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A new crystal form and antimicrobial activity of (*E*)-1-[3-(2-hydroxybenzylideneamino)phenyl]-ethanone

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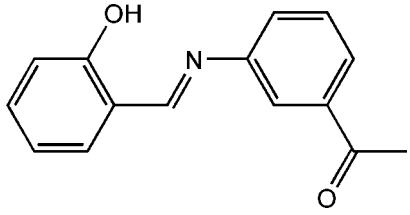
The title Schiff base compound, $C_{15}H_{13}NO_2$, crystallizes in a new crystal form in the space group $P2_12_12_1$, which is different from the monoclinic $P2_1/n$ space group reported previously [De *et al.* (2009). *Indian J. Chem. Sect. B*, **48**, 595–598]. An intramolecular O—H···N hydrogen bond occurs between the hydroxy and azomethine moieties. In the crystal, molecules are linked by weak C—H···O hydrogen bonds into supramolecular chains propagating along the *b*-axis direction with a $C(8)$ graph-set motif. The contribution of these two contacts in Hirshfeld surface area are around 19 and 21%, respectively. The title compound was screened for its antibacterial activity against two gram-negative (*Escherichia coli* and *Salmonella typhimurium*) and one gram-positive (*Staphyloccus aureus)* bacteria. The results of this study reveal that this Schiff base shows good activity against only one bacterium, *i.e. Salmonella typhimurium*.

1. Chemical context

Schiff bases (Wang *et al.*, 2008) are versatile ligands synthesized from the condensation of primary amines with carbonyl groups. These compounds have been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, anti-malarial, antiproliferative, anti-inflammatory, antiviral and antipyretic properties (Dhar & Taploo, 1982; Przybylski *et al.*, 2009). Imine or azomethine groups are present in various natural, natural-derived and non-natural compounds and have been shown to be critical for their biological activity (Bringmann *et al.*, 2004; de Souza *et al.*, 2007).



In this paper, we report the structural characterization using X-ray diffraction of the title Schiff base derived from salicylaldehyde, including an investigation of the Hirshfeld surfaces and its antimicrobial activity against two gram-negative (*Escherichia coli* and *Salmonella* typhimurium) and one gram-positive (Staphyloccus aureus*)* bacteria. Schiff bases derived from this benzaldehyde are members of one of the most commonly investigated classes of compound, and have attracted the interest of chemists and physicists because they show photochromism and thermochromism in the solid state.

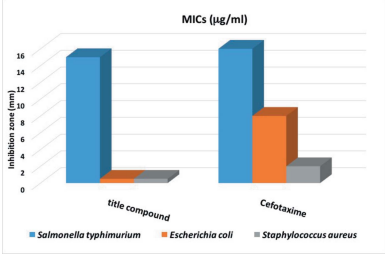


Table 1
Selected geometric parameters (Å, °).

O1—C9	1.349 (3)	N1—C1	1.415 (3)
O2—C14	1.213 (3)	N1—C7	1.293 (3)
C1—N1—C7	121.25 (18)	O1—C9—C10	119.10 (19)
N1—C1—C2	115.86 (19)	O1—C9—C8	121.3 (2)
N1—C1—C6	125.3 (2)	O2—C14—C15	120.9 (2)
N1—C7—C8	120.8 (2)	O2—C14—C3	120.5 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.84	1.84	2.590 (2)	148
C7—H7···O2^{i}	0.95	2.40	3.321 (3)	162

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

These photo- and thermochromic features are caused by proton transfer to the N atom from the O atom under the influence of light or temperature, respectively. It has been proposed that molecules showing thermochromism are planar and those showing photochromism are non-planar (Moustakali-Mavridis *et al.*, 1980; Hadjoudis *et al.*, 1987).

2. Structural commentary

The title Schiff base (Fig. 1) consists of two aromatic phenyl rings linked *via* an azomethine (C═N) group. The two phenyl rings are monosubstituted by a hydroxyl group on the same side as the azomethine carbon atom and by an aceto group on the other side. Relevant bond distances and angles are in good agreement with those reported in similar Schiff base compounds (Benarous *et al.*, 2016; Chen *et al.*, 2011). The C1—N1—C7—C8 torsion angle of −179.4 (2)° indicates an almost planar *E* configuration with respect to the imine C═N bond, as expected for a compound having an azomethine HC═N bond as this is the most thermodynamically stable configuration (Ciciani *et al.*, 2008). The N1═C7 [1.293 (3) Å] and C9—C10 [1.394 (3) Å] bond distances indicate that the compound adopts the phenol–imine tautomeric form with an N═C double bond and a C—C single bond (Table 1). Comparable values are observed in Schiff bases obtained from the same salicylaldehyde derivative with phenol–imine tautomeric form (Albayrak *et al.*, 2010; Şahin *et al.*, 2009).

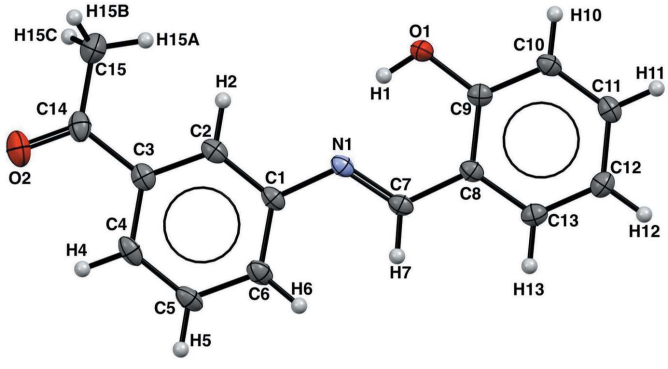


Figure 1
View of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

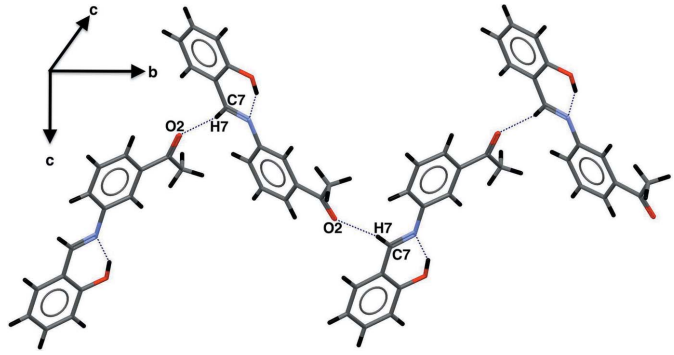


Figure 2
C—H···O hydrogen bonds (Table 1) in the title compound.

An intramolecular O—H···N hydrogen bond (Table 2) occurs between the O-hydroxyl and N azomethine atoms, forming an *S*(6) ring motif. Such a hydrogen bond is frequently observed in Schiff bases derived from salicylaldehyde (Alpaslan *et al.*, 2011).

3. Supramolecular features

In the crystal, the molecules are linked *via* C—H···O hydrogen bonds between the carbon atom of azomethine group and the oxygen atom of the methoxy substituent, generating infinite chains with graph-set motif *C*(8) along the *b*-axis direction (Table 2, Fig. 2). The chains are linked *via* *π*–*π* interactions (3.535 Å) between the C8–C13 benzene ring and the C═N double bond (Fig. 3).

4. Hirshfeld surfaces analysis

Hirshfeld surfaces and two-dimensional fingerprint plots were generated by *CrystalExplorer* 3.1 (Wolff *et al.*, 2012) to visualize and explore the intermolecular interactions. These molecular surfaces reflect intermolecular contacts based on colour coding distances from the surface to the nearest atom exterior (d_e) or interior (d_i) to the surface. In the Hirshfeld

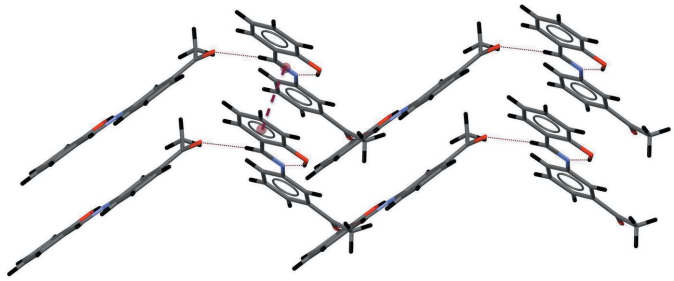


Figure 3
π–*π* interaction between a benzene ring and the C═N double bond.

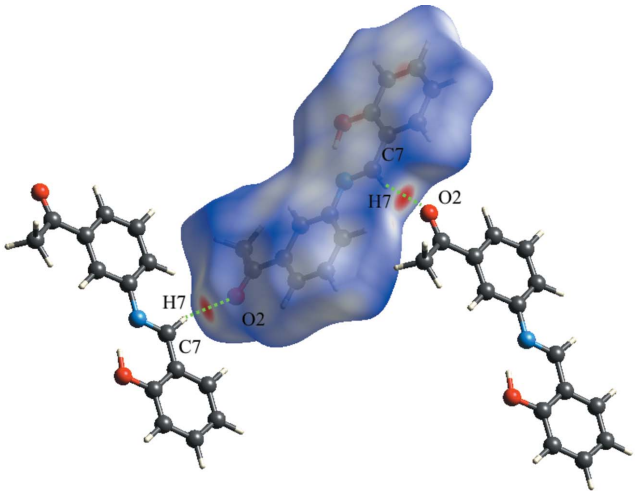


Figure 4
Hirshfeld surface of the title compound mapped over d_{norm} (−0.60 to 0.90 a.u.).

surface mapped over d_{norm} (Fig. 4), red indicates the presence of short contacts and white represents contacts around the van der Waals separation, while the blue areas are completely devoid of close contacts. The intermolecular interactions were analysed by a combination of 3D Hirshfeld surfaces and 2D fingerprint plots, showing that the intermolecular H···H contacts make the largest contribution, corresponding to 46% of the total Hirshfeld surface area (Fig. 5). The presence of short intermolecular H···H contacts is observed in the vicinity of 2.30 Å. These contacts are manifested as white spots on the d_{norm} surface and are considered to be weak interactions. In the fingerprint plots (Fig. 6), the C···H/H···C contacts, representing 21.6% of the total Hirshfeld surface, appear as two short spikes. The red spots on the d_{norm} surface in Fig. 4 are due to the CH···O contacts corresponding to the C—H···O hydrogen bond. The O···H contacts (19.4% of the total Hirshfeld surface) show up as a sharp spike in the fingerprint plots at $d_e + d_i \simeq 2.3$ Å. Finally, the packing cohesion in this structure is also provided by C···N and C···C interactions, which correspond to π–π stacking interactions.

5. Database survey

A search for the title compound in the Cambridge Structural Database (Version 2.39; Groom *et al.*, 2016) revealed that the crystal structure of the title compound had been previously been reported in the monoclinic $P2_1/n$ space group (De *et al.*, 2009). The latter differs from the title structure at position 3 of

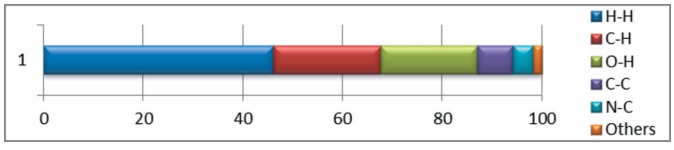


Figure 5
Relative contributions of various interactions to the Hirshfeld surface area.

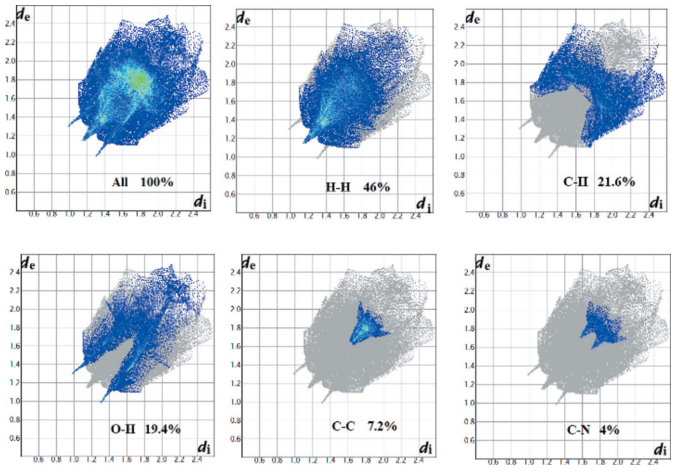


Figure 6
Two-dimensional fingerprints of the compound, showing all interactions and H···H, C···H, O···H, C···C and C···N contacts.

the aceto substituent, which is on the same side of the hydroxyl group in the title compound and in the opposite side in the reported one. This difference in position directly affects the hydrogen-bonding pattern. Similar infinite C—H···O chains occur in both compounds but the angle between linked molecules is *ca* 67.99° in the title compound and 77.61° in the reported one. The CSD search also found seven hits for structures containing the title molecule but with additional substituents (a methoxy or an additional hydroxyl group).

6. Antimicrobial activity

The title compound was screened for its antibacterial activity against two gram-negative (*Escherichia coli* and *Salmonella typhimurium*) and one gram-positive (*Staphyloccus aureus)* bacterial strains by the agar-well diffusion method (Cruick-shank,1970). The solvent DMSO was used as negative control. A 0.5 ml spore suspension (10^{-6}–10^{-7} spore ml^{-1}) of each of

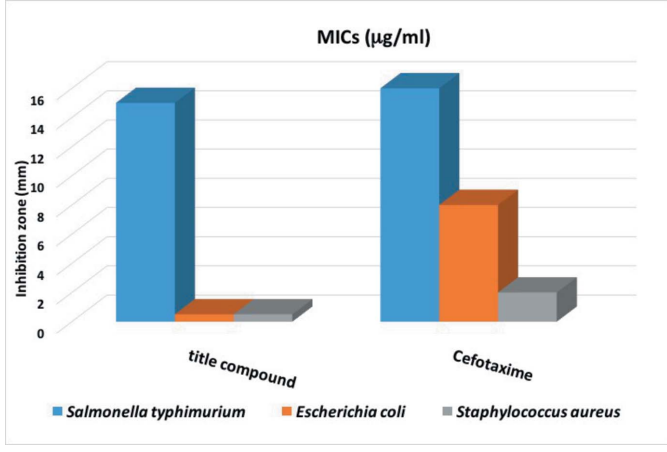


Figure 7
MICs (minimum inhibitory concentrations) antibacterial activity of the title compound and the standard Cefotaxime

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{13}NO_2$
M_r	239.26
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	4.8637 (3), 14.6601 (10), 16.6512 (9)
V (Å^3)	1187.27 (13)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.72
Crystal size (mm)	0.1 × 0.1 × 0.08
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire2 CCD
Absorption correction	Integration (*ABSORB*; DeTitta, 1985)
T_{min}, T_{max}	0.966, 0.991
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10738, 2461, 2222
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.632
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.131, 1.07
No. of reflections	2461
No. of parameters	163
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.36
Absolute structure	Flack x determined using 838 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.00 (19)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SIR2004* (Burla *et al.*, 2005), *SHELXL* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

the investigated organisms was added to a sterile agar medium just before solidification, then poured into sterile petri dishes (9 cm in diameter) and left to solidify. Using a sterile cork borer (6 mm in diameter), five holes (wells) were made in each dish, and then 5 µL of the tested compound, dissolved in DMSO with different concentrations (C, C/2, C/4, C/8), was poured into these holes. Finally, the dishes were incubated at 310 K for 48 h. Clear or inhibition zones were detected around each hole. DMSO alone (0.5 µL) was used as a control under the same conditions for each organism by subtracting the diameter of inhibition zone resulting with DMSO from that obtained in the study compound. The antibacterial activity of Cefotaxime was also measured in comparison to the title compound and used as a standard to reveal the potency of synthesized derivative.

The results of the antimicrobial screening indicate that the compound shows significant activity only against *Salmonella typhimurium* with an inhibition zone diameter of 15 mm. This value is close to that observed with the standard used (Cefotaxime) against the same bacterium (16 mm). For the two other bacteria, *Escherichia coli* and *Staphyloccus aureus*, the standard exhibits a higher activity than the study compound, for which the inhibition zone diameter is under 2 mm. These results are summarized in Fig. 7, which gives the MICs (minimum inhibitory concentrations) of the title Schiff base and Cefotaxime.

7. Synthesis and crystallization

The title Schiff base was synthesized by reacting 3-amino-acetophenone (0.13 g, 1 mmol) and salicylaldehyde (0.12 g, 1 mmol) in ethanol (20 ml). The resulting mixture was refluxed for 3 h. Yellow single crystals suitable for single crystal X-ray diffraction studies were obtained by slow evaporation of the solution.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were located in difference electron-density maps and treated as riding on their parent atoms, with C—H = 0.95–0.98 Å with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(Cmethyl) and O—H = 0.84 Å with U_{iso}(H) = 1.5U_{eq}(O).

Acknowledgements

The authors acknowledge CRM2, Institut Jean Barriol (UMR 7036 CNRS, University de Lorraine, France), for providing access to the experimental crystallographic facilities. Dr Slimane Dahaoui and Dr Emmanuel Wenger are thanked for their help in collectiong diffraction data, Professor Foudil Khelifa, Director of the Laboratoire d'hygiène de la Wilaya de Constantine, Algeria, for use of the antimicrobial facility and biologists Zine Faiza, Haifi Maya and Belhaffaf Mouni for their help.

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supporting information

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A new crystal form and antimicrobial activity of (*E*)-1-[3-(2-hydroxybenzylideneamino)phenyl]ethanone

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

(*E*)-1-[3-(2-Hydroxybenzylideneamino)phenyl]ethanone

Crystal data

$C_{15}H_{13}NO_2$
$M_r = 239.26$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.8637$ (3) Å
$b = 14.6601$ (10) Å
$c = 16.6512$ (9) Å
$V = 1187.27$ (13) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.339$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 10738 reflections
$\theta = 4.0$–$77.0°$
$\mu = 0.72$ mm^{-1}
$T = 100$ K
Prism, yellow
0.1 × 0.1 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur Sapphire2 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: integration (ABSORB; DeTitta, 1985)
$T_{min} = 0.966$, $T_{max} = 0.991$
10738 measured reflections
2461 independent reflections
2222 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 77.0°$, $\theta_{min} = 4.0°$
$h = -5 \rightarrow 6$
$k = -18 \rightarrow 18$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.131$
$S = 1.07$
2461 reflections
163 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.6475P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Absolute structure: Flack *x* determined using 838 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.00 (19)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.2016 (4)	0.05562 (11)	0.50631 (9)	0.0218 (4)
O2	0.9481 (4)	0.29985 (14)	0.27572 (12)	0.0356 (6)
N1	0.0723 (4)	0.03478 (13)	0.37418 (10)	0.0173 (5)
C1	0.2784 (5)	0.06549 (15)	0.32063 (12)	0.0175 (6)
C2	0.4217 (5)	0.14349 (16)	0.34432 (13)	0.0202 (6)
C3	0.6303 (5)	0.17989 (15)	0.29616 (13)	0.0201 (6)
C4	0.6939 (5)	0.13896 (17)	0.22293 (13)	0.0225 (6)
C5	0.5530 (5)	0.06016 (17)	0.19957 (13)	0.0233 (6)
C6	0.3462 (5)	0.02425 (17)	0.24763 (13)	0.0225 (7)
C7	−0.0656 (5)	−0.03891 (15)	0.36008 (13)	0.0183 (6)
C8	−0.2785 (5)	−0.06931 (15)	0.41500 (13)	0.0173 (6)
C9	−0.3394 (5)	−0.02099 (14)	0.48639 (12)	0.0174 (6)
C10	−0.5447 (5)	−0.05303 (15)	0.53750 (13)	0.0196 (6)
C11	−0.6896 (5)	−0.13149 (15)	0.51850 (13)	0.0203 (6)
C12	−0.6348 (5)	−0.17920 (15)	0.44765 (14)	0.0208 (6)
C13	−0.4307 (5)	−0.14870 (15)	0.39705 (13)	0.0199 (6)
C14	0.7915 (5)	0.26259 (16)	0.32198 (15)	0.0243 (7)
C15	0.7613 (6)	0.29571 (19)	0.40716 (18)	0.0354 (8)
H1	−0.08597	0.06773	0.47038	0.0326*
H2	0.37706	0.17221	0.39377	0.0242*
H4	0.83170	0.16435	0.18922	0.0270*
H5	0.59906	0.03096	0.15045	0.0280*
H6	0.25006	−0.02878	0.23072	0.0270*
H7	−0.02712	−0.07358	0.31321	0.0219*
H10	−0.58548	−0.02092	0.58561	0.0235*
H11	−0.82777	−0.15309	0.55406	0.0244*
H12	−0.73747	−0.23226	0.43451	0.0250*
H13	−0.39171	−0.18163	0.34925	0.0239*
H15A	0.62683	0.25763	0.43540	0.0531*
H15B	0.69811	0.35919	0.40695	0.0531*
H15C	0.93926	0.29186	0.43454	0.0531*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0218 (8)	0.0262 (8)	0.0173 (7)	−0.0043 (6)	0.0034 (6)	−0.0041 (6)
O2	0.0345 (11)	0.0348 (10)	0.0376 (10)	−0.0070 (8)	0.0067 (9)	0.0108 (8)
N1	0.0156 (9)	0.0224 (9)	0.0140 (8)	0.0023 (7)	0.0002 (7)	0.0011 (7)
C1	0.0160 (10)	0.0225 (10)	0.0141 (10)	0.0040 (9)	0.0006 (8)	0.0045 (8)
C2	0.0193 (11)	0.0239 (11)	0.0173 (10)	0.0052 (9)	0.0013 (9)	0.0024 (8)

C3	0.0181 (12)	0.0220 (10)	0.0202 (10)	0.0032 (8)	−0.0003 (8)	0.0057 (8)
C4	0.0174 (11)	0.0333 (12)	0.0169 (10)	0.0035 (10)	0.0011 (9)	0.0103 (9)
C5	0.0212 (12)	0.0349 (12)	0.0139 (9)	0.0035 (10)	0.0016 (9)	0.0015 (9)
C6	0.0182 (12)	0.0337 (12)	0.0155 (10)	−0.0008 (10)	−0.0008 (8)	0.0002 (9)
C7	0.0196 (11)	0.0217 (10)	0.0135 (9)	0.0035 (9)	−0.0005 (9)	0.0011 (8)
C8	0.0173 (11)	0.0206 (10)	0.0141 (10)	0.0021 (8)	−0.0011 (8)	0.0014 (8)
C9	0.0183 (11)	0.0188 (9)	0.0151 (9)	0.0027 (8)	−0.0033 (8)	0.0015 (8)
C10	0.0201 (11)	0.0239 (10)	0.0147 (9)	0.0016 (9)	0.0001 (8)	−0.0002 (8)
C11	0.0175 (10)	0.0242 (10)	0.0193 (10)	0.0014 (9)	0.0014 (9)	0.0043 (8)
C12	0.0207 (12)	0.0183 (10)	0.0235 (11)	−0.0008 (8)	−0.0030 (9)	0.0008 (8)
C13	0.0220 (12)	0.0193 (10)	0.0185 (10)	0.0021 (9)	−0.0034 (9)	−0.0006 (8)
C14	0.0210 (12)	0.0200 (10)	0.0320 (12)	0.0013 (9)	0.0027 (10)	0.0066 (9)
C15	0.0385 (16)	0.0290 (12)	0.0388 (15)	−0.0107 (11)	0.0066 (12)	−0.0038 (11)

Geometric parameters (Å, º)

O1—C9	1.349 (3)	C10—C11	1.386 (3)
O2—C14	1.213 (3)	C11—C12	1.397 (3)
O1—H1	0.8400	C12—C13	1.377 (3)
N1—C1	1.415 (3)	C14—C15	1.506 (4)
N1—C7	1.293 (3)	C2—H2	0.9500
C1—C6	1.397 (3)	C4—H4	0.9500
C1—C2	1.396 (3)	C5—H5	0.9500
C2—C3	1.399 (3)	C6—H6	0.9500
C3—C4	1.394 (3)	C7—H7	0.9500
C3—C14	1.507 (3)	C10—H10	0.9500
C4—C5	1.398 (3)	C11—H11	0.9500
C5—C6	1.389 (3)	C12—H12	0.9500
C7—C8	1.452 (3)	C13—H13	0.9500
C8—C13	1.411 (3)	C15—H15A	0.9800
C8—C9	1.415 (3)	C15—H15B	0.9800
C9—C10	1.394 (3)	C15—H15C	0.9800
O1···N1	2.590 (2)	C5···H11^{ix}	2.9900
O2···C7^{i}	3.321 (3)	C5···H10^{ix}	3.0200
O1···H15B^{ii}	2.7200	C6···H10^{ix}	2.9800
O1···H5^{iii}	2.7600	C6···H7	2.5600
O2···H4	2.5200	C7···H6	2.6500
O2···H6^{i}	2.6900	C7···H1	2.4100
O2···H7^{i}	2.4000	C9···H5^{iii}	2.9700
N1···O1	2.590 (2)	C10···H5^{iii}	2.8900
N1···C3^{iv}	3.292 (3)	C11···H12^{viii}	3.0700
N1···H1	1.8400	C12···H11^{viii}	2.8800
C1···C3^{iv}	3.594 (3)	C12···H12^{viii}	3.0400
C1···C4^{iv}	3.448 (3)	C13···H11^{viii}	3.0600
C1···C7^{v}	3.599 (3)	C15···H2	2.6100
C1···C8^{v}	3.320 (3)	H1···N1	1.8400
C1···C9^{v}	3.561 (3)	H1···C1	3.0600

C2···C9^{v}	3.572 (3)	H1···C7	2.4100
C2···C14^{iv}	3.547 (3)	H2···C15	2.6100
C3···N1^{v}	3.292 (3)	H2···H15A	1.8800
C3···C1^{v}	3.594 (3)	H4···O2	2.5200
C4···C1^{v}	3.448 (3)	H4···H12^{x}	2.6000
C5···C7^{v}	3.563 (3)	H5···O1^{xi}	2.7600
C6···C7^{v}	3.542 (3)	H5···C9^{xi}	2.9700
C7···C6^{iv}	3.542 (3)	H5···C10^{xi}	2.8900
C7···C5^{iv}	3.563 (3)	H6···C7	2.6500
C7···C11^{v}	3.485 (3)	H6···H7	2.0300
C7···C13^{v}	3.536 (3)	H6···O2^{vi}	2.6900
C7···C12^{v}	3.278 (3)	H7···C6	2.5600
C7···O2^{vi}	3.321 (3)	H7···H6	2.0300
C7···C1^{iv}	3.599 (3)	H7···H13	2.4500
C8···C11^{v}	3.465 (3)	H7···O2^{vi}	2.4000
C8···C1^{iv}	3.320 (3)	H10···C5^{xii}	3.0200
C8···C12^{v}	3.563 (3)	H10···C6^{xii}	2.9800
C9···C2^{iv}	3.572 (3)	H10···H15B^{xiii}	2.6000
C9···C11^{v}	3.591 (3)	H11···C5^{xii}	2.9900
C9···C1^{iv}	3.561 (3)	H11···C12^{vii}	2.8800
C11···C8^{iv}	3.465 (3)	H11···C13^{vii}	3.0600
C11···C7^{iv}	3.485 (3)	H12···C11^{vii}	3.0700
C11···C12^{vii}	3.565 (3)	H12···C12^{vii}	3.0400
C11···C9^{iv}	3.591 (3)	H12···H4^{xiv}	2.6000
C12···C11^{viii}	3.565 (3)	H13···H7	2.4500
C12···C7^{iv}	3.278 (3)	H15A···C2	2.4700
C12···C8^{iv}	3.563 (3)	H15A···H2	1.8800
C13···C7^{iv}	3.536 (3)	H15A···H15C^{ii}	2.4600
C14···C2^{v}	3.547 (3)	H15B···O1^{xv}	2.7200
C1···H1	3.0600	H15B···H10^{xvi}	2.6000
C2···H15A	2.4700	H15C···H15A^{xv}	2.4600

C9—O1—H1	109.00	O2—C14—C3	120.5 (2)
C1—N1—C7	121.25 (18)	C1—C2—H2	120.00
N1—C1—C2	115.86 (19)	C3—C2—H2	120.00
N1—C1—C6	125.3 (2)	C3—C4—H4	120.00
C2—C1—C6	118.9 (2)	C5—C4—H4	120.00
C1—C2—C3	120.8 (2)	C4—C5—H5	120.00
C2—C3—C4	119.9 (2)	C6—C5—H5	120.00
C2—C3—C14	121.4 (2)	C1—C6—H6	120.00
C4—C3—C14	118.7 (2)	C5—C6—H6	120.00
C3—C4—C5	119.4 (2)	N1—C7—H7	120.00
C4—C5—C6	120.5 (2)	C8—C7—H7	120.00
C1—C6—C5	120.5 (2)	C9—C10—H10	120.00
N1—C7—C8	120.8 (2)	C11—C10—H10	120.00
C7—C8—C13	119.6 (2)	C10—C11—H11	120.00
C9—C8—C13	118.7 (2)	C12—C11—H11	120.00
C7—C8—C9	121.7 (2)	C11—C12—H12	120.00

O1—C9—C10	119.10 (19)	C13—C12—H12	120.00
C8—C9—C10	119.6 (2)	C8—C13—H13	119.00
O1—C9—C8	121.3 (2)	C12—C13—H13	119.00
C9—C10—C11	120.3 (2)	C14—C15—H15A	109.00
C10—C11—C12	120.8 (2)	C14—C15—H15B	109.00
C11—C12—C13	119.5 (2)	C14—C15—H15C	109.00
C8—C13—C12	121.1 (2)	H15A—C15—H15B	109.00
O2—C14—C15	120.9 (2)	H15A—C15—H15C	109.00
C3—C14—C15	118.5 (2)	H15B—C15—H15C	109.00

C7—N1—C1—C2	−177.1 (2)	C3—C4—C5—C6	1.8 (4)
C7—N1—C1—C6	3.0 (3)	C4—C5—C6—C1	−1.0 (4)
C1—N1—C7—C8	−179.4 (2)	N1—C7—C8—C9	−1.3 (3)
N1—C1—C2—C3	179.9 (2)	N1—C7—C8—C13	178.4 (2)
C6—C1—C2—C3	−0.2 (3)	C7—C8—C9—O1	0.1 (3)
N1—C1—C6—C5	−179.9 (2)	C7—C8—C9—C10	−179.7 (2)
C2—C1—C6—C5	0.1 (4)	C13—C8—C9—O1	−179.6 (2)
C1—C2—C3—C4	1.0 (4)	C13—C8—C9—C10	0.7 (3)
C1—C2—C3—C14	−178.4 (2)	C7—C8—C13—C12	−179.7 (2)
C2—C3—C4—C5	−1.8 (3)	C9—C8—C13—C12	−0.1 (3)
C14—C3—C4—C5	177.6 (2)	O1—C9—C10—C11	180.0 (2)
C2—C3—C14—O2	−171.1 (2)	C8—C9—C10—C11	−0.3 (3)
C2—C3—C14—C15	11.1 (3)	C9—C10—C11—C12	−0.7 (3)
C4—C3—C14—O2	9.5 (4)	C10—C11—C12—C13	1.3 (4)
C4—C3—C14—C15	−168.4 (2)	C11—C12—C13—C8	−0.9 (3)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+1/2, −*z*+1; (iii) −*x*+1/2, −*y*, *z*+1/2; (iv) *x*−1, *y*, *z*; (v) *x*+1, *y*, *z*; (vi) −*x*+1, *y*−1/2, −*z*+1/2; (vii) *x*−1/2, −*y*−1/2, −*z*+1; (viii) *x*+1/2, −*y*−1/2, −*z*+1; (ix) −*x*−1/2, −*y*, *z*−1/2; (x) −*x*, *y*+1/2, −*z*+1/2; (xi) −*x*+1/2, −*y*, *z*−1/2; (xii) −*x*−1/2, −*y*, *z*+1/2; (xiii) *x*−3/2, −*y*+1/2, −*z*+1; (xiv) −*x*, *y*−1/2, −*z*+1/2; (xv) *x*+1/2, −*y*+1/2, −*z*+1; (xvi) *x*+3/2, −*y*+1/2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.84	1.84	2.590 (2)	148
C7—H7···O2^{vi}	0.95	2.40	3.321 (3)	162

Symmetry code: (vi) −*x*+1, *y*−1/2, −*z*+1/2.