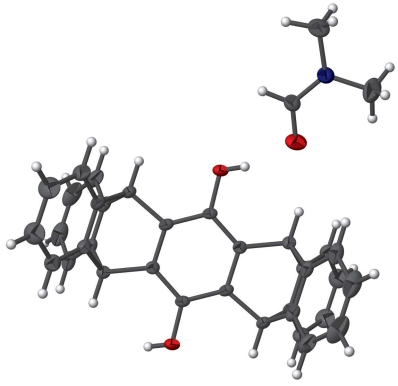
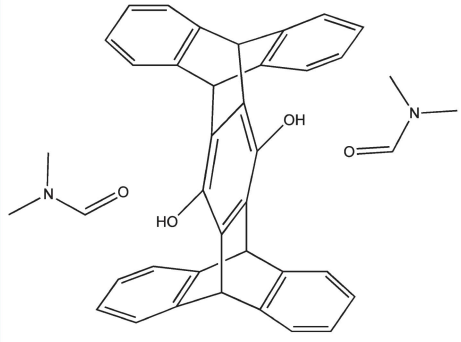
The crystal lattice of the title compound, $C_{34}H_{22}O_2\cdot 2C_3H_7NO$, at 173 K has monoclinic ($P2_1/n$) symmetry. Molecules are located on crystallographic centers of symmetry and have approximate non-crystallographic *mmm* symmetry, indicating that in solution the chemical and spectroscopic behavior would be that of a D_{2h} molecule. The compound has applications in gas-separation membranes fabricated from polymers of intrinsic microporosity (PIM). The compound is the product of reduction of the corresponding quinone by $Na_2S_2O_4$ in DMF/$NaHCO_3$.

3D view



Chemical scheme



Structure description

Pentiptycenes are a member of the iptycene family that possess a rigid, bulky, aromatic, π-electron-rich three-dimensional structure. In addition to various applications of pentiptycene in fluorescence and chemical sensing (Yang and Swager, 1998) and a light-driven molecular brake (Sun *et al.*, 2010), pentiptycene-6,13-diol is currently used as a principal reactant for preparing polymers. Gong & Zhang (2011) synthesized poly(arylene ether sulfone)s to fabricate highly conductive polymer electrolyte membranes for high temperature and low humidity conditions. Luo *et al.* (2015, 2016) have reported pentiptycene-based diamines for the preparation of polyimides with controlled molecular cavities appropriate for gas separation with PIM membranes.

In the title compound, $C_{34}H_{22}O_2\cdot 2C_3H_7NO$, an inversion center ($-x$, $-y$, $-z$) is present between atoms C1, C2 and C3 of the central hydroquinone ring, which yields a $C_{22}H_{22}N_2O$ substructure and a solvent molecule, $C_2H_3O_2$, in the asymmetric unit, generating a rigid tweezer-like molecule (Fig. 1). The dihedral angle between the terminal

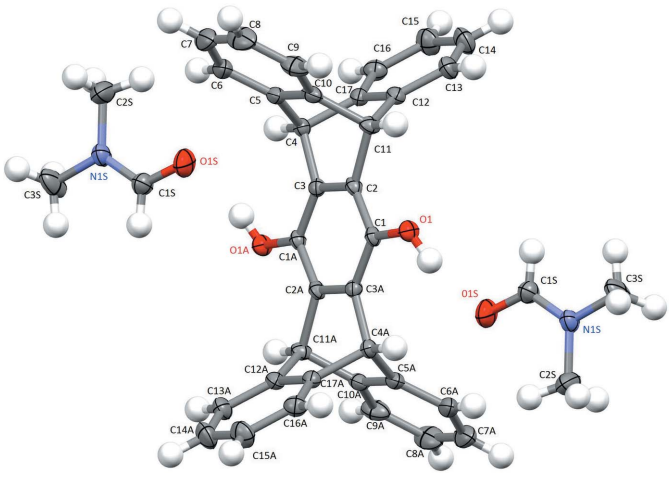


Figure 1
The title compound, showing its relationship to the two DMF solvent molecules. Displacement ellipsoids are drawn at the 50% probability level. H atoms are rendered as spheres of arbitrary radius. The complementary atoms are generated by the symmetry operation $(-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2})$.

benzene rings in each of the two symmetry-related sets is 64.9 (9)°. The three six-membered carbon rings fused between the benzene rings and the central hydroquinone ring for both symmetry sets adopt a boat conformation (puckering parameters Q, θ, and φ = 0.7976 (15) Å, 90.3 (11)°, 299.85 (11)° for C2/C3/C4/C5/C10/C11; 0.8113 (15) Å, 90.36 (11)°, 120.38° for C2/C3/C4/C17/C12/C11; and 0.8238 (15) Å, 89.91 (10), 0.20 (11) for C4/C5/C10C11/C12/C17, respectively. Bond lengths are within normal ranges.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1S	0.84	1.86	2.6818 (16)	164
C6—H6···O1^{i}	0.95	2.70	3.4912 (19)	141
C11—H11···O1S	1.00	2.42	3.2771 (18)	143

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

In the crystal, two DMF molecules solvate the compound with O—H···O hydrogen bonds and weak C—H···O intermolecular interactions (Table 1). On each half of the title compound these interactions involve the oxygen atom from DMF interacting with the OH group and a CH unit from a boat-formed six-membered carbon ring of the compound (Fig. 2). In the lattice, the central symmetry-related hydroquinone rings are aligned along the a axis, as two stack orientations, with an angle of 56 (1)° between them (Fig 3).

Some derivatives of this compound for which X-ray structures have previously been reported include bis(trimethylsilylethynyl)pentiptycene (Yang & Swager, 1998). A long-chain ether derivative was reported (Yang *et al.*, 2000*a*), as well as an arylsulfonyl diamide derivative of the title compound (Yang *et al.*, 2000*b*), while a 4′-carboxybenzyl ether derivative was described by Crane *et al.* (2013).

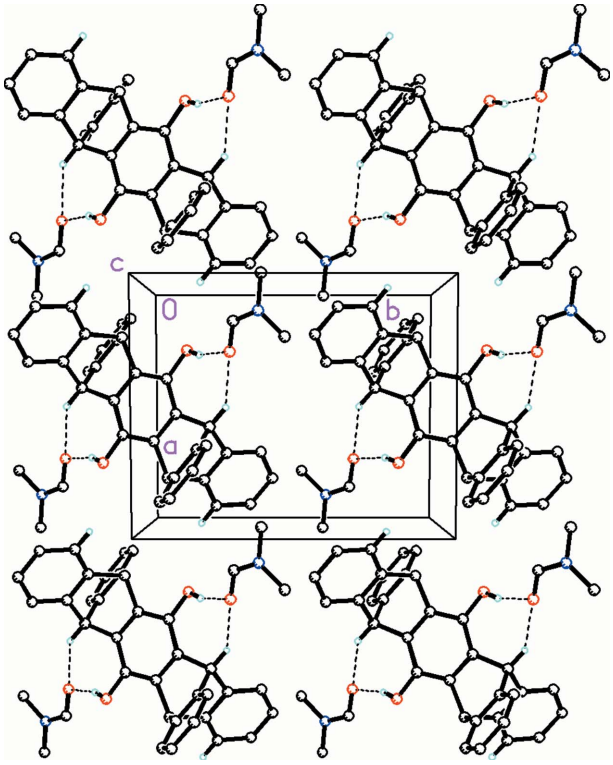


Figure 2
The crystal packing of the compound along the c-axis direction. Hydrogen bonds are shown as dashed lines. Hydrogen bonds are formed between the O atom of DMF interacting with the OH group and C—H unit of the title compound.

Synthesis and crystallization

The title pentiptycene-6,13-diol was prepared using a modified version of the Yang *et al.* (2000*a*) method (Fig. 4). For 2.5 g

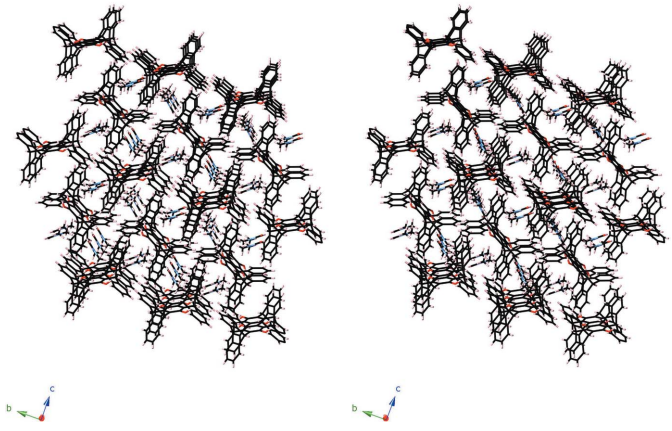


Figure 3
A stick-structure view of the lattice along the a-axis direction (inverse stereo).

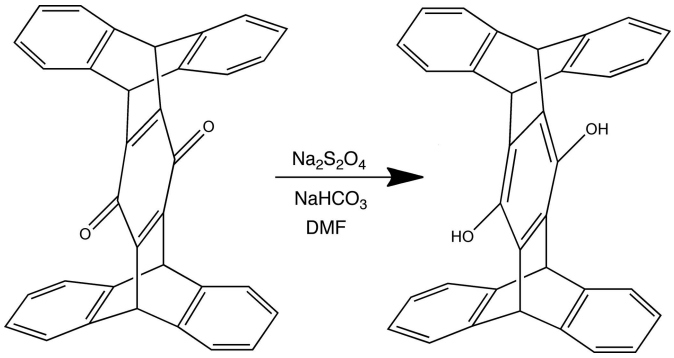


Figure 4
Synthesis of the title compound.

(5.5 mmol) of pentiptycene quinone (Cao *et al.* 2009), 50 ml of DMF was used as the solvent, with 3.25 g (39 mmol) $NaHCO_3$ and 3.25 g (19 mmol) $Na_2S_2O_4$. The reaction was carried out at 100°C under N_2. The total reaction time was adjusted to 16 h, with $Na_2S_2O_4$ being added in four portions at 4 h intervals. The crude product, precipitated by the addition of water to the cooled solution, was filtered off, washed with water, and vacuum desiccated, yielding a straw-colored powder (2.45 g, 97%), which was recrystallized from DMF.

Refinement

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{34}H_{22}O_2{\cdot}2C_3H_7NO$
M_r	608.71
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, b, c (Å)	9.3621 (2), 11.3559 (2), 15.1920 (3)
β (°)	98.0838 (18)
V (Å^3)	1599.10 (6)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.65
Crystal size (mm)	0.22 × 0.16 × 0.14
Data collection	
Diffractometer	Rigaku Oxford Diffraction EOS Gemini
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.690, 0.930
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5606, 3057, 2694
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.144, 1.04
No. of reflections	3057
No. of parameters	212
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.24

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161130 [https://doi.org/10.1107/S2414314616011305]

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Crystal data

$C_{34}H_{22}O_2·2C_3H_7NO$
M_r = 608.71
Monoclinic, $P2_1/n$
a = 9.3621 (2) Å
b = 11.3559 (2) Å
c = 15.1920 (3) Å
β = 98.0838 (18)°
V = 1599.10 (6) Å^3
Z = 2
$F(000)$ = 644
D_x = 1.264 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2642 reflections
θ = 3.9–71.4°
μ = 0.65 mm^{-1}
T = 173 K
Prism, clear yellow
0.22 × 0.16 × 0.14 mm

Data collection

Rigaku Oxford Diffraction EOS Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.690, T_{max} = 0.930
5606 measured reflections
3057 independent reflections
2694 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 71.4°, θ_{min} = 4.9°
h = −9→11
k = −13→8
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.144
S = 1.04
3057 reflections
212 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
$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2 + 0.2964P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0072 (8)

Special details

Experimental. CrysAlisPro (Agilent Technologies, 2014) Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.26816 (10)	0.14464 (9)	0.52443 (7)	0.0248 (3)
H1	0.2885	0.1882	0.5691	0.037*
O1S	0.27960 (13)	0.29190 (12)	0.66325 (9)	0.0421 (3)
N1S	0.12086 (14)	0.38670 (12)	0.73798 (9)	0.0300 (3)
C1	0.38466 (14)	0.07611 (12)	0.51417 (8)	0.0183 (3)
C1S	0.15856 (17)	0.30504 (14)	0.68404 (11)	0.0300 (4)
H1S	0.0857	0.2517	0.6591	0.036*
C2	0.52795 (14)	0.10760 (12)	0.54195 (8)	0.0187 (3)
C2S	0.2248 (2)	0.4717 (2)	0.77894 (15)	0.0579 (6)
H2SA	0.2535	0.4510	0.8416	0.087*
H2SB	0.1813	0.5504	0.7750	0.087*
H2SC	0.3099	0.4713	0.7480	0.087*
C3	0.64047 (14)	0.03218 (13)	0.52840 (9)	0.0189 (3)
C3S	−0.0241 (2)	0.39602 (19)	0.75999 (17)	0.0535 (6)
H3SA	−0.0650	0.4726	0.7403	0.080*
H3SB	−0.0215	0.3888	0.8245	0.080*
H3SC	−0.0839	0.3330	0.7301	0.080*
C4	0.78952 (14)	0.08027 (13)	0.56470 (9)	0.0218 (3)
H4	0.8697	0.0255	0.5556	0.026*
C5	0.80135 (15)	0.20036 (13)	0.52093 (9)	0.0222 (3)
C6	0.91058 (17)	0.23797 (15)	0.47492 (11)	0.0306 (4)
H6	0.9867	0.1862	0.4655	0.037*
C7	0.9069 (2)	0.35308 (17)	0.44255 (12)	0.0400 (4)
H7	0.9813	0.3799	0.4109	0.048*
C8	0.7963 (2)	0.42845 (16)	0.45601 (12)	0.0379 (4)
H8	0.7954	0.5068	0.4340	0.045*
C9	0.68584 (17)	0.38998 (14)	0.50192 (10)	0.0278 (4)
H9	0.6094	0.4417	0.5109	0.033*
C10	0.68865 (14)	0.27602 (13)	0.53422 (9)	0.0211 (3)
C11	0.58057 (15)	0.22199 (12)	0.58893 (9)	0.0206 (3)
H11	0.5003	0.2768	0.5980	0.025*
C12	0.67140 (15)	0.18401 (13)	0.67575 (9)	0.0232 (3)
C13	0.65195 (18)	0.21928 (15)	0.76050 (10)	0.0321 (4)
H13	0.5744	0.2699	0.7693	0.038*
C14	0.7483 (2)	0.17921 (19)	0.83278 (11)	0.0436 (5)
H14	0.7375	0.2037	0.8913	0.052*

C15	0.8586 (2)	0.1045 (2)	0.81966 (11)	0.0469 (5)
H15	0.9235	0.0778	0.8694	0.056*
C16	0.87715 (18)	0.06715 (16)	0.73443 (11)	0.0351 (4)
H16	0.9530	0.0145	0.7260	0.042*
C17	0.78356 (15)	0.10789 (13)	0.66261 (10)	0.0241 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0166 (5)	0.0267 (6)	0.0313 (6)	0.0050 (4)	0.0045 (4)	−0.0052 (4)
O1S	0.0316 (7)	0.0533 (8)	0.0444 (7)	0.0065 (6)	0.0156 (5)	−0.0142 (6)
N1S	0.0257 (7)	0.0310 (7)	0.0361 (7)	−0.0025 (5)	0.0138 (6)	−0.0064 (6)
C1	0.0142 (6)	0.0221 (7)	0.0193 (6)	0.0025 (5)	0.0043 (5)	0.0013 (5)
C1S	0.0285 (8)	0.0279 (8)	0.0349 (8)	−0.0004 (6)	0.0090 (6)	−0.0053 (6)
C2	0.0178 (7)	0.0213 (7)	0.0174 (6)	0.0006 (5)	0.0035 (5)	−0.0002 (5)
C2S	0.0451 (11)	0.0764 (16)	0.0556 (12)	−0.0217 (11)	0.0185 (9)	−0.0386 (11)
C3	0.0134 (6)	0.0246 (7)	0.0189 (6)	0.0005 (5)	0.0027 (5)	0.0018 (5)
C3S	0.0345 (10)	0.0454 (11)	0.0881 (16)	−0.0054 (9)	0.0344 (10)	−0.0184 (11)
C4	0.0137 (7)	0.0231 (7)	0.0282 (7)	0.0000 (5)	0.0016 (5)	−0.0020 (5)
C5	0.0176 (7)	0.0269 (7)	0.0221 (7)	−0.0032 (5)	0.0030 (5)	−0.0018 (5)
C6	0.0224 (7)	0.0357 (9)	0.0359 (8)	−0.0040 (7)	0.0115 (6)	−0.0047 (7)
C7	0.0385 (10)	0.0429 (10)	0.0428 (9)	−0.0094 (8)	0.0200 (8)	0.0041 (8)
C8	0.0436 (10)	0.0315 (9)	0.0404 (9)	−0.0047 (8)	0.0121 (8)	0.0088 (7)
C9	0.0269 (8)	0.0262 (8)	0.0296 (8)	−0.0003 (6)	0.0016 (6)	0.0006 (6)
C10	0.0182 (7)	0.0254 (7)	0.0197 (6)	−0.0023 (5)	0.0029 (5)	−0.0030 (5)
C11	0.0174 (7)	0.0228 (7)	0.0224 (7)	−0.0004 (5)	0.0051 (5)	−0.0033 (5)
C12	0.0219 (7)	0.0260 (7)	0.0215 (7)	−0.0086 (6)	0.0028 (5)	−0.0019 (5)
C13	0.0352 (9)	0.0376 (9)	0.0247 (8)	−0.0153 (7)	0.0089 (6)	−0.0067 (6)
C14	0.0503 (11)	0.0587 (12)	0.0218 (8)	−0.0237 (10)	0.0045 (7)	−0.0029 (7)
C15	0.0464 (11)	0.0636 (13)	0.0257 (9)	−0.0191 (10)	−0.0125 (7)	0.0141 (8)
C16	0.0269 (8)	0.0367 (9)	0.0383 (9)	−0.0061 (7)	−0.0073 (7)	0.0074 (7)
C17	0.0198 (7)	0.0259 (7)	0.0255 (7)	−0.0075 (6)	0.0003 (5)	0.0017 (6)

Geometric parameters (Å, º)

O1—H1	0.8400	C5—C10	1.397 (2)
O1—C1	1.3665 (16)	C6—H6	0.9500
O1S—C1S	1.2275 (19)	C6—C7	1.395 (2)
N1S—C1S	1.318 (2)	C7—H7	0.9500
N1S—C2S	1.448 (2)	C7—C8	1.381 (3)
N1S—C3S	1.446 (2)	C8—H8	0.9500
C1—C2	1.3951 (19)	C8—C9	1.396 (2)
C1—C3^{i}	1.3939 (19)	C9—H9	0.9500
C1S—H1S	0.9500	C9—C10	1.383 (2)
C2—C3	1.3951 (19)	C10—C11	1.5257 (18)
C2—C11	1.5299 (18)	C11—H11	1.0000
C2S—H2SA	0.9800	C11—C12	1.5275 (19)
C2S—H2SB	0.9800	C12—C13	1.385 (2)

C2S—H2SC	0.9800	C12—C17	1.396 (2)
C3—C1^{i}	1.3939 (19)	C13—H13	0.9500
C3—C4	1.5275 (18)	C13—C14	1.396 (3)
C3S—H3SA	0.9800	C14—H14	0.9500
C3S—H3SB	0.9800	C14—C15	1.373 (3)
C3S—H3SC	0.9800	C15—H15	0.9500
C4—H4	1.0000	C15—C16	1.396 (3)
C4—C5	1.528 (2)	C16—H16	0.9500
C4—C17	1.529 (2)	C16—C17	1.380 (2)
C5—C6	1.385 (2)		

C1—O1—H1	109.5	C5—C6—C7	118.92 (15)
C1S—N1S—C2S	120.93 (14)	C7—C6—H6	120.5
C1S—N1S—C3S	122.50 (15)	C6—C7—H7	119.7
C3S—N1S—C2S	116.58 (15)	C8—C7—C6	120.68 (15)
O1—C1—C2	124.64 (13)	C8—C7—H7	119.7
O1—C1—C3^{i}	118.03 (12)	C7—C8—H8	119.9
C3^{i}—C1—C2	117.31 (12)	C7—C8—C9	120.24 (16)
O1S—C1S—N1S	125.67 (15)	C9—C8—H8	119.9
O1S—C1S—H1S	117.2	C8—C9—H9	120.3
N1S—C1S—H1S	117.2	C10—C9—C8	119.44 (15)
C1—C2—C11	126.27 (12)	C10—C9—H9	120.3
C3—C2—C1	120.79 (13)	C5—C10—C11	113.80 (13)
C3—C2—C11	112.94 (12)	C9—C10—C5	120.08 (13)
N1S—C2S—H2SA	109.5	C9—C10—C11	126.06 (13)
N1S—C2S—H2SB	109.5	C2—C11—H11	113.2
N1S—C2S—H2SC	109.5	C10—C11—C2	106.38 (11)
H2SA—C2S—H2SB	109.5	C10—C11—H11	113.2
H2SA—C2S—H2SC	109.5	C10—C11—C12	104.63 (11)
H2SB—C2S—H2SC	109.5	C12—C11—C2	105.47 (11)
C1^{i}—C3—C2	121.89 (12)	C12—C11—H11	113.2
C1^{i}—C3—C4	124.68 (12)	C13—C12—C11	126.24 (14)
C2—C3—C4	113.43 (12)	C13—C12—C17	120.79 (14)
N1S—C3S—H3SA	109.5	C17—C12—C11	112.96 (12)
N1S—C3S—H3SB	109.5	C12—C13—H13	120.6
N1S—C3S—H3SC	109.5	C12—C13—C14	118.84 (17)
H3SA—C3S—H3SB	109.5	C14—C13—H13	120.6
H3SA—C3S—H3SC	109.5	C13—C14—H14	119.9
H3SB—C3S—H3SC	109.5	C15—C14—C13	120.23 (16)
C3—C4—H4	113.2	C15—C14—H14	119.9
C3—C4—C5	106.28 (11)	C14—C15—H15	119.4
C3—C4—C17	105.55 (11)	C14—C15—C16	121.11 (16)
C5—C4—H4	113.2	C16—C15—H15	119.4
C5—C4—C17	104.78 (11)	C15—C16—H16	120.5
C17—C4—H4	113.2	C17—C16—C15	118.91 (17)
C6—C5—C4	126.78 (13)	C17—C16—H16	120.5
C6—C5—C10	120.63 (14)	C12—C17—C4	113.40 (12)
C10—C5—C4	112.55 (12)	C16—C17—C4	126.49 (15)

C5—C6—H6	120.5	C16—C17—C12	120.10 (14)
O1—C1—C2—C3	−179.72 (12)	C5—C10—C11—C2	−55.02 (15)
O1—C1—C2—C11	0.6 (2)	C5—C10—C11—C12	56.33 (15)
C1—C2—C3—C1^{i}	0.9 (2)	C6—C5—C10—C9	−0.5 (2)
C1—C2—C3—C4	−178.88 (12)	C6—C5—C10—C11	−177.95 (13)
C1—C2—C11—C10	−126.00 (14)	C6—C7—C8—C9	−0.4 (3)
C1—C2—C11—C12	123.23 (14)	C7—C8—C9—C10	0.4 (3)
C1^{i}—C3—C4—C5	124.20 (14)	C8—C9—C10—C5	0.0 (2)
C1^{i}—C3—C4—C17	−124.87 (14)	C8—C9—C10—C11	177.18 (14)
C2—C3—C4—C5	−55.99 (15)	C9—C10—C11—C2	127.69 (14)
C2—C3—C4—C17	54.95 (15)	C9—C10—C11—C12	−120.96 (15)
C2—C11—C12—C13	−125.00 (15)	C10—C5—C6—C7	0.5 (2)
C2—C11—C12—C17	55.92 (15)	C10—C11—C12—C13	122.99 (15)
C2S—N1S—C1S—O1S	0.1 (3)	C10—C11—C12—C17	−56.08 (15)
C3^{i}—C1—C2—C3	−0.9 (2)	C11—C2—C3—C1^{i}	−179.33 (12)
C3^{i}—C1—C2—C11	179.41 (12)	C11—C2—C3—C4	0.85 (16)
C3—C2—C11—C10	54.29 (15)	C11—C12—C13—C14	−177.77 (14)
C3—C2—C11—C12	−56.48 (14)	C11—C12—C17—C4	0.02 (17)
C3—C4—C5—C6	−126.94 (15)	C11—C12—C17—C16	178.85 (13)
C3—C4—C5—C10	55.23 (15)	C12—C13—C14—C15	−1.1 (3)
C3—C4—C17—C12	−55.49 (15)	C13—C12—C17—C4	−179.11 (13)
C3—C4—C17—C16	125.76 (15)	C13—C12—C17—C16	−0.3 (2)
C3S—N1S—C1S—O1S	−179.44 (19)	C13—C14—C15—C16	0.0 (3)
C4—C5—C6—C7	−177.19 (14)	C14—C15—C16—C17	0.9 (3)
C4—C5—C10—C9	177.50 (12)	C15—C16—C17—C4	177.86 (15)
C4—C5—C10—C11	0.03 (16)	C15—C16—C17—C12	−0.8 (2)
C5—C4—C17—C12	56.50 (15)	C17—C4—C5—C6	121.59 (15)
C5—C4—C17—C16	−122.25 (16)	C17—C4—C5—C10	−56.24 (14)
C5—C6—C7—C8	−0.1 (3)	C17—C12—C13—C14	1.2 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1*S*	0.84	1.86	2.6818 (16)	164
C6—H6···O1^{ii}	0.95	2.70	3.4912 (19)	141
C11—H11···O1*S*	1.00	2.42	3.2771 (18)	143

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