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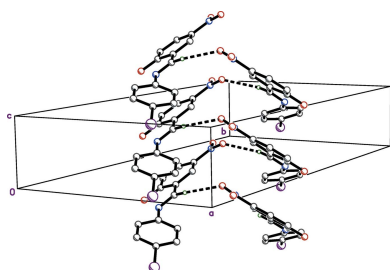
Crystal structure and Hirshfeld surface analysis of 2-[[4-iodophenyl]imino]methyl]-4-nitrophenol

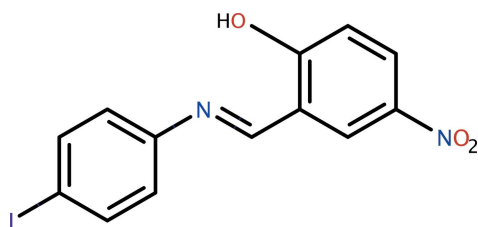
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The title compound, C₁₃H₉IN₂O₃, was synthesized by a condensation reaction between 2-hydroxy-5-nitrobenzaldehyde and 4-iodoaniline, and crystallizes in the orthorhombic space group *Pna*2₁. The 4-iodobenzene ring is inclined to the phenol ring by a dihedral angle of 39.1 (2)°. The configuration about the C=N double bond is *E*. The crystal structure features C—H···O hydrogen-bonding interactions. A Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the packing arrangement are O···H/H···O (26.9%) and H···H (22.0%) interactions.

1. Chemical context

Over the past 25 years, extensive research has been directed towards the synthesis and use of Schiff base compounds in organic and inorganic chemistry as they have important medicinal and pharmaceutical applications. These compounds exhibit biological activities, including antibacterial, antifungal, anticancer and herbicidal properties (Desai *et al.*, 2001; Singh & Dash, 1988; Karia & Parsania, 1999). They may also show useful photochromic properties, leading to applications in various fields such as the measurement and control of radiation intensities in imaging systems and optical computers, electronics, optoelectronics and photonics (Iwan *et al.*, 2007). Schiff bases derived from 2-hydroxy-5-nitrobenzaldehyde are widely used either as materials or as intermediates in explosives, dyestuffs, pesticides and organic synthesis (Yan *et al.*, 2006). Intramolecular hydrogen-atom transfer (tautomerism) from the *o*-hydroxy group to the imine-N atom is of prime importance with respect to the solvato-, thermo- and photochromic properties of *o*-hydroxy Schiff bases (Filarowski, 2005; Hadjoudis & Mavridis 2004). Such proton-exchanging materials can be utilized for the design of various molecular electronic devices (Alarcón *et al.*, 1999). The present work is a part of an ongoing structural study of Schiff bases and their utilization in the synthesis of quinoxaline derivatives (Faizi *et al.*, 2018), fluorescence sensors (Faizi *et al.*, 2016; Mukherjee *et al.*, 2018; Kumar *et al.*, 2017; 2018) and non-linear optical properties (Faizi *et al.*, 2020). We report herein the synthesis (from 2-hydroxy-5-nitrobenzaldehyde and 4-iodoaniline) and crystal structure of the title compound (I), along with the findings of a Hirshfeld surface analysis.





2. Structural commentary

The molecular structure of compound (I) is shown in Fig. 1. An intramolecular O—H···N hydrogen bond is observed (Table 1 and Fig. 1). This is a relatively common feature in analogous imine–phenol compounds (see *Database survey* section). The imine group displays a C8—C7—N1—C4 torsion angle of 174.5 (6)°. The 4-iodobenzene ring (C1—C6) is inclined by a dihedral angle of 39.1 (2)° to the phenol ring (C8—C13), which renders the molecule non-planar. The configuration of the C7=N1 bond of this Schiff base is *E*, and the intramolecular O1—H1···N1 hydrogen bond forms an *S*(6) ring motif (Fig. 1 and Table 1). The 4-nitro group is slightly tilted away from co-planarity with the benzene ring to which it is attached [O2—N2—C10—C9 = −7.4 (10)° and O3—N2—C10—C11 = −7.4 (10)°]. The C13—O1 distance [1.330 (7) Å] is close to normal for values reported for single C—O bonds in phenols and salicylideneamines (Ozeryanskii *et al.*, 2006). The N1=C7 bond is short at 1.264 (8) Å, indicative of double-bond character, while the long C7—C8 bond [1.444 (8) Å] implies a single bond. All these data support the existence of the phenol–imine tautomer for (I) in its crystal-line state. These features are similar to those observed in related 4-dimethylamino-*N*-salicylideneanilines (Filipenko *et al.*, 1983; Aldoshin *et al.*, 1984; Wozniak *et al.*, 1995; Pizzala *et al.*, 2000). The C—N, C=N and C—C bond lengths are normal and close to the values observed in related structures (Faizi *et al.*, 2017a,b).

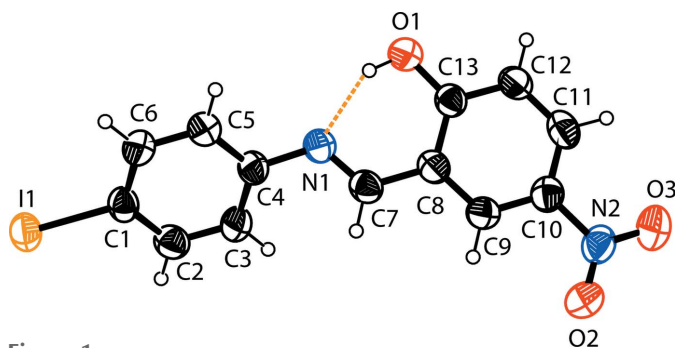


Figure 1

The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 40% probability level. The intramolecular N—H···O hydrogen bond (see Table 1), forming an *S*(6) ring motif, is shown as a dashed line.

Table 1
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>
O1—H1···N1	0.82	1.86	2.591 (6)	148
C7—H7···O2 ⁱ	0.93	2.45	3.309 (8)	154

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

3. Supramolecular features

In the crystal packing of (I), the most important intermolecular interactions are weak C7—H7···O2ⁱ [symmetry code: (i) $1 - x, 1 - y, -\frac{1}{2} + z$] hydrogen bonds between screw-related molecules, which form helical chains propagating along the crystallographic screw axis parallel to *c* (Fig. 2, Table 1). The shortest intermolecular contact involving the iodine is 3.351 (5) Å, between glide-related molecules, I1···O1ⁱⁱ [symmetry code: (ii) $x + \frac{1}{2}, \frac{1}{2} - y, -1 + z$], which makes a zigzag tape motif (Fig. 3). There are no other

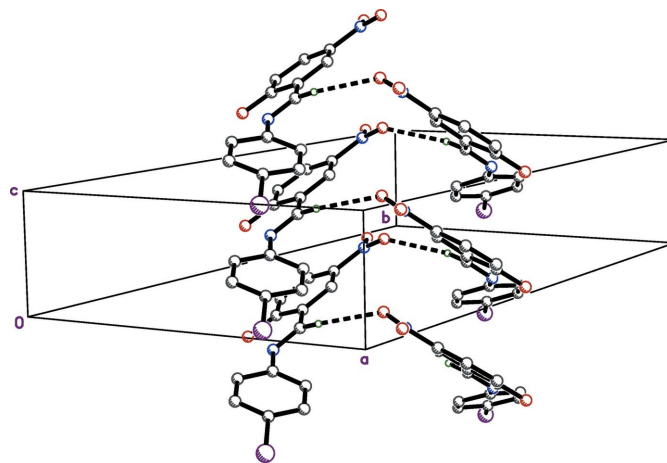


Figure 2

A partial packing plot showing the C—H···O hydrogen-bonded (thick dashed lines) helical chains about the crystallographic 2_1 screw axis parallel to *c*.

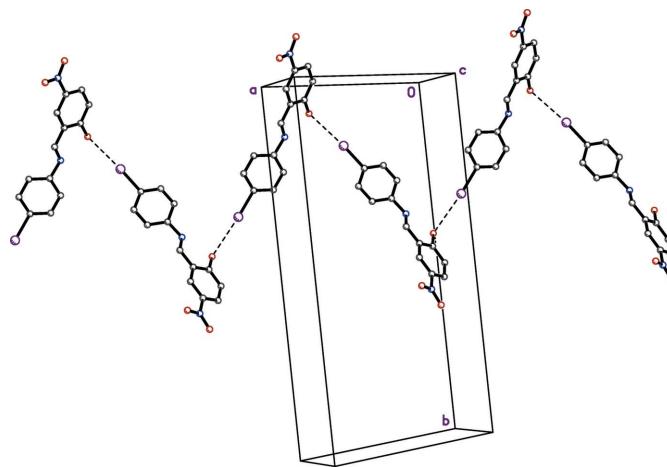


Figure 3

A partial packing plot showing close contacts (dashed lines) between iodine and the phenolic oxygen of glide-related ($x + \frac{1}{2}, \frac{1}{2} - y, -1 + z$) molecules.

significant intermolecular interactions present in the crystal. The Hirshfeld surface analysis confirms the role of the C—H...O interactions in the packing arrangement.

4. Hirshfeld surface analysis

In order to visualize the intermolecular interactions in the crystal packing of (I), a Hirshfeld surface (HS) analysis (Hirshfeld, 1977; Spackman & Jayatilaka, 2009) was carried out using *Crystal Explorer 17.5* (Turner *et al.*, 2017). In the HS plotted over d_{norm} (Fig. 4), white surfaces indicate contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter (*i.e.*, in close contact) or longer than the van der Waals radii sum, respectively (Venkatesan *et al.*, 2016). The two-dimensional fingerprint plots are depicted in Fig. 5. The O...H/H...O (26.9%) interactions form the majority of contacts, with H...H (22.0%) interactions representing the next highest contribution. The percentage contributions of other interactions are: I...H/H...I (16.3%), C...H/H...C (10.5%), C...C (8.7%), O...C/C...O (4.7%), N...C/C...N (3.8%), I...C/C...I (2.3%), H...N/N...H (1.4%), I...O/O...I (2.0%), I...N/N...I (0.6%), I...I (0.5%), O...N/N...O (0.2%), N...N (0.1%) and O...O (0.1%).

5. Database survey

A search of the Cambridge Structural Database (CSD, version 5.39; Groom *et al.*, 2016) gave 26 hits for the (*E*)-2-[[4-iodophenyl]imino]methyl]-phenol fragment. Of these 26, the most similar to (I), are as follows. In *p*-iodo-*N*-(*p*-cyanobenzylidene)aniline (LALMEQ; Ojala *et al.*, 1999), the OH group is absent and the NO₂ group is replaced by a cyano group. In (*E*)-5-(diethylamino)-2-[[4-iodophenylimino]methyl]phenol (VEFPED; Kaştaş *et al.*, 2012), the NO₂ is replaced by an *N,N* diethyl group. In *N*-(3,5-di-*tert*-butylsalicylidene)-4-iodobenzene; (MILFET; Spangenberg *et al.*, 2007), the NO₂ group is absent but a pair of ^tBu groups occupy the 3,5 positions of the salicylidene group. In 2-[[4-iodophenyl]imino]methyl]-6-

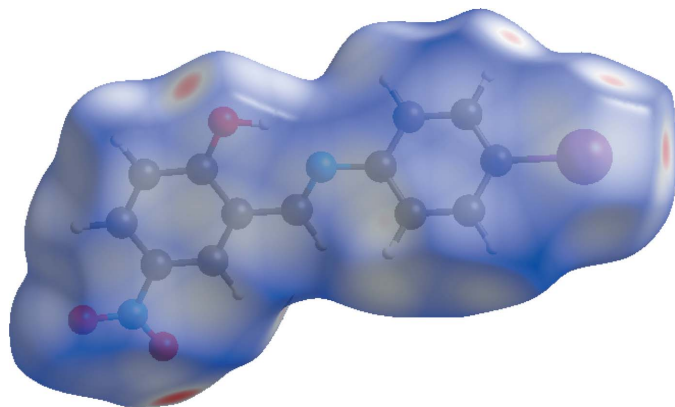


Figure 4
Hirshfeld surface of the title compound plotted over d_{norm} .

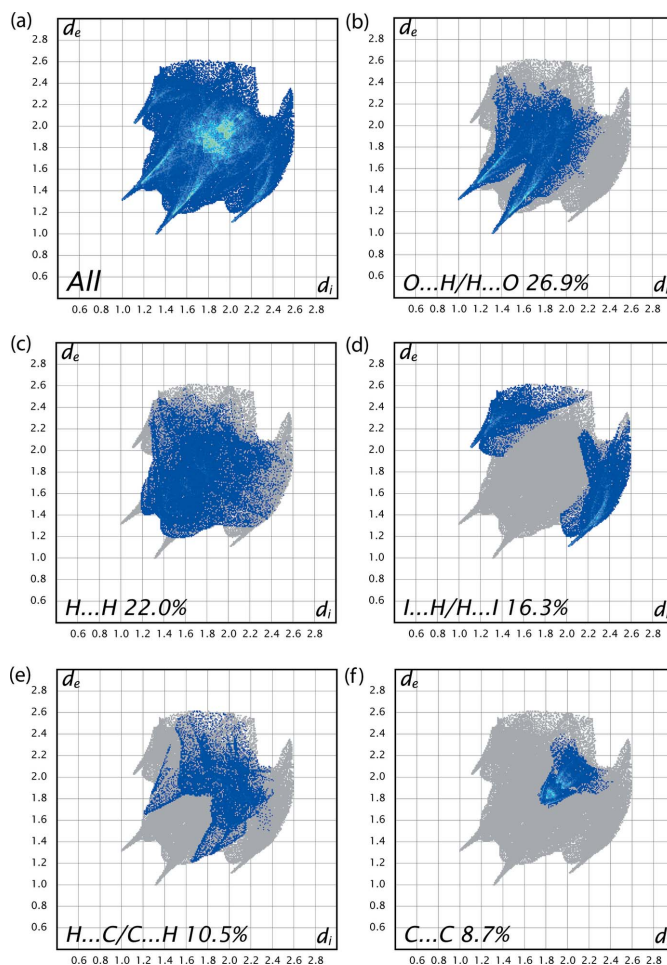


Figure 5
Two-dimensional fingerprint plots of the crystal with the relative contributions of the atom pairs to the Hirshfeld surface along with d_{norm} full.

methoxyphenol (SEDBIP; Carletta, *et al.*, 2017), the NO₂ group is absent and a methoxy group is *ortho* to the hydroxyl. Lastly, in *N*-(2-cyanobenzylidene)-4-iodoaniline (XOXXIF; Ojala *et al.*, 1999) the NO₂ is absent and the OH is replaced by cyano. All these compounds have an *E* configuration about the C=N bond and form the *S*(6) ring motif.

6. Synthesis and crystallization

The title compound was synthesized by condensation of 2-hydroxy-5-nitrobenzaldehyde (11.0 mg, 0.066 mmol) and 4-iodoaniline (14.4 mg, 0.066 mmol) in ethanol (15 ml). After the mixture had refluxed for about 15 h, the orange product was washed with ether and dried at room temperature (yield 60%, m.p. 484–486 K). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The OH hydrogen atoms and the

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₃ H ₉ IN ₂ O ₃
<i>M_r</i>	368.12
Crystal system, space group	Orthorhombic, <i>Pna</i> 2 ₁
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	12.8022 (4), 24.4556 (9), 4.1459 (1)
<i>V</i> (Å ³)	1298.02 (7)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm ⁻¹)	2.47
Crystal size (mm)	0.42 × 0.34 × 0.21
Data collection	
Diffraction	Stoe IPDS 2
Absorption correction	Integration (<i>X-RED32</i> ; Stoe & Cie, 2002)
<i>T_{min}</i> , <i>T_{max}</i>	0.944, 0.981
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	15403, 2508, 2231
<i>R_{int}</i>	0.084
(sin θ/λ) _{max} (Å ⁻¹)	0.617
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.037, 0.094, 1.05
No. of reflections	2508
No. of parameters	173
No. of restraints	1
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.81, -0.25
Absolute structure	Flack <i>x</i> determined using 814 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.00 (4)

Computer programs: *X-AREA* and *X-SHAPE* (Stoe & Cie, 2002), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *XP in SHELXTL* (Sheldrick, 2008).

C-bound H atoms were included in calculated positions and allowed to ride on the parent atoms: O–H = 0.82 Å, C–H = 0.93–0.96 Å with *U*_{iso}(H) = 1.5*U*_{eq}(C-methyl) and 1.2*U*_{eq}(C) for other H atoms.

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Crystal structure and Hirshfeld surface analysis of 2-[[4-iodophenyl]imino]-methyl]-4-nitrophenol

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-SHAPE* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXT2018/2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *XP* in *SHELXTL* (Sheldrick, 2008).

2-[[4-Iodophenyl]imino]methyl]-4-nitrophenol

Crystal data

$C_{13}H_9IN_2O_3$

$M_r = 368.12$

Orthorhombic, *Pna2₁*

$a = 12.8022$ (4) Å

$b = 24.4556$ (9) Å

$c = 4.1459$ (1) Å

$V = 1298.02$ (7) Å³

$Z = 4$

$F(000) = 712$

$D_x = 1.884$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25449 reflections

$\theta = 1.7$ – 29.9°

$\mu = 2.47$ mm⁻¹

$T = 296$ K

Prism, colorless

$0.42 \times 0.34 \times 0.21$ mm

Data collection

STOE IPDS 2

diffractometer

Radiation source: sealed X-ray tube, 12 x 0.4

mm long-fine focus

Plane graphite monochromator

Detector resolution: 6.67 pixels mm⁻¹

rotation method scans

Absorption correction: integration

(X-RED32; Stoe & Cie, 2002)

$T_{\min} = 0.944$, $T_{\max} = 0.981$

15403 measured reflections

2508 independent reflections

2231 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.084$

$\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 1.8^\circ$

$h = -15 \rightarrow 15$

$k = -30 \rightarrow 30$

$l = -5 \rightarrow 4$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.094$

$S = 1.05$

2508 reflections

173 parameters

1 restraint

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.81$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ e Å⁻³

Absolute structure: Flack x determined using
814 quotients $[(F^-)-(F)]/[(F^+)+(F)]$ (Parsons *et al.*,
2013)
Absolute structure parameter: 0.00 (4)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
II	0.50544 (3)	0.16672 (2)	-0.1482 (5)	0.06914 (19)
O1	0.1141 (3)	0.43513 (17)	0.4285 (13)	0.0721 (13)
H1	0.148899	0.407604	0.393280	0.108*
N1	0.2784 (3)	0.37325 (18)	0.4176 (13)	0.0605 (13)
C8	0.2751 (4)	0.4584 (2)	0.6892 (16)	0.0575 (13)
C1	0.4310 (5)	0.2352 (2)	0.0459 (14)	0.0578 (12)
C9	0.3293 (4)	0.4960 (2)	0.875 (2)	0.0614 (12)
H9	0.397472	0.488588	0.939016	0.074*
C13	0.1701 (4)	0.4702 (2)	0.6025 (17)	0.0564 (12)
C11	0.1808 (4)	0.5566 (2)	0.870 (2)	0.0688 (15)
H11	0.151047	0.589917	0.926484	0.083*
C6	0.3291 (5)	0.2321 (2)	0.1458 (18)	0.0689 (16)
H6	0.293084	0.199177	0.130244	0.083*
C10	0.2824 (4)	0.5444 (2)	0.9636 (16)	0.0618 (15)
O3	0.3034 (5)	0.6282 (2)	1.207 (2)	0.120 (3)
N2	0.3421 (4)	0.5833 (2)	1.1596 (16)	0.0751 (16)
C7	0.3254 (4)	0.4086 (2)	0.5862 (18)	0.0592 (12)
H7	0.394439	0.402274	0.645609	0.071*
C3	0.4358 (5)	0.3296 (2)	0.1943 (18)	0.0654 (15)
H3	0.472183	0.362411	0.211171	0.079*
C12	0.1245 (4)	0.5192 (3)	0.6944 (17)	0.0655 (15)
H12	0.055800	0.526809	0.636953	0.079*
C2	0.4847 (4)	0.2839 (3)	0.067 (2)	0.0679 (16)
H2	0.553487	0.286056	-0.003196	0.082*
C4	0.3331 (4)	0.3266 (2)	0.2959 (17)	0.0575 (15)
C5	0.2798 (4)	0.2777 (2)	0.2694 (15)	0.0660 (18)
H5	0.210481	0.275532	0.335185	0.079*
O2	0.4261 (4)	0.5694 (2)	1.2640 (15)	0.0946 (19)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.0821 (3)	0.0597 (3)	0.0656 (3)	0.01321 (13)	-0.0044 (3)	-0.0083 (2)
O1	0.0590 (19)	0.063 (2)	0.094 (4)	0.0028 (16)	-0.007 (2)	-0.007 (2)
N1	0.061 (2)	0.051 (2)	0.069 (4)	0.0033 (17)	-0.004 (2)	0.002 (2)

C8	0.054 (3)	0.052 (3)	0.066 (4)	0.002 (2)	0.007 (3)	0.004 (2)
C1	0.071 (3)	0.049 (3)	0.054 (3)	0.009 (2)	-0.006 (3)	0.002 (2)
C9	0.054 (2)	0.060 (3)	0.070 (4)	0.0001 (18)	-0.001 (4)	-0.001 (3)
C13	0.056 (3)	0.049 (3)	0.064 (3)	0.001 (2)	0.004 (3)	0.003 (3)
C11	0.067 (3)	0.058 (3)	0.081 (4)	0.007 (2)	0.022 (4)	0.000 (4)
C6	0.070 (3)	0.054 (3)	0.083 (5)	-0.002 (2)	-0.006 (3)	-0.007 (3)
C10	0.064 (3)	0.056 (3)	0.066 (4)	-0.006 (2)	0.010 (2)	-0.001 (2)
O3	0.111 (4)	0.077 (3)	0.171 (8)	0.001 (3)	-0.007 (4)	-0.049 (4)
N2	0.074 (3)	0.066 (3)	0.085 (5)	-0.015 (2)	0.014 (3)	-0.015 (3)
C7	0.057 (3)	0.052 (3)	0.068 (3)	0.004 (2)	-0.001 (3)	0.008 (3)
C3	0.068 (3)	0.049 (3)	0.079 (4)	-0.004 (2)	0.001 (3)	-0.004 (2)
C12	0.054 (3)	0.061 (3)	0.081 (4)	0.006 (2)	0.003 (3)	-0.001 (3)
C2	0.059 (3)	0.069 (4)	0.075 (5)	0.002 (2)	0.005 (3)	0.003 (4)
C4	0.062 (2)	0.046 (2)	0.064 (5)	0.0059 (19)	-0.005 (3)	0.002 (2)
C5	0.060 (3)	0.060 (3)	0.078 (5)	0.003 (2)	-0.003 (3)	-0.005 (3)
O2	0.072 (2)	0.088 (3)	0.124 (6)	-0.012 (2)	-0.008 (3)	-0.028 (3)

Geometric parameters (Å, °)

II—C1	2.089 (5)	C11—H11	0.9300
O1—C13	1.330 (7)	C6—C5	1.381 (8)
O1—H1	0.8200	C6—H6	0.9300
N1—C7	1.264 (8)	C10—N2	1.466 (8)
N1—C4	1.430 (7)	O3—N2	1.220 (8)
C8—C9	1.385 (9)	N2—O2	1.208 (8)
C8—C13	1.421 (7)	C7—H7	0.9300
C8—C7	1.444 (8)	C3—C4	1.383 (8)
C1—C6	1.371 (9)	C3—C2	1.385 (10)
C1—C2	1.377 (9)	C3—H3	0.9300
C9—C10	1.377 (7)	C12—H12	0.9300
C9—H9	0.9300	C2—H2	0.9300
C13—C12	1.388 (8)	C4—C5	1.381 (8)
C11—C12	1.373 (10)	C5—H5	0.9300
C11—C10	1.389 (9)		
C13—O1—H1	109.5	C11—C10—N2	120.2 (5)
C7—N1—C4	120.5 (5)	O2—N2—O3	123.8 (6)
C9—C8—C13	118.7 (5)	O2—N2—C10	118.8 (5)
C9—C8—C7	120.1 (5)	O3—N2—C10	117.4 (6)
C13—C8—C7	121.2 (5)	N1—C7—C8	121.9 (5)
C6—C1—C2	120.2 (6)	N1—C7—H7	119.1
C6—C1—H1	120.4 (4)	C8—C7—H7	119.1
C2—C1—H1	119.4 (4)	C4—C3—C2	120.2 (5)
C10—C9—C8	120.0 (5)	C4—C3—H3	119.9
C10—C9—H9	120.0	C2—C3—H3	119.9
C8—C9—H9	120.0	C11—C12—C13	120.0 (5)
O1—C13—C12	118.6 (5)	C11—C12—H12	120.0
O1—C13—C8	121.1 (5)	C13—C12—H12	120.0

C12—C13—C8	120.2 (5)	C1—C2—C3	119.8 (6)
C12—C11—C10	119.8 (5)	C1—C2—H2	120.1
C12—C11—H11	120.1	C3—C2—H2	120.1
C10—C11—H11	120.1	C5—C4—C3	119.4 (5)
C1—C6—C5	120.1 (6)	C5—C4—N1	118.5 (5)
C1—C6—H6	119.9	C3—C4—N1	122.1 (5)
C5—C6—H6	119.9	C6—C5—C4	120.2 (5)
C9—C10—C11	121.2 (6)	C6—C5—H5	119.9
C9—C10—N2	118.6 (5)	C4—C5—H5	119.9
C13—C8—C9—C10	-1.8 (10)	C4—N1—C7—C8	174.5 (6)
C7—C8—C9—C10	177.8 (7)	C9—C8—C7—N1	179.8 (6)
C9—C8—C13—O1	-179.1 (6)	C13—C8—C7—N1	-0.5 (10)
C7—C8—C13—O1	1.3 (9)	C10—C11—C12—C13	-1.9 (11)
C9—C8—C13—C12	2.0 (9)	O1—C13—C12—C11	-179.1 (7)
C7—C8—C13—C12	-177.7 (6)	C8—C13—C12—C11	-0.1 (10)
C2—C1—C6—C5	-0.2 (10)	C6—C1—C2—C3	0.9 (11)
I1—C1—C6—C5	-179.0 (5)	I1—C1—C2—C3	179.7 (6)
C8—C9—C10—C11	-0.1 (10)	C4—C3—C2—C1	-0.7 (12)
C8—C9—C10—N2	-179.9 (6)	C2—C3—C4—C5	0.0 (11)
C12—C11—C10—C9	2.1 (11)	C2—C3—C4—N1	-177.7 (7)
C12—C11—C10—N2	-178.2 (7)	C7—N1—C4—C5	146.2 (7)
C9—C10—N2—O2	-7.4 (10)	C7—N1—C4—C3	-36.1 (10)
C11—C10—N2—O2	172.8 (7)	C1—C6—C5—C4	-0.6 (10)
C9—C10—N2—O3	172.4 (7)	C3—C4—C5—C6	0.7 (10)
C11—C10—N2—O3	-7.4 (10)	N1—C4—C5—C6	178.5 (6)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots N1	0.82	1.86	2.591 (6)	148
C7—H7 \cdots O2 ⁱ	0.93	2.45	3.309 (8)	154

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