

1,1'-[*m*-Phenylenebis(nitrilomethanylylidene)]dinaphthalen-2-ol–1,1'-[*m*-phenylenebis(iminomethanylylidene)]-dinaphthalen-2(1*H*)-one (0.58/0.42)

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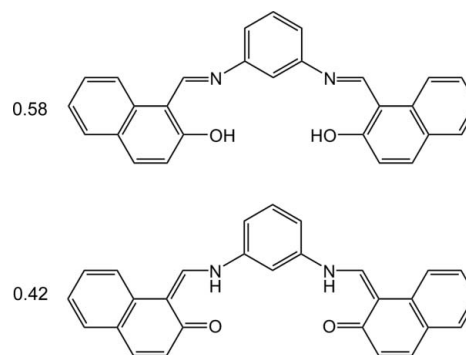
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.059; wR factor = 0.166; data-to-parameter ratio = 13.7.

In the solid state the title Schiff base, $0.58\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_2 \cdot 0.42\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_2$, exists both as the keto–imino and as the enol–amino tautomer, which is manifested in the disorder of the H atom in the intramolecular hydrogen-bonded ring. The naphthalene ring systems show some distortion, which is consistent with the quinoid effect. The ratio of the enol form refined to 58 (5)%. The molecule has crystallographically imposed symmetry: a twofold axis passes through the central benzene ring. Crystals are built up of layers parallel to (010). Stacking interactions between the layers involve only standard van der Waals attraction forces between apolar groups. The alignment of the aromatic rings in neighbouring layers shows a herringbone motif. A weak $\text{C}-\text{H} \cdots \text{O}$ interaction is observed.

Related literature

For general background to Schiff bases, see: Blagus *et al.* (2010). For applications of Schiff bases and derivatives as ligands, see: Hernández-Molina *et al.* (1997); Torayama *et al.* (1997); Elerman *et al.* (1998); Ganjali *et al.* (2008). For discussion of the quinoid effect, see: Gavranic *et al.* (1996, 1997); Frišćić *et al.* (1998). For structures with a herringbone arrangement, see: Desiraju & Gavezzotti (1989). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$0.58\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_2 \cdot 0.42\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_2$ $V = 2131.6$ (5) Å³
 $M_r = 416.46$ $Z = 4$
 Orthorhombic, *Pccn* $\text{Mo } K\alpha$ radiation
 $a = 5.4292$ (9) Å $\mu = 0.08$ mm⁻¹
 $b = 26.496$ (3) Å $T = 298$ K
 $c = 14.818$ (2) Å $0.60 \times 0.50 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur CCD 2089 independent reflections
 diffractometer 1699 reflections with $I > 2\sigma(I)$
 11523 measured reflections $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.166$ $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $S = 1.11$ $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³
 2089 reflections
 153 parameters
 1 restraint

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1} \cdots \text{N1}$	0.96	1.66	2.569 (2)	154
$\text{N1}-\text{H2} \cdots \text{O1}$	0.99	1.82	2.569 (2)	130
$\text{C13}-\text{H13} \cdots \text{O1}^{\dagger}$	0.93	2.67	3.413 (3)	137

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999), *PARST97* (Nardelli, 1995) and *Mercury* (Macrae *et al.*, 2006).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2025).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L. A. & Orpen, G. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Blagus, A., Cinčić, D., Friščić, T., Kaitner, B. & Stilinović, V. (2010). *Maced. J. Chem. Chem. Eng.* **29**, 117–138.
- Desiraju, G. R. & Gavezzotti, A. (1989). *J. Chem. Soc. Chem. Commun.* pp. 621–623.
- Elerman, Y., Kabak, M., Elmali, A. & Svoboda, I. (1998). *Acta Cryst.* **C54**, 128–130.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Friščić, T., Kaitner, B. & Meštrović, E. (1998). *Croat. Chem. Acta*, **71**, 87–98.
- Ganjali, M. R., Tavakoli, M., Faridbod, F., Riahi, S., Norouzi, P. & Salavati-Niassari, M. (2008). *Int. J. Electrochem. Sci.* **3**, 1559–1573.
- Gavranić, M., Kaitner, B. & Meštrović, E. (1996). *J. Chem. Crystallogr.* **26**, 23–28.
- Gavranić, M., Kaitner, B. & Meštrović, E. (1997). *Acta Cryst.* **C53**, 1232–1234.
- Hernández-Molina, R., Mederos, A., Gili, P., Domínguez, S., Lloret, F., Cano, J., Julve, M., Ruiz-Pérez, C. & Solans, X. (1997). *J. Chem. Soc. Dalton Trans.* pp. 4327–4334.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Oxford Diffraction (2003). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Wrocław, Poland.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Torayama, H., Nishide, T., Asada, H., Fujiwara, M. & Matsushita, T. (1997). *Polyhedron*, **16**, 3787–3794.

supporting information

Acta Cryst. (2011). E67, o2908–o2909 [doi:10.1107/S1600536811040918]

1,1'-[*m*-Phenylenebis(nitrilomethanylylidene)]dinaphthalen-2-ol–1,1'-[*m*-phenylenebis(iminomethanylylidene)]dinaphthalen-2(1*H*)-one (0.58/0.42)

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S1. Comment

Schiff bases are among the most fundamental chelating systems in coordination chemistry. They have been used extensively as ligands with numerous transition and *p*-block metals (Blagus *et al.*, 2010). The conformation of the free ligand is of interest for comparison with the coordinated one in a corresponding metal complex (Gavranić *et al.*, 1997). Schiff bases derived from *m*-phenylenediamine can coordinate only one of their two ligand nitrogen atoms to a particular metal cation, thereby facilitating the formation of binuclear complexes, in which two coordination centres are bridged by two Schiff base molecules (Hernández-Molina, *et al.*, 1997; Torayama *et al.*, 1997). The environment of the coordination centre in transition metal complexes with bidentate Schiff base ligands can be modified by attaching different substituents to the ligand. Substituents have been used for tuning both steric and electronic properties, which are important for differences in structure and reactivity (Elerman *et al.*, 1998; Ganjali *et al.*, 2008).

The title compound is a stretched, non-planar aromatic system consisting of five aromatic and two hydrogen-bonded pseudo-aromatic rings with certain possibility of electron flow through the system (Fig. 1). The molecules are present in the crystals as disordered keto- and enol tautomers in an approximate ratio of 42 (5) : 58 (5).

The shape of the molecule can be conveniently described *via* three best planes, calculated through the two terminal naphthalene fragments and the central benzene ring. Because of the imposed crystallographic twofold symmetry, both naphthalene–benzene angles are 20.66 (7)°. The angle between the two naphthalene rings is 40.58 (6)°. Two hydrogen-bond rings, closed by a strong intramolecular hydrogen-bond [$d(\text{N}\cdots\text{O}) = 2.568(2) \text{ \AA}$], separate the central benzene ring from the terminal aromatic rings.

The actual nature of the tautomeric form of the title compound could not be clearly established on the bases of the C2—O1 [1.316 (2) Å] and C11—N1 [1.308 (2) Å] bond lengths. Both bond distances are intermediate between a single and a double bond, indicating the simultaneous presence of the keto-amine and the enol-imine tautomer. [The typical values of carbon-to-oxygen bond distances in quinones and phenols are 1.279 and 1.339 Å, respectively, while the corresponding carbon-to-nitrogen distances in imines and secondary amines are 1.222 and 1.362 Å, respectively (Allen *et al.*, 1987)]. In accordance with the intermediate bond lengths, the position of the H atom in the hydrogen-bonded ring could not be determined unequivocally. Its location was represented in the δF map as a large diffuse maximum of 0.29 $e/\text{\AA}^3$, positioned closer to the oxygen atom O1 than to the nitrogen atom N1. The enol to ketone tautomer ratio was determined to be 0.58 (5) : 0.42 (5) by refinement of a disordered hydrogen atom model. No significant residual electron density was detected in the area of the two disordered hydrogen atom positions in the δF map at the end of the refinement.

The peculiar bond distance scheme in naphthalene, a special arrangement of shorter and longer bond lengths is very well known (Allen *et al.*, 1987). Generally, the same naphthalene topology applies to compounds containing substituted naphthane fragments. With oxygen substitution in position 2 of naphthalene, two forms, quinoid and benzenoid (if the

oxygen is protonated) can be recognised. The presence of the quionid form, *i.e.*, the quinoid effect is the cause of the quite short C3—C4 bond distance of 1.352 (3) Å in the title compound (Gavranić *et al.*, 1996; Friščić *et al.*, 1998). However, there is also a slightly longer than usual C—C bond present in the naphthalene fragment. The C1—C10 bond distance is 1.452 (3) Å. The corresponding bond is 1.420 (3) Å in naphthalene. There are two possible reasons for this unusual bond distance: (i) The positions of the atoms of the enol and keto tautomer do not necessarily overlap fully, which may lead to an apparent shift of C10 closer to C9, although no anomaly in the contour δF map was observed in the region of C10 carbon atom. (ii) The possibility of electron flow to the central aromatic ring and the two highly electronegative nitrogen atoms lower the electron density in the naphthalene rings and thus weaken the aromaticity in the region of the C10 atom. Shifting of the electron density from the naphthalene core may be further assisted by the fused hydrogen-bond ring.

There are 4 molecules in the unit cell of the title compound. The molecules lie in 4(*c*) special positions of the space group with a crystallographic 2-fold axis passing through atoms C14 and C14*a*, respectively. Intermolecular interactions are dominated by van der Waals forces. There is only one very weak C—H \cdots O contact [C13 \cdots O1ⁱ 3.414 (3) Å; (i): $-x + 3/2, y, z + 1/2$]. The molecular packing framework consists of infinite layers in the (010) plane having a thickness of half of the *b*-axis (Fig. 2). Parallel layers in a stack are connected *via* extremely weak C6—H6 \cdots C6ⁱⁱ interactions [d(C6 \cdots C6ⁱⁱ) = 3.668 (3) Å; (ii): $x + 1/2, -y + 1, -z + 1/2$]. The mutual orientation of the naphthalene rings involved in this interaction creates a herringbone motif. The herringbone motif is characteristic of planar aromatic systems (Desiraju & Gavezzotti, 1989) and is stabilised by electrostatic interactions.

S2. Experimental

The title compound was synthesized by applying the standard condensation procedure for the preparation of imino compounds. An ethanolic solution of 2-hydroxy-1-naphthaldehyde (10 mmol) and an ethanolic solution of *m*-phenylenediamine (5 mmol) were mixed and heated in the presence of acetic acid as catalyst. The mixture was refluxed at 331 K for 2 h. After cooling, the precipitated Schiff base was separated by vacuum filtration with a yield of 94%. A suitable single-crystal was obtained by slow recrystallization from dichloromethane solution at room temperature.

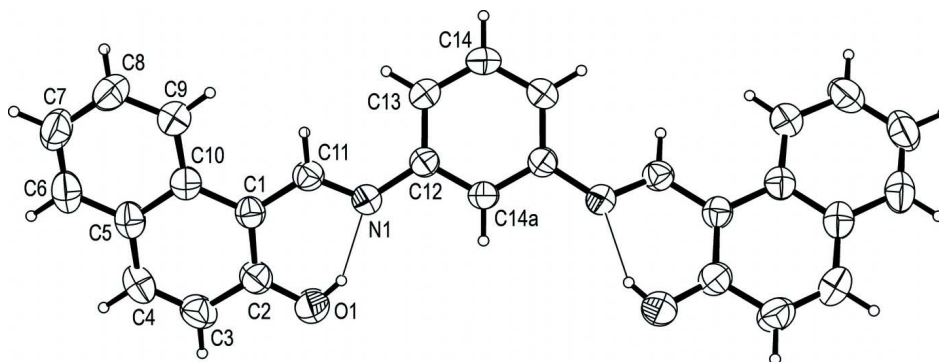
The purity of the compound was determined by NMR, FT—IR, TGA and DSC techniques. **M.p.** 506 K. **IR** / cm⁻¹: 3449, 3059, 1624, 1561, 1327, 863, 825, 750. **¹H NMR** (in CDCl₃) d/p.p.m.: 15.37 (s, 1H, OH and NH), 9.38 (s, 1H, CH=N and CH—NH), 7.11 – 8.11 (m, 9H, ar). **¹³C NMR** (in CDCl₃) d/p.p.m.: 155.26 (1 C, C=N); 146.81, 136.80, 130.67, 133.01, 129.26, 128.10, 127.30, 123.63, 121.95, 118.94, 118.11, 112.28 (12 C, ar).

S3. Refinement

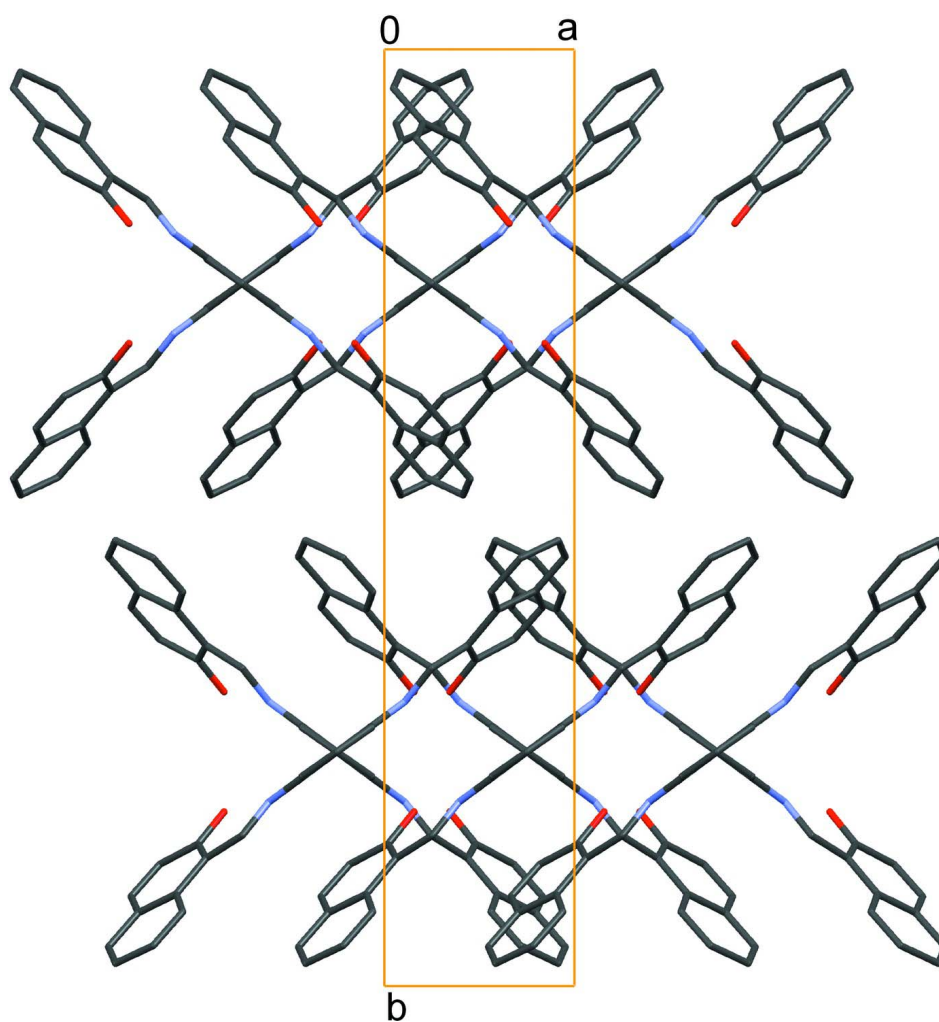
The positional parameters and the occupancies of atoms H1 and H2, which belong to the enol and keto tautomer, respectively, were refined freely. The sum of the occupancies was constrained to 1 by using the same *SHELXL* FVAR variable [*i.e.*, occupancies of fv(2) and 1-fv(2)]. U_{iso} values of both H1 and H2 were calculated as 120% of the equivalent isotropic thermal parameters of the corresponding heavy atoms. No electron density residues were observed after the refinement converged with a stable 58 (5) : 42 (5) occupancy ratio for H1 (enol tautomer) and H2 (keto tautomer), respectively.

All other hydrogen atoms were treated as riding atoms using instruction AFIX 43 (C—H 0.93 Å) with $U_{\text{iso}}(\text{H})$ being 1.2 times the equivalent isotropic thermal parameter of corresponding carbon atom.

Due to now unclear reasons, at the time of data collection in 2006 the crystal-to-detector distance was set up to 70 mm. That is the likely reason that some reflections at low theta angle were not recorded.

**Figure 1**

ORTEP-3 view of the enol tautomer of 1,3-bis(2-hydroxy-1-naphthylmethylideneamino)benzene with anisotropic thermal ellipsoids drawn at the 50% probability level. Only symmetry independent atoms are labelled.

**Figure 2**

Packing diagram projected down the *c*-axis showing the herringbone motif between layers stacked along the *b*-axis.

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Crystal data

0.58C₂₈H₂₀N₂O₂·0.42C₂₈H₂₀N₂O₂

M_r = 416.46

Orthorhombic, *Pccn*

Hall symbol: -P 2ab 2ac

a = 5.4292 (9) Å

b = 26.496 (3) Å

c = 14.818 (2) Å

V = 2131.6 (5) Å³

Z = 4

F(000) = 872

D_x = 1.298 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 2089 reflections

θ = 4.7–26°

μ = 0.08 mm⁻¹

T = 298 K

Table, yellow

0.60 × 0.50 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

11523 measured reflections

2089 independent reflections

1699 reflections with *I* > 2σ(*I*)

R_{int} = 0.035

θ_{max} = 26.0°, θ_{min} = 4.7°

h = -6→5

k = -32→32

l = -18→18

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.059

wR(*F*²) = 0.166

S = 1.11

2089 reflections

153 parameters

1 restraint

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0822*P*)² + 0.5104*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.18 e Å⁻³

Δρ_{min} = -0.15 e Å⁻³

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.

Refinement. Refinement of *F*² against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on *F*², conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative *F*². The threshold expression of *F*² > σ(*F*²) is used only for calculating *R*-factors(gt) etc. and is not relevant to the choice of reflections for refinement. *R*-factors based on *F*² are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}	Occ. (<1)
O1	0.8437 (3)	0.31403 (6)	0.12718 (11)	0.0677 (5)	
H1	0.734 (12)	0.3023 (18)	0.174 (4)	0.081*	0.58 (5)
N1	0.6078 (3)	0.30360 (6)	0.27628 (11)	0.0426 (4)	
H2	0.617 (11)	0.295 (2)	0.211 (4)	0.051*	0.42 (5)

C1	0.9376 (3)	0.36355 (7)	0.25869 (12)	0.0414 (4)
C2	0.9799 (4)	0.34832 (7)	0.16768 (13)	0.0496 (5)
C3	1.1822 (4)	0.37011 (8)	0.11911 (14)	0.0605 (6)
H3	1.2103	0.3604	0.0597	0.073*
C4	1.3328 (4)	0.40449 (8)	0.15786 (15)	0.0570 (6)
H4	1.4647	0.4172	0.1249	0.068*
C5	1.2949 (3)	0.42177 (7)	0.24800 (14)	0.0475 (5)
C6	1.4520 (4)	0.45830 (8)	0.28698 (16)	0.0589 (6)
H6	1.5839	0.4707	0.2536	0.071*
C7	1.4143 (4)	0.47577 (9)	0.37243 (16)	0.0685 (7)
H7	1.5189	0.4999	0.3970	0.082*
C8	1.2165 (5)	0.45689 (9)	0.42269 (16)	0.0695 (7)
H8	1.1896	0.4689	0.4808	0.083*
C9	1.0609 (4)	0.42084 (9)	0.38751 (14)	0.0585 (6)
H9	0.9312	0.4089	0.4224	0.070*
C10	1.0948 (3)	0.40169 (7)	0.29907 (13)	0.0433 (5)
C11	0.7478 (3)	0.33924 (6)	0.30943 (12)	0.0421 (4)
H11	0.7226	0.3492	0.3689	0.050*
C12	0.4305 (3)	0.27711 (6)	0.32839 (12)	0.0382 (4)
C13	0.4336 (4)	0.27599 (7)	0.42280 (13)	0.0519 (5)
H13	0.5573	0.2925	0.4547	0.062*
C14	0.2500	0.2500	0.46826 (19)	0.0619 (8)
H14	0.2500	0.2500	0.5310	0.074*
C14A	0.2500	0.2500	0.28224 (16)	0.0379 (5)
H14A	0.2500	0.2500	0.2195	0.046*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0791 (11)	0.0713 (11)	0.0527 (8)	-0.0218 (9)	0.0128 (8)	-0.0176 (8)
N1	0.0444 (8)	0.0424 (8)	0.0409 (8)	-0.0046 (7)	0.0010 (7)	0.0006 (7)
C1	0.0412 (9)	0.0384 (9)	0.0444 (10)	0.0004 (7)	-0.0004 (7)	0.0018 (7)
C2	0.0543 (11)	0.0461 (10)	0.0484 (11)	-0.0017 (9)	0.0059 (9)	-0.0005 (8)
C3	0.0679 (13)	0.0622 (13)	0.0514 (12)	-0.0038 (11)	0.0165 (10)	-0.0030 (10)
C4	0.0524 (11)	0.0527 (12)	0.0659 (14)	-0.0044 (9)	0.0136 (10)	0.0083 (10)
C5	0.0412 (10)	0.0404 (10)	0.0610 (12)	0.0008 (8)	-0.0041 (8)	0.0084 (9)
C6	0.0466 (11)	0.0512 (12)	0.0789 (15)	-0.0097 (9)	-0.0091 (10)	0.0152 (11)
C7	0.0681 (14)	0.0631 (14)	0.0742 (15)	-0.0208 (11)	-0.0240 (12)	0.0047 (12)
C8	0.0804 (16)	0.0723 (15)	0.0559 (13)	-0.0186 (13)	-0.0171 (12)	-0.0048 (11)
C9	0.0616 (12)	0.0628 (13)	0.0510 (12)	-0.0142 (10)	-0.0038 (10)	-0.0007 (10)
C10	0.0405 (9)	0.0405 (9)	0.0488 (10)	0.0023 (7)	-0.0052 (8)	0.0053 (8)
C11	0.0471 (10)	0.0392 (9)	0.0400 (9)	0.0004 (8)	0.0014 (8)	-0.0022 (7)
C12	0.0400 (9)	0.0356 (9)	0.0391 (9)	0.0004 (7)	0.0019 (7)	-0.0021 (7)
C13	0.0575 (11)	0.0577 (12)	0.0404 (10)	-0.0141 (10)	-0.0053 (8)	-0.0024 (8)
C14	0.079 (2)	0.074 (2)	0.0326 (13)	-0.0218 (17)	0.000	0.000
C14A	0.0450 (12)	0.0360 (12)	0.0328 (12)	0.0018 (10)	0.000	0.000

Geometric parameters (Å, °)

O1—C2	1.316 (2)	C6—H6	0.9300
O1—H1	0.96 (7)	C7—C8	1.399 (3)
N1—C11	1.308 (2)	C7—H7	0.9300
N1—C12	1.420 (2)	C8—C9	1.378 (3)
N1—H2	0.99 (6)	C8—H8	0.9300
C1—C2	1.426 (3)	C9—C10	1.417 (3)
C1—C11	1.429 (2)	C9—H9	0.9300
C1—C10	1.452 (2)	C11—H11	0.9300
C2—C3	1.434 (3)	C12—C14A	1.394 (2)
C3—C4	1.352 (3)	C12—C13	1.399 (3)
C3—H3	0.9300	C13—C14	1.386 (2)
C4—C5	1.427 (3)	C13—H13	0.9300
C4—H4	0.9300	C14—C13 ⁱ	1.386 (2)
C5—C6	1.414 (3)	C14—H14	0.9300
C5—C10	1.427 (3)	C14A—C12 ⁱ	1.394 (2)
C6—C7	1.364 (3)	C14A—H14A	0.9300
C2—O1—H1	104 (3)	C9—C8—C7	121.1 (2)
C11—N1—C12	123.14 (16)	C9—C8—H8	119.4
C11—N1—H2	120 (4)	C7—C8—H8	119.4
C12—N1—H2	117 (4)	C8—C9—C10	121.2 (2)
C2—C1—C11	119.07 (16)	C8—C9—H9	119.4
C2—C1—C10	119.47 (16)	C10—C9—H9	119.4
C11—C1—C10	121.40 (16)	C9—C10—C5	117.12 (17)
O1—C2—C1	122.40 (17)	C9—C10—C1	123.64 (17)
O1—C2—C3	118.63 (18)	C5—C10—C1	119.24 (17)
C1—C2—C3	118.94 (18)	N1—C11—C1	123.16 (17)
C4—C3—C2	121.40 (19)	N1—C11—H11	118.4
C4—C3—H3	119.3	C1—C11—H11	118.4
C2—C3—H3	119.3	C14A—C12—C13	119.18 (16)
C3—C4—C5	121.77 (18)	C14A—C12—N1	117.69 (16)
C3—C4—H4	119.1	C13—C12—N1	123.11 (16)
C5—C4—H4	119.1	C14—C13—C12	119.21 (18)
C6—C5—C10	119.87 (19)	C14—C13—H13	120.4
C6—C5—C4	121.00 (19)	C12—C13—H13	120.4
C10—C5—C4	119.12 (18)	C13—C14—C13 ⁱ	121.8 (3)
C7—C6—C5	121.4 (2)	C13—C14—H14	119.1
C7—C6—H6	119.3	C13 ⁱ —C14—H14	119.1
C5—C6—H6	119.3	C12—C14A—C12 ⁱ	121.3 (2)
C6—C7—C8	119.2 (2)	C12—C14A—H14A	119.4
C6—C7—H7	120.4	C12 ⁱ —C14A—H14A	119.4
C8—C7—H7	120.4		
C11—C1—C2—O1	2.9 (3)	C4—C5—C10—C9	-178.57 (18)
C10—C1—C2—O1	-179.85 (18)	C6—C5—C10—C1	-178.92 (16)
C11—C1—C2—C3	-175.43 (17)	C4—C5—C10—C1	1.1 (3)

C10—C1—C2—C3	1.8 (3)	C2—C1—C10—C9	177.18 (18)
O1—C2—C3—C4	-178.2 (2)	C11—C1—C10—C9	-5.6 (3)
C1—C2—C3—C4	0.2 (3)	C2—C1—C10—C5	-2.5 (3)
C2—C3—C4—C5	-1.6 (3)	C11—C1—C10—C5	174.74 (16)
C3—C4—C5—C6	-179.0 (2)	C12—N1—C11—C1	175.63 (15)
C3—C4—C5—C10	0.9 (3)	C2—C1—C11—N1	-0.5 (3)
C10—C5—C6—C7	-1.3 (3)	C10—C1—C11—N1	-177.74 (16)
C4—C5—C6—C7	178.7 (2)	C11—N1—C12—C14A	163.40 (14)
C5—C6—C7—C8	0.3 (3)	C11—N1—C12—C13	-18.4 (3)
C6—C7—C8—C9	0.4 (4)	C14A—C12—C13—C14	-3.2 (2)
C7—C8—C9—C10	-0.2 (4)	N1—C12—C13—C14	178.67 (14)
C8—C9—C10—C5	-0.7 (3)	C12—C13—C14—C13 ⁱ	1.59 (12)
C8—C9—C10—C1	179.7 (2)	C13—C12—C14A—C12 ⁱ	1.58 (12)
C6—C5—C10—C9	1.4 (3)	N1—C12—C14A—C12 ⁱ	179.86 (16)

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...N1	0.96	1.66	2.569 (2)	154
N1—H2...O1	0.99	1.82	2.569 (2)	130
C13—H13...O1 ⁱⁱ	0.93	2.67	3.413 (3)	137

Symmetry code: (ii) $-x+3/2, y, z+1/2$.