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Crystal structure of 1,1'-[imidazolidine-1,3-diylbis(methylene)]bis(naphthalen-2-ol)

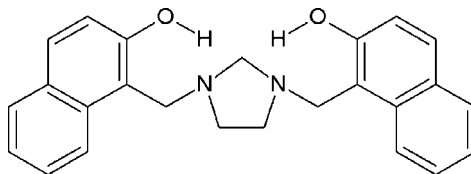
Augusto Rivera,^{a*} Jicli José Rojas,^a Jaime Ríos-Motta^a and Michael Bolte^b

^aDepartamento de Química, Facultad de Ciencias, Universidad Nacional de Colombia, Sede Bogotá, Cra 30 No. 45-03, Bogotá, Colombia, and ^bInstitut für Anorganische Chemie, Goethe-Universität, Max-von-Laue-Strasse 7, Frankfurt/Main D-60438, Germany. *Correspondence e-mail: ariverau@unal.edu.co

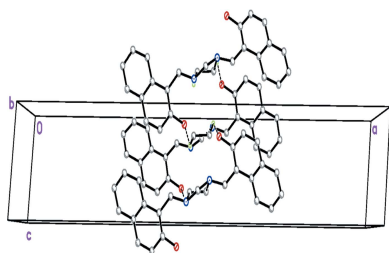
The crystal structure of the title compound, C₂₅H₂₄N₂O₂, at 173 K has monoclinic (*C2/c*) symmetry. The molecule is located on a crystallographic twofold rotation axis with only half a molecule in the asymmetric unit. The imidazolidine ring adopts a twist conformation, with a twist about the ring C—C bond. The crystal structure shows the *anticlinal* disposition of the two (2-hydroxynaphthalen-1-yl)methyl substituents of the imidazolidine ring. The structure displays two intramolecular O—H···N hydrogen bonds, each forming an *S*(6) ring motif.

1. Chemical context

We have been interested in the synthesis and characterization of a family of symmetrical *N,N'*-disubstituted imidazolidines by the use of a Mannich-type condensation of *cyclic cage amins* with phenols in a one-pot reaction. The main structural feature of the symmetrical *N,N'*-disubstituted imidazolidines, the so-called aromatic *di*-Mannich bases, is to form intramolecular hydrogen bonds that reveal great structural and thermodynamic stability. These *di*-Mannich bases which contain a phenolic or naphtholic hydroxyl group as a proton donor, as well as an *ortho*-aminomethyl group as a proton acceptor in the same molecule are convenient models for studying the nature of hydrogen bonding and other weak non-covalent interactions (Koll *et al.*, 2006).



In previous studies (Rivera *et al.*, 2006), 1,1'-[imidazolidine-1,3-diylbis(methylene)]bis(naphthalen-2-ol), (I), was obtained in good yields by an one-pot Mannich-type reaction involving 1,3,6,8-tetraazatricyclo[4.4.1.1^{3,8}]dodecane (TATD) and naphthalen-2-ol in classical solvents for Mannich reactions, such as dioxane or ethanol. Intriguingly, reactions of 1,3,6,8-tetraazatricyclo[4.4.1.1^{3,8}]dodecane (TATD) with naphthalen-2-ol may lead to other results. It has been found (Rivera & Quevedo, 2013) that interaction of TATD with naphthalen-2-ol in solvent-free conditions by heating in an oil bath a 1:4 mixture with stirring at 423 K for 20 min gives 1,1'-methylenebis(naphthalen-2-ol) in good yields. On the other hand, the reactions of TATD with naphthalen-2-ol under solvent-free



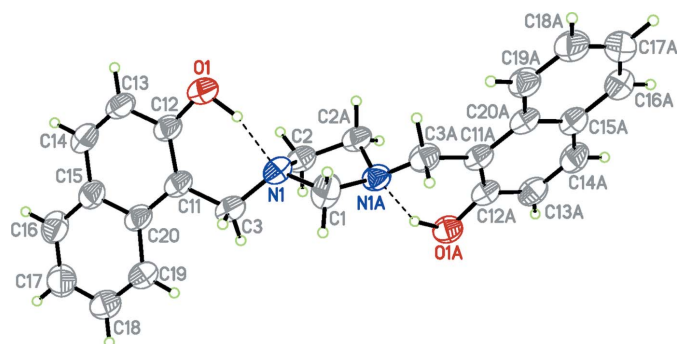


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are drawn as dashed lines. Atoms labelled with the suffix 'A' are generated using the symmetry operator $(-x + 1, y, -z + \frac{1}{2})$.

microwave-assisted conditions yields the title compound and no formation of 1,1'-methylenebis(naphthalen-2-ol) was observed. In contrast to classical Mannich reaction conditions this reaction required neither solvent nor inert atmosphere conditions.

2. Structural commentary

In contrast to the closely related structure (Rivera *et al.*, 2012*a*), which crystallized in the monoclinic $P2_1/n$ space group, the title compound crystallizes in the $C2/c$ space group. The molecular structure is shown in Fig. 1. The asymmetric unit contains one half molecule and the whole molecule is generated by twofold rotational symmetry (see Fig. 1). The near planarity of the fused aromatic ring system is illustrated by the very small deviation of all the atoms from the plane [largest deviation = 0.0227 (17) Å for atom C11]. The imidazolidine ring (C1/N1/C2/C2'/N1') is in a twisted conformation on C2–C2', with puckering parameters $Q(2) = 0.4126$ (17) Å and $\varphi(2) = 126.0$ (2)° (Cremer & Pople, 1975). The crystal structure shows the *anticlinal* disposition of the two (2-hydroxynaphthalen-1-yl)methyl substituents of the imidazolidine ring [pseudo-torsion angle $\text{CH}_2\text{—N}\cdots\text{N—CH}_2 = -121.77$ (18)°]. The mean plane of the imidazolidine ring, defined by atoms N1, C1 and N1', makes a dihedral angle of 70.92 (4)° with the pendant aromatic rings (C11–C20). The dihedral angle between the planes of the naphthyl rings is 60.55 (4)°.

As with related structures in this series, the molecular conformation is stabilized by two intramolecular O—H...N hydrogen-bond interactions with $S(6)$ graph-set motifs (Bernstein *et al.*, 1995). Due to symmetry and contrary to other structures, where hydrogen-bond distances were different, the two observed intramolecular hydrogen-bond distances were identical (Table 1).

3. Supramolecular features

Unlike the situation found in related structures, there is only one significant intermolecular interaction involving the O—H group (as acceptor) and a methylene-H atom (as donor) to

Table 1

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H1...N1	1.05 (2)	1.65 (2)	2.6143 (19)	151.0 (19)
C2—H2A...O1 ⁱ	0.99	2.64	3.257 (2)	121

Symmetry code: (i) $x, -y + 2, z - \frac{1}{2}$

consolidate the crystal packing. These weak interactions led to the formation of parallel sets of zigzag chains extending along the c axis of the crystal (Fig. 2).

4. Database survey

A search in the Cambridge Structural Database (Groom & Allen, 2014) for the fragment 2,2'-[imidazolidine-1,3-diyl bis(methylene)]diphenol yielded seven hits, namely 2,2'-[imidazolidine-1,3-diylbis(methylene)]bis(4-*tert*-butylphenol) (Rivera, Nerio & Bolte, 2013), 2,2'-[imidazolidine-1,3-diylbis(methylene)]bis(4-chlorophenol) (Rivera *et al.*, 2011), 2,2'-[imidazolidine-1,3-diylbis(methylene)]bis[4-(2,4,4-trimethylpentan-2-yl)phenol] (Kober *et al.*, 2012), 4,4'-difluoro-2,2'-[imidazolidine-1,3-diylbis(methylene)]diphenol (Rivera *et al.*, 2012*b*) 2,2'-[imidazolidine-1,3-diylbis(methylene)]bis(6-methylphenol) (Rivera *et al.*, 2014), 2,2'-[imidazolidine-1,3-diylbis(methylene)]diphenol (Rivera *et al.*, 2012*b*) and 4,4'-dimethyl-2,2'-[imidazolidine-1,3-diylbis(methylene)]diphenol (Rivera *et al.*, 2012*c*). In all of these compounds, the hydroxy groups in the *ortho* position of the aromatic ring form an intramolecular hydrogen bond to an N atom of the imidazolidine ring.

5. Synthesis and crystallization

The title compound has been synthesized in solution according to a literature procedure (Rivera *et al.*, 2006); however, in this instance, the synthesis was carried out under microwave-assisted solvent free conditions. A mixture of 1 mmol of 1,3,6,8-tetraazatricyclo[4.4.1.1^{3,8}]dodecane (TATD) and

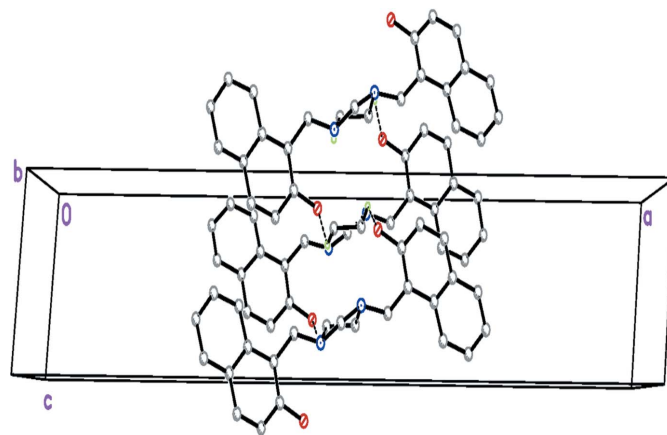


Figure 2

The crystal packing of the title compound, showing one of the zigzag chains that extend along the crystal c -axis direction. Hydrogen bonds are drawn as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₂₅ H ₂₄ N ₂ O ₂
<i>M_r</i>	384.46
Crystal system, space group	Monoclinic, <i>C2/c</i>
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	34.883 (5), 8.3956 (9), 6.5830 (8)
β (°)	95.650 (11)
<i>V</i> (Å ³)	1918.6 (4)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.09
Crystal size (mm)	0.19 × 0.17 × 0.11
Data collection	
Diffractometer	Stoe IPDS II two circle
Absorption correction	Multi-scan (<i>X-AREA</i> ; Stoe & Cie, 2001)
<i>T_{min}</i> , <i>T_{max}</i>	0.972, 0.989
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	8297, 1852, 1451
<i>R_{int}</i>	0.090
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.616
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.055, 0.159, 1.09
No. of reflections	1852
No. of parameters	136
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.24, -0.23

Computer programs: *X-AREA* (Stoe & Cie, 2001), *SHELXS97* and *XP* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

2 mmol of naphthalen-2-ol was subjected to microwave irradiation (200 W) for 10 min at a temperature of 373 K. The product was washed with water and then with benzene (yield 94%, m.p. 435–436 K). Crystals suitable for X-ray diffraction were obtained from a methanol solution upon slow evaporation of the solvent at room temperature.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were located in the

difference electron-density map. The hydroxy H atom was refined freely, while C-bound H atoms were fixed geometrically (C–H = 0.95 or 0.99 Å) and refined using a riding model, with *U*_{iso}(H) values set at 1.2*U*_{eq} of the parent atom.

Acknowledgements

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA* (Stoe & Cie, 2001); data reduction: *X-AREA* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *pubCIF* (Westrip, 2010).

1-({3-[(2-Hydroxynaphthalen-1-yl)methyl]imidazolidin-1-yl)methylnaphthalen-2-ol

Crystal data

$C_{25}H_{24}N_2O_2$	$F(000) = 816$
$M_r = 384.46$	$D_x = 1.331 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 34.883 (5) \text{ \AA}$	Cell parameters from 8026 reflections
$b = 8.3956 (9) \text{ \AA}$	$\theta = 2.4\text{--}26.2^\circ$
$c = 6.5830 (8) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 95.650 (11)^\circ$	$T = 173 \text{ K}$
$V = 1918.6 (4) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.19 \times 0.17 \times 0.11 \text{ mm}$

Data collection

Stoe IPDS II two-circle diffractometer	1852 independent reflections
ω scans	1451 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan <i>X-AREA</i> (Stoe & Cie, 2001)	$R_{\text{int}} = 0.090$
$T_{\text{min}} = 0.972$, $T_{\text{max}} = 0.989$	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 2.5^\circ$
8297 measured reflections	$h = -42 \rightarrow 34$
	$k = -10 \rightarrow 10$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.055$	$w = 1/[\sigma^2(F_o^2) + (0.085P)^2 + 0.4089P]$
$wR(F^2) = 0.159$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1852 reflections	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
136 parameters	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
0 restraints	

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.45368 (4)	0.77294 (16)	0.70176 (19)	0.0507 (4)	
H1	0.4677 (6)	0.723 (3)	0.583 (4)	0.061 (6)*	
N1	0.47179 (4)	0.69601 (18)	0.3384 (2)	0.0455 (4)	
C1	0.5000	0.5948 (4)	0.2500	0.0627 (8)	
H1A	0.5132	0.5258	0.3570	0.075*	0.5
H1B	0.4868	0.5258	0.1430	0.075*	0.5
C2	0.47856 (5)	0.8543 (2)	0.2548 (2)	0.0488 (5)	
H2A	0.4707	0.9396	0.3459	0.059*	
H2B	0.4646	0.8678	0.1175	0.059*	
C3	0.43203 (5)	0.6374 (2)	0.2997 (3)	0.0491 (5)	
H3A	0.4319	0.5212	0.3249	0.059*	
H3B	0.4231	0.6549	0.1540	0.059*	
C11	0.40403 (5)	0.7157 (2)	0.4287 (2)	0.0437 (4)	
C12	0.41619 (5)	0.7796 (2)	0.6188 (2)	0.0446 (4)	
C13	0.38994 (6)	0.8541 (2)	0.7379 (3)	0.0511 (5)	
H13	0.3990	0.8993	0.8660	0.061*	
C14	0.35197 (6)	0.8623 (2)	0.6724 (3)	0.0541 (5)	
H14	0.3348	0.9149	0.7536	0.065*	
C15	0.33758 (5)	0.7931 (2)	0.4833 (3)	0.0499 (5)	
C16	0.29795 (6)	0.7964 (3)	0.4150 (3)	0.0611 (6)	
H16	0.2805	0.8489	0.4948	0.073*	
C17	0.28426 (6)	0.7252 (3)	0.2355 (3)	0.0682 (6)	
H17	0.2575	0.7286	0.1910	0.082*	
C18	0.30975 (6)	0.6476 (3)	0.1178 (3)	0.0662 (6)	
H18	0.3001	0.5965	-0.0055	0.079*	
C19	0.34828 (6)	0.6438 (3)	0.1770 (3)	0.0545 (5)	
H19	0.3650	0.5908	0.0938	0.065*	
C20	0.36387 (5)	0.7181 (2)	0.3618 (2)	0.0455 (5)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0588 (8)	0.0562 (8)	0.0367 (6)	-0.0007 (6)	0.0024 (5)	-0.0039 (5)
N1	0.0551 (9)	0.0469 (8)	0.0350 (7)	0.0057 (6)	0.0072 (6)	-0.0001 (6)
C1	0.0688 (18)	0.0559 (17)	0.0670 (17)	0.000	0.0256 (14)	0.000
C2	0.0617 (11)	0.0505 (11)	0.0342 (8)	0.0027 (8)	0.0047 (7)	0.0023 (7)
C3	0.0615 (12)	0.0496 (10)	0.0364 (8)	-0.0001 (8)	0.0061 (7)	-0.0058 (7)
C11	0.0590 (11)	0.0412 (9)	0.0318 (8)	0.0009 (8)	0.0086 (7)	0.0016 (6)
C12	0.0574 (11)	0.0438 (10)	0.0331 (8)	-0.0026 (7)	0.0067 (7)	0.0024 (6)

C13	0.0676 (12)	0.0525 (11)	0.0342 (8)	-0.0042 (9)	0.0098 (8)	-0.0063 (7)
C14	0.0667 (12)	0.0548 (11)	0.0436 (9)	0.0030 (9)	0.0192 (8)	-0.0039 (8)
C15	0.0570 (11)	0.0539 (11)	0.0399 (9)	-0.0012 (8)	0.0107 (7)	0.0054 (7)
C16	0.0597 (12)	0.0746 (14)	0.0508 (11)	0.0032 (10)	0.0141 (9)	0.0079 (9)
C17	0.0549 (12)	0.0951 (18)	0.0539 (12)	-0.0018 (11)	0.0020 (9)	0.0091 (11)
C18	0.0678 (14)	0.0890 (16)	0.0409 (10)	-0.0065 (12)	0.0010 (9)	0.0002 (10)
C19	0.0614 (12)	0.0646 (12)	0.0376 (9)	-0.0022 (9)	0.0055 (8)	-0.0020 (8)
C20	0.0602 (11)	0.0455 (10)	0.0316 (8)	-0.0021 (8)	0.0082 (7)	0.0036 (6)

Geometric parameters (Å, °)

O1—C12	1.368 (2)	C12—C13	1.409 (3)
O1—H1	1.05 (2)	C13—C14	1.354 (3)
N1—C1	1.464 (2)	C13—H13	0.9500
N1—C2	1.467 (2)	C14—C15	1.420 (3)
N1—C3	1.470 (2)	C14—H14	0.9500
C1—N1 ⁱ	1.464 (2)	C15—C16	1.411 (3)
C1—H1A	0.9900	C15—C20	1.422 (3)
C1—H1B	0.9900	C16—C17	1.368 (3)
C2—C2 ⁱ	1.503 (4)	C16—H16	0.9500
C2—H2A	0.9900	C17—C18	1.397 (3)
C2—H2B	0.9900	C17—H17	0.9500
C3—C11	1.507 (2)	C18—C19	1.362 (3)
C3—H3A	0.9900	C18—H18	0.9500
C3—H3B	0.9900	C19—C20	1.427 (2)
C11—C12	1.389 (2)	C19—H19	0.9500
C11—C20	1.427 (3)		
C12—O1—H1	102.4 (12)	O1—C12—C13	116.36 (15)
C1—N1—C2	103.73 (15)	C11—C12—C13	121.01 (17)
C1—N1—C3	113.36 (14)	C14—C13—C12	120.94 (16)
C2—N1—C3	114.98 (14)	C14—C13—H13	119.5
N1 ⁱ —C1—N1	109.0 (2)	C12—C13—H13	119.5
N1 ⁱ —C1—H1A	109.9	C13—C14—C15	120.60 (17)
N1—C1—H1A	109.9	C13—C14—H14	119.7
N1 ⁱ —C1—H1B	109.9	C15—C14—H14	119.7
N1—C1—H1B	109.9	C16—C15—C14	121.46 (18)
H1A—C1—H1B	108.3	C16—C15—C20	119.72 (18)
N1—C2—C2 ⁱ	102.34 (10)	C14—C15—C20	118.82 (18)
N1—C2—H2A	111.3	C17—C16—C15	120.9 (2)
C2 ⁱ —C2—H2A	111.3	C17—C16—H16	119.5
N1—C2—H2B	111.3	C15—C16—H16	119.5
C2 ⁱ —C2—H2B	111.3	C16—C17—C18	119.7 (2)
H2A—C2—H2B	109.2	C16—C17—H17	120.1
N1—C3—C11	114.13 (14)	C18—C17—H17	120.1
N1—C3—H3A	108.7	C19—C18—C17	121.1 (2)
C11—C3—H3A	108.7	C19—C18—H18	119.5
N1—C3—H3B	108.7	C17—C18—H18	119.5

C11—C3—H3B	108.7	C18—C19—C20	121.08 (19)
H3A—C3—H3B	107.6	C18—C19—H19	119.5
C12—C11—C20	118.43 (16)	C20—C19—H19	119.5
C12—C11—C3	121.27 (17)	C15—C20—C11	120.07 (16)
C20—C11—C3	120.22 (15)	C15—C20—C19	117.41 (18)
O1—C12—C11	122.62 (16)	C11—C20—C19	122.50 (17)
C2—N1—C1—N1 ⁱ	13.70 (8)	C13—C14—C15—C20	-1.4 (3)
C3—N1—C1—N1 ⁱ	139.08 (14)	C14—C15—C16—C17	-178.1 (2)
C1—N1—C2—C2 ⁱ	-34.89 (17)	C20—C15—C16—C17	1.6 (3)
C3—N1—C2—C2 ⁱ	-159.24 (14)	C15—C16—C17—C18	0.2 (4)
C1—N1—C3—C11	166.25 (14)	C16—C17—C18—C19	-1.2 (4)
C2—N1—C3—C11	-74.64 (18)	C17—C18—C19—C20	0.4 (3)
N1—C3—C11—C12	-26.7 (2)	C16—C15—C20—C11	179.33 (16)
N1—C3—C11—C20	156.64 (15)	C14—C15—C20—C11	-1.0 (3)
C20—C11—C12—O1	175.45 (15)	C16—C15—C20—C19	-2.2 (3)
C3—C11—C12—O1	-1.3 (3)	C14—C15—C20—C19	177.39 (17)
C20—C11—C12—C13	-3.9 (3)	C12—C11—C20—C15	3.6 (3)
C3—C11—C12—C13	179.33 (16)	C3—C11—C20—C15	-179.57 (16)
O1—C12—C13—C14	-177.87 (16)	C12—C11—C20—C19	-174.69 (17)
C11—C12—C13—C14	1.5 (3)	C3—C11—C20—C19	2.1 (3)
C12—C13—C14—C15	1.2 (3)	C18—C19—C20—C15	1.3 (3)
C13—C14—C15—C16	178.20 (18)	C18—C19—C20—C11	179.66 (18)

Symmetry code: (i) $-x+1, y, -z+1/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	1.05 (2)	1.65 (2)	2.6143 (19)	151.0 (19)
C2—H2 <i>A</i> \cdots O1 ⁱⁱ	0.99	2.64	3.257 (2)	121

Symmetry code: (ii) $x, -y+2, z-1/2$.