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Crystal structure of 1,1'-{[(pentane-1,5-diyl)bis-[(azaniumylidene)methanylylidene]]bis-(naphthalen-2-olate)}

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The whole molecule of the title compound, C₂₇H₂₆N₂O₂, is generated by twofold rotational symmetry, with the central C atom of the pentyl chain located on the twofold rotation axis. The compound crystallizes as a bis-zwitterion, and there are two intramolecular N—H···O hydrogen bonds generating S(6) ring motifs. In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds, forming ribbons propagating along [001], and enclosing R₂²(22) ring motifs.

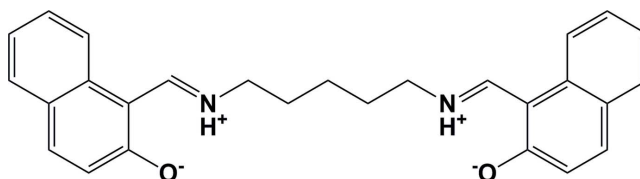
Keywords: crystal structure; 1,5-diaminopentane; 2-hydroxy-1-naphthaldehyde; zwitterion; bis-zwitterion; hydrogen bonding

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Supporting information: this article has supporting information at journals.iucr.org/e

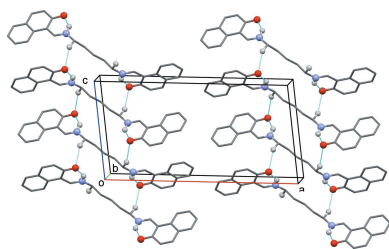
1. Chemical context

Tetradentate NNOO Schiff-bases have been used extensively as supporting ligands in *d*-block chemistry because of their ability to stabilize metals in various oxidation states (Alaghaz *et al.*, 2014; Kianfar *et al.*, 2015; Mikhalyova *et al.*, 2014; Borthakur *et al.*, 2014; Basumatary *et al.*, 2015). For many years, particular attention has been devoted to imines because of their uses as catalysts in various organic transformations (Khorshidifard *et al.*, 2015), and for their anticancer (Shiju *et al.*, 2015), antifungal (Abo-Aly *et al.*, 2015) and antibacterial (Salehi *et al.*, 2015) properties. They have also been used as sensors (Bandi *et al.*, 2013), corrosion inhibitors (Dasami *et al.*, 2015) and optical and fluorescent probes (Shoora *et al.*, 2015; Prabhakara *et al.*, 2015).



The microwave-assisted synthesis method, in solvent or solvent-free, is efficient and rapid. It gives cleaner reactions, is easy to use, gives higher yields and is a more economical synthetic process for the preparation of Schiff base compounds compared to conventional methods. It has been used to enhance the yield and reduce the time of certain reactions: for example, a one-step synthesis of D-A-D chromophores as active materials for organic solar cells (Jeux *et al.*, 2015), or the synthesis of a series of acyclic Schiff base-chromium(III) complexes (Kumar *et al.*, 2015).

In a continuation of our work on Schiff base ligands, we report herein on the crystal structure of the title compound, synthesized using two methods, *viz.* microwave irradiation and



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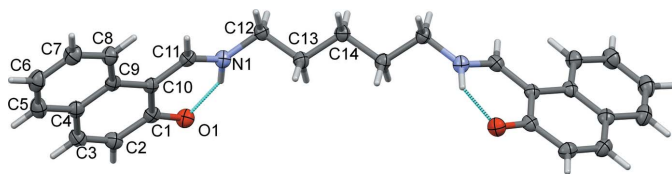


Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bonds are shown as dashed lines (see Table 1). The unlabelled atoms are related to the labelled atoms by twofold rotational symmetry (atom C14 lies on the twofold axis; symmetry code: $-x, y, -z + \frac{1}{2}$).

conventional, by condensing *o*-hydroxynaphthaldehyde and 1,5-diaminopentane.

2. Structural commentary

The whole molecule of the title compound, Fig. 1, is generated by twofold rotational symmetry, with the central C atom of the pentyl chain, C14, located on the twofold rotation axis. It crystallizes as a bis-zwitterion, with strong intramolecular N—H \cdots O hydrogen bonding between the imino N atom N1 (N1ⁱ), and the O atom, O1 (O1ⁱ) [$d(\text{O}\cdots\text{N}) = 2.5437(17)$ Å; symmetry code: (i) $-x, y, -z + \frac{1}{2}$], forming *S*(6) ring motifs (Fig. 1 and Table 1). The pentyl chain has an extended conformation with the naphthalene rings inclined to one another by 89.94 (5)°.

3. Supramolecular features

In the crystal, molecules are linked by pairs of C—H \cdots O hydrogen bonds, forming ribbons propagating along [001] and enclosing $R_2^2(22)$ ring motifs (Table 1 and Fig. 2).

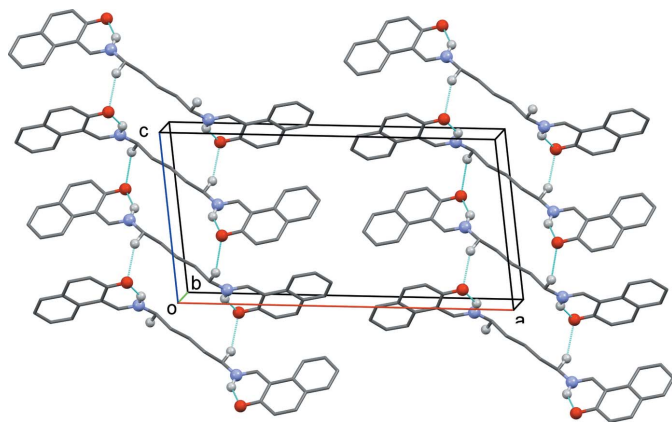


Figure 2

Crystal packing of the title compound viewed along the *b* axis. The hydrogen bonds are shown as dashed lines (see Table 1). For clarity, only the H atoms involved in hydrogen bonding have been included.

Table 1

Hydrogen-bond geometry (Å, °).

<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>
N1—H1N \cdots O1	0.96 (2)	1.72 (2)	2.5437 (17)	141.3 (16)
C12—H12A \cdots O1 ⁱ	0.99	2.45	3.2871 (19)	142

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

4. Database survey

Recently, our group reported the crystal structures of three new Schiff bases synthesized using conventional or ultrasonic irradiation methods by reacting primary amines and *o*-hydroxynaphthaldehyde (Ouari *et al.*, 2015*a,b,c*). They too crystallize as bis-zwitterionic compounds with strong intramolecular N—H \cdots O hydrogen bonds forming *S*(6) ring motifs.

5. Synthesis and crystallization

Method 1: Microwave synthesis

2-Hydroxy-1-naphthaldehyde (0.344g, 2 mmol), mixed and ground in a mortar, was placed in a reaction flask, and then 1,5-diaminopentane (0.109 g, 1 mmol) in 2 ml of methanol was added. The reaction mixture was then irradiated in a microwave oven for 1 min at 600 W. Upon completion, based on TLC analysis (silica gel, CH₂Cl₂/MeOH, 9.5/0.5, *v/v*), the product was washed with methanol (3 × 3 ml) and diethyl ether (3 × 3 ml) and filtered. Yellow crystals of the title compound, suitable for X-ray diffraction analysis, were obtained after two days by slow evaporation of a solution in DMSO/MeOH (yield: 95%, m.p.: 438–440 K). Elemental analysis calculated for C₂₇H₂₆N₂O₂: C, 80.00; H, 6.38; N, 6.82%; found: C, 80.42; H, 6.63; N, 6.56%.

Method 2: Conventional synthesis

The title Schiff base was prepared by condensation between 1,5-diaminopentane (51 mg, 0.5 mmol) and 2-hydroxy-1-naphthaldehyde (172 mg, 1 mmol) in methanol (10 ml). The mixture was refluxed and stirred under a nitrogen atmosphere for 3 h. The precipitate obtained was filtered, washed with methanol and diethyl ether and dried in vacuum overnight. Yellow single crystals of the title compound were obtained by slow evaporation of a solution in methanol (yield 71%; m.p.: 438–440 K).

As expected, the yield using method 1 (95%) is significantly greater than that using method 2 (71%).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The iminium H atom was located from a difference Fourier map and freely refined. C-bound H atoms were included in calculated positions and treated as riding atoms: C—H = 0.95 – 0.99 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Atom C14 lies on the twofold rotation axis and the H atoms were placed using instruction HFIX 23 (Sheldrick, 2015); the

occupancy of the methylene H atoms were fixed automatically at 0.5.

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	C ₂₇ H ₂₆ N ₂ O ₂
<i>M_r</i>	410.50
Crystal system, space group	Monoclinic, <i>P2/c</i>
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	20.9080 (13), 4.7429 (2), 10.6810 (6)
β (°)	96.419 (3)
<i>V</i> (Å ³)	1052.54 (10)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.08
Crystal size (mm)	0.45 × 0.20 × 0.10
Data collection	
Diffractometer	Nonius KappaCCD
Absorption correction	Multi-scan (<i>MULSCAN</i> in <i>PLATON</i> ; Spek, 2009)
<i>T_{min}</i> , <i>T_{max}</i>	0.792, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5781, 1958, 1402
<i>R_{int}</i>	0.049
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.606
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.049, 0.120, 1.08
No. of reflections	1958
No. of parameters	146
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.16, -0.14

Computer programs: *COLLECT* (Nonius, 1998), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

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supporting information

Acta Cryst. (2015). E71, 1010-1012 [https://doi.org/10.1107/S2056989015014437]

Crystal structure of 1,1'-{(pentane-1,5-diyl)bis[(azaniumylidene)methanylylidene]}bis(naphthalen-2-olate)

Kamel Ouari, Moufida Merzougui and Lydia Karmazin

Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

1,1'-{(Pentane-1,5-diyl)bis[(azaniumylidene)methanylylidene]}bis(naphthalen-2-olate)

Crystal data

$C_{27}H_{26}N_2O_2$
 $M_r = 410.50$
 Monoclinic, *P2/c*
 $a = 20.9080$ (13) Å
 $b = 4.7429$ (2) Å
 $c = 10.6810$ (6) Å
 $\beta = 96.419$ (3)°
 $V = 1052.54$ (10) Å³
 $Z = 2$

$F(000) = 436$
 $D_x = 1.295$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 7575 reflections
 $\theta = 1.0$ – 27.5°
 $\mu = 0.08$ mm⁻¹
 $T = 173$ K
 Plate, yellow
 $0.45 \times 0.20 \times 0.10$ mm

Data collection

Nonius KappaCCD
 diffractometer
 Radiation source: fine-focus sealed tube
 phi and ω scans
 Absorption correction: multi-scan
 (MULSCAN in PLATON; Spek, 2009)
 $T_{\min} = 0.792$, $T_{\max} = 1.000$
 5781 measured reflections

1958 independent reflections
 1402 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 2.9^\circ$
 $h = -25 \rightarrow 23$
 $k = -5 \rightarrow 5$
 $l = -12 \rightarrow 8$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.120$
 $S = 1.08$
 1958 reflections
 146 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.0593P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Extinction correction: SHELXL2014
 (Sheldrick, 2015),
 $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.033 (7)

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	0.14942 (5)	0.8929 (2)	-0.10611 (10)	0.0508 (4)	
N1	0.13436 (6)	0.5012 (3)	0.05060 (12)	0.0436 (4)	
H1N	0.1201 (8)	0.643 (4)	-0.0110 (19)	0.080 (6)*	
C1	0.21054 (7)	0.8695 (3)	-0.07692 (14)	0.0412 (4)	
C2	0.25371 (8)	1.0411 (3)	-0.13960 (15)	0.0497 (5)	
H2	0.2364	1.1708	-0.2022	0.060*	
C3	0.31790 (9)	1.0235 (3)	-0.11224 (17)	0.0546 (5)	
H3	0.3446	1.1413	-0.1562	0.065*	
C4	0.34731 (8)	0.8329 (3)	-0.01900 (15)	0.0469 (4)	
C5	0.41450 (8)	0.8204 (4)	0.00866 (18)	0.0612 (5)	
H5	0.4407	0.9383	-0.0362	0.073*	
C6	0.44291 (8)	0.6423 (4)	0.09873 (19)	0.0632 (5)	
H6	0.4884	0.6365	0.1169	0.076*	
C7	0.40413 (8)	0.4698 (4)	0.16322 (18)	0.0589 (5)	
H7	0.4235	0.3446	0.2258	0.071*	
C8	0.33846 (7)	0.4769 (3)	0.13821 (16)	0.0508 (5)	
H8	0.3132	0.3566	0.1841	0.061*	
C9	0.30745 (7)	0.6580 (3)	0.04622 (14)	0.0403 (4)	
C10	0.23834 (7)	0.6743 (3)	0.01610 (13)	0.0377 (4)	
C11	0.19669 (7)	0.4956 (3)	0.07450 (14)	0.0405 (4)	
H11	0.2152	0.3635	0.1348	0.049*	
C12	0.09067 (7)	0.3217 (3)	0.11165 (15)	0.0440 (4)	
H12A	0.1159	0.1903	0.1699	0.053*	
H12B	0.0644	0.2083	0.0472	0.053*	
C13	0.04669 (7)	0.4962 (3)	0.18441 (15)	0.0454 (4)	
H13A	0.0219	0.6281	0.1257	0.054*	
H13B	0.0733	0.6100	0.2482	0.054*	
C14	0.0000	0.3185 (4)	0.2500	0.0449 (6)	
H14A	-0.0247	0.1956	0.1871	0.054*	0.5
H14B	0.0247	0.1955	0.3129	0.054*	0.5

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0484 (7)	0.0583 (7)	0.0443 (7)	0.0043 (5)	-0.0014 (5)	0.0039 (5)

N1	0.0412 (8)	0.0500 (8)	0.0395 (8)	0.0013 (6)	0.0038 (6)	0.0011 (6)
C1	0.0460 (9)	0.0454 (9)	0.0314 (8)	0.0001 (7)	0.0015 (7)	-0.0083 (7)
C2	0.0615 (12)	0.0473 (9)	0.0399 (10)	-0.0025 (8)	0.0040 (8)	0.0025 (7)
C3	0.0581 (11)	0.0563 (10)	0.0504 (11)	-0.0114 (8)	0.0112 (9)	0.0009 (8)
C4	0.0462 (10)	0.0501 (10)	0.0442 (10)	-0.0049 (8)	0.0049 (7)	-0.0107 (8)
C5	0.0470 (11)	0.0745 (12)	0.0629 (12)	-0.0127 (9)	0.0096 (9)	-0.0054 (10)
C6	0.0395 (10)	0.0817 (13)	0.0674 (13)	-0.0006 (9)	0.0010 (9)	-0.0129 (11)
C7	0.0472 (10)	0.0683 (12)	0.0587 (12)	0.0052 (9)	-0.0051 (9)	-0.0027 (9)
C8	0.0434 (10)	0.0578 (10)	0.0501 (11)	0.0005 (8)	0.0008 (8)	-0.0002 (8)
C9	0.0416 (9)	0.0435 (9)	0.0356 (9)	-0.0006 (7)	0.0042 (7)	-0.0103 (7)
C10	0.0402 (8)	0.0405 (8)	0.0322 (8)	-0.0008 (7)	0.0038 (7)	-0.0059 (6)
C11	0.0407 (9)	0.0446 (9)	0.0352 (9)	0.0049 (7)	-0.0002 (7)	-0.0047 (7)
C12	0.0405 (9)	0.0463 (9)	0.0450 (10)	-0.0027 (7)	0.0035 (7)	-0.0002 (7)
C13	0.0415 (9)	0.0479 (9)	0.0467 (10)	-0.0006 (7)	0.0049 (7)	0.0007 (7)
C14	0.0375 (12)	0.0466 (12)	0.0502 (14)	0.000	0.0023 (10)	0.000

Geometric parameters (Å, °)

O1—C1	1.2858 (17)	C7—C8	1.369 (2)
N1—C11	1.2999 (19)	C7—H7	0.9500
N1—C12	1.4551 (19)	C8—C9	1.408 (2)
N1—H1N	0.96 (2)	C8—H8	0.9500
C1—C10	1.433 (2)	C9—C10	1.447 (2)
C1—C2	1.435 (2)	C10—C11	1.410 (2)
C2—C3	1.344 (2)	C11—H11	0.9500
C2—H2	0.9500	C12—C13	1.515 (2)
C3—C4	1.432 (2)	C12—H12A	0.9900
C3—H3	0.9500	C12—H12B	0.9900
C4—C5	1.404 (2)	C13—C14	1.5191 (18)
C4—C9	1.413 (2)	C13—H13A	0.9900
C5—C6	1.365 (3)	C13—H13B	0.9900
C5—H5	0.9500	C14—C13 ⁱ	1.5190 (18)
C6—C7	1.388 (3)	C14—H14A	0.9900
C6—H6	0.9500	C14—H14B	0.9900
C11—N1—C12	124.46 (14)	C8—C9—C4	116.82 (14)
C11—N1—H1N	112.0 (11)	C8—C9—C10	123.95 (14)
C12—N1—H1N	123.5 (11)	C4—C9—C10	119.23 (14)
O1—C1—C10	122.62 (14)	C11—C10—C1	118.19 (14)
O1—C1—C2	119.85 (14)	C11—C10—C9	121.36 (14)
C10—C1—C2	117.52 (14)	C1—C10—C9	120.43 (13)
C3—C2—C1	121.89 (16)	N1—C11—C10	123.79 (14)
C3—C2—H2	119.1	N1—C11—H11	118.1
C1—C2—H2	119.1	C10—C11—H11	118.1
C2—C3—C4	122.09 (16)	N1—C12—C13	110.97 (12)
C2—C3—H3	119.0	N1—C12—H12A	109.4
C4—C3—H3	119.0	C13—C12—H12A	109.4
C5—C4—C9	120.18 (16)	N1—C12—H12B	109.4

C5—C4—C3	120.99 (16)	C13—C12—H12B	109.4
C9—C4—C3	118.83 (15)	H12A—C12—H12B	108.0
C6—C5—C4	121.37 (17)	C12—C13—C14	113.06 (12)
C6—C5—H5	119.3	C12—C13—H13A	109.0
C4—C5—H5	119.3	C14—C13—H13A	109.0
C5—C6—C7	118.82 (17)	C12—C13—H13B	109.0
C5—C6—H6	120.6	C14—C13—H13B	109.0
C7—C6—H6	120.6	H13A—C13—H13B	107.8
C8—C7—C6	121.16 (18)	C13 ⁱ —C14—C13	112.58 (17)
C8—C7—H7	119.4	C13 ⁱ —C14—H14A	109.1
C6—C7—H7	119.4	C13—C14—H14A	109.1
C7—C8—C9	121.65 (16)	C13 ⁱ —C14—H14B	109.1
C7—C8—H8	119.2	C13—C14—H14B	109.1
C9—C8—H8	119.2	H14A—C14—H14B	107.8
O1—C1—C2—C3	-179.92 (15)	C3—C4—C9—C10	0.6 (2)
C10—C1—C2—C3	-0.6 (2)	O1—C1—C10—C11	2.0 (2)
C1—C2—C3—C4	0.0 (3)	C2—C1—C10—C11	-177.25 (12)
C2—C3—C4—C5	-179.42 (16)	O1—C1—C10—C9	-179.47 (13)
C2—C3—C4—C9	0.0 (2)	C2—C1—C10—C9	1.2 (2)
C9—C4—C5—C6	-0.4 (3)	C8—C9—C10—C11	-3.1 (2)
C3—C4—C5—C6	179.02 (17)	C4—C9—C10—C11	177.16 (13)
C4—C5—C6—C7	0.4 (3)	C8—C9—C10—C1	178.51 (14)
C5—C6—C7—C8	-0.3 (3)	C4—C9—C10—C1	-1.3 (2)
C6—C7—C8—C9	0.2 (3)	C12—N1—C11—C10	-178.86 (13)
C7—C8—C9—C4	-0.2 (2)	C1—C10—C11—N1	-1.3 (2)
C7—C8—C9—C10	-179.94 (15)	C9—C10—C11—N1	-179.82 (13)
C5—C4—C9—C8	0.3 (2)	C11—N1—C12—C13	117.93 (15)
C3—C4—C9—C8	-179.15 (13)	N1—C12—C13—C14	179.96 (11)
C5—C4—C9—C10	-179.95 (14)	C12—C13—C14—C13 ⁱ	-176.30 (15)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H [⋯] <i>A</i>	<i>D</i> —H	H [⋯] <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> —H [⋯] <i>A</i>
N1—H1N [⋯] O1	0.96 (2)	1.72 (2)	2.5437 (17)	141.3 (16)
C12—H12A [⋯] O1 ⁱⁱ	0.99	2.45	3.2871 (19)	142

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