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(Z)-6-[(5-Chloro-2-hydroxyanilino)-methylene]-4-methoxycyclohexa-2,4-dienone 0.25-hydrateArzu Özek,^a Orhan Büyükgüngör,^{a*} Çiğdem Albayrak^b and Mustafa Odabaşoğlu^c^aDepartment of Physics, Ondokuz Mayıs University, TR-55139 Samsun, Turkey,^bFaculty of Education, Sinop University, Turkey, and ^cPamukkale University, Denizli Technical Vocational School, Turkey

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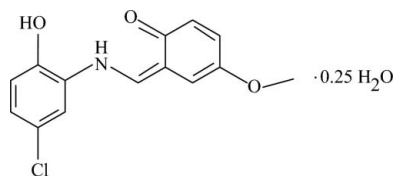
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; some non-H atoms missing; disorder in solvent or counterion; R factor = 0.031; wR factor = 0.086; data-to-parameter ratio = 13.9.

The title compound, $\text{C}_{14}\text{H}_{12}\text{ClNO}_3 \cdot 0.25\text{H}_2\text{O}$, exists in the keto-amine form, and the aromatic rings are oriented at a dihedral angle of 7.24 (7)°. Bifurcated intramolecular $\text{N}-\text{H} \cdots (\text{O}, \text{O})$ hydrogen bonds result in the formation of planar six- and five-membered rings. In the crystal structure, intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds link the molecules into chains. $\pi-\pi$ contacts between benzene rings [centroid-centroid distance = 3.5065 (9) Å] may further stabilize the structure. There also exists a weak $\text{C}-\text{H} \cdots \pi$ interaction.

Related literature

For general background, see: Büyükgüngör *et al.* (2007); Hökelek *et al.* (2004); Odabaşoğlu *et al.* (2004). For related structures, see: Özek *et al.* (2007, 2008); Ersanlı *et al.* (2003).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{12}\text{ClNO}_3 \cdot 0.25\text{H}_2\text{O}$ $M_r = 279.95$ Monoclinic, $C2/c$ $a = 21.3670$ (11) Å $b = 6.7600$ (3) Å $c = 17.7404$ (9) Å $\beta = 103.841$ (4)° $V = 2488.0$ (2) Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 0.31$ mm⁻¹ $T = 100$ K $0.68 \times 0.54 \times 0.41$ mm

Data collection

Stoe IPDS-II diffractometer

Absorption correction: integration
(*X-RED32*; Stoe & Cie, 2002) $T_{\min} = 0.970$, $T_{\max} = 0.970$

6976 measured reflections

2588 independent reflections

2352 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.022$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.086$ $S = 1.09$

2588 reflections

186 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1} \cdots \text{O1}$	0.86	1.84	2.5511 (16)	140
$\text{N1}-\text{H1} \cdots \text{O3}$	0.86	2.19	2.6063 (17)	109
$\text{C3}-\text{H3} \cdots \text{O4}$	0.93	2.43	3.279 (5)	151
$\text{O4}-\text{H4A} \cdots \text{O2}^{\text{i}}$	0.831 (19)	2.029 (19)	2.842 (3)	166 (6)
$\text{O3}-\text{H3A} \cdots \text{O1}^{\text{ii}}$	0.852 (17)	1.743 (18)	2.5652 (16)	162 (3)
$\text{C12}-\text{H12} \cdots \text{O2}^{\text{iii}}$	0.93	2.56	3.4372 (18)	157
$\text{C7}-\text{H7A} \cdots \text{Cg1}^{\text{iv}}$	0.96	2.83	3.644 (2)	143

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$; (iii) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$; (iv) $x + \frac{1}{2}, y + \frac{3}{2}, z$. Cg1 is the centroid of the C1-C6 ring.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-RED32* (Stoe & Cie, 2002); data reduction: *X-RED32*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2658).

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supplementary materials

Acta Cryst. (2009). E65, o1104 [doi:10.1107/S160053680901410X]

(Z)-6-[(5-Chloro-2-hydroxyanilino)methylene]-4-methoxycyclohexa-2,4-dienone 0.25-hydrate

A. Özek, O. Büyükgüngör, Ç. Albayrak and M. Odabasoglu

Comment

As part of our ongoing studies on the syntheses and structural characterizations of Schiff-base compounds (Özek *et al.*, 2008; Özek *et al.*, 2007), we report herein the crystal structure of the title compound.

In general, *o*-hydroxy Schiff bases exhibit two possible tautomeric forms, namely, phenol-imine and keto-amine. In the solid state, the keto-amine form is observed in naphthaldimine (Odabaşoğlu *et al.*, 2004), while the phenol-imine form is observed in salicylaldimine (Büyükgüngör *et al.*, 2007) Schiff bases. However, naphthaldimine and salicylaldimine can also exist in the phenol-imine and keto-amine forms, respectively depending on the stereochemistry of the molecule and the type of nitrogen substituents in naphthaldimine and salicylaldimine Schiff bases (Hökelek *et al.*, 2004).

In the title compound (Fig. 1), the keto-amine form is favored over the phenol-imine form, as indicated by C2—O1 [1.2924 (18) Å], C8—N1 [1.3122 (19) Å], C1—C8 [1.412 (2) Å] and C1—C2 [1.433 (2) Å] bonds. The C2—O1 and C8—N1 bonds indicate double-bond and a high degree of single-bond characters, respectively. Similar results were observed for 2-[(2-hydroxy-4-nitrophenyl)-aminomethylene]cyclohexa-3,5-dien-1(2*H*)-one [C—O = 1.298 (2) and C—N = 1.308 (2) Å; Ersanlı *et al.*, 2003].

It is known that Schiff bases may exhibit thermochromism or photochromism, depending on the planarity or non-planarity of the molecule, respectively. Therefore, one can expect thermochromic properties in the title compound caused by planarity of the molecule; the dihedral angle between rings A (C1—C6) and B (C9—C14) is 7.24 (7)°. Intramolecular N—H···O hydrogen bonds (Table 1) result in the formations of planar six- and five-membered rings C (O1/N1/C1/C2/C8/H1) and D (O3/N1/C9/C10/H1). They are oriented with respect to the adjacent rings at dihedral angles of A/C = 4.44 (9), A/D = 9.6 (9), B/C = 5.42 (9), B/D = 2.96 (9) and C/D = 6.55 (9)°. So, they are nearly coplanar.

In the crystal structure, intermolecular O—H···O and C—H···O hydrogen bonds (Table 1) link the molecules into chains (Fig. 2), in which they may be effective in the stabilization of the structure. The π – π contact between the phenyl rings, Cg1—Cg2ⁱ [symmetry code: (i) 1/2 - x, 3/2 - y, -z, where Cg1 and Cg2 are centroids of the rings A (C1—C6) and B (C9—C14), respectively] may further stabilize the structure, with centroid-centroid distance of 3.5065 (9) Å. There also exists a weak C—H··· π interaction (Table 1).

Experimental

For the preparation of the title compound, the mixture of 5-methoxysalicyl- aldehyde (0.5 g, 3.3 mmol) in ethanol (20 ml) and 2-hydroxy-5-chloroaniline (0.47 g, 3.3 mmol) in ethanol (20 ml) was stirred for 1 h under reflux. Crystals suitable for X-ray analysis were obtained from methanol by slow evaporation (yield; % 84, m.p. 415–416 K).

Refinement

H atoms of water molecule and hydroxy group were located in difference Fourier maps and refined isotropically, with restraints of O3—H3A = 0.852 (17) and O4—H4A = 0.831 (19) Å. The remaining H atoms were positioned geometrically with N—H = 0.86 Å (for NH) and C—H = 0.93 and 0.96 Å, for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{N})$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms.

Figures

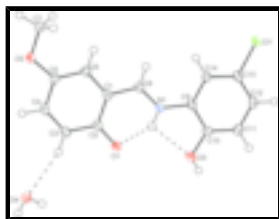


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

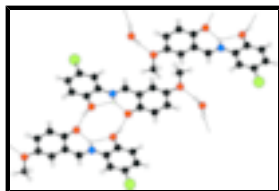


Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

(Z)-6-[(5-Chloro-2-hydroxyanilino)methylene]-4-methoxycyclohexa-2,4-dienone 0.25-hydrate

Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClNO}_3 \cdot 0.25\text{H}_2\text{O}$

$M_r = 279.95$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 21.3670$ (11) Å

$b = 6.7600$ (3) Å

$c = 17.7404$ (9) Å

$\beta = 103.841$ (4)°

$V = 2488.0$ (2) Å³

$Z = 8$

$F_{000} = 1162$

$D_x = 1.495$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 6976 reflections

$\theta = 2.0$ – 28.0 °

$\mu = 0.31$ mm⁻¹

$T = 100$ K

Prism, red

$0.68 \times 0.54 \times 0.41$ mm

Data collection

Stoe IPDS-II
diffractometer

2588 independent reflections

Radiation source: fine-focus sealed tube

2352 reflections with $I > 2\sigma(I)$

Monochromator: plane graphite

$R_{\text{int}} = 0.022$

Detector resolution: 6.67 pixels mm⁻¹

$\theta_{\text{max}} = 26.5$ °

$T = 100$ K

$\theta_{\text{min}} = 2.0$ °

ω scans $h = -25 \rightarrow 26$
 Absorption correction: integration $k = -8 \rightarrow 7$
 (X-RED32; Stoe & Cie, 2002) $l = -22 \rightarrow 22$
 $T_{\min} = 0.970$, $T_{\max} = 0.970$
 6976 measured reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.031$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.086$ $w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 3.3917P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.09$ $(\Delta/\sigma)_{\max} = 0.001$
 2588 reflections $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 186 parameters $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$
 2 restraints Extinction correction: none
 Primary atom site location: structure-invariant direct methods

Special details

Experimental. 141 frames, detector distance = 100 mm

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.64391 (7)	0.8209 (2)	0.49336 (8)	0.0163 (3)	
C2	0.63352 (7)	0.6614 (2)	0.43854 (8)	0.0171 (3)	
C3	0.58193 (7)	0.6836 (2)	0.37118 (9)	0.0191 (3)	
H3	0.5736	0.5834	0.3342	0.023*	
C4	0.54480 (7)	0.8493 (2)	0.36036 (8)	0.0189 (3)	
H4	0.5112	0.8593	0.3162	0.023*	
C5	0.55578 (7)	1.0069 (2)	0.41439 (8)	0.0175 (3)	
C6	0.60491 (7)	0.9945 (2)	0.47962 (8)	0.0170 (3)	
H6	0.6129	1.0986	0.5149	0.020*	
C7	0.51514 (8)	1.3060 (2)	0.45380 (9)	0.0211 (3)	

supplementary materials

H7A	0.5063	1.2434	0.4987	0.032*	
H7B	0.4832	1.4055	0.4349	0.032*	
H7C	0.5570	1.3663	0.4675	0.032*	
C8	0.69160 (7)	0.8057 (2)	0.56359 (8)	0.0164 (3)	
H8	0.6987	0.9125	0.5976	0.020*	
C9	0.77299 (7)	0.6029 (2)	0.65047 (8)	0.0158 (3)	
C10	0.79878 (7)	0.4110 (2)	0.65524 (9)	0.0181 (3)	
C11	0.84717 (7)	0.3589 (2)	0.71999 (9)	0.0201 (3)	
H11	0.8645	0.2321	0.7238	0.024*	
C12	0.86950 (7)	0.4950 (2)	0.77875 (8)	0.0190 (3)	
H12	0.9020	0.4604	0.8218	0.023*	
C13	0.84290 (7)	0.6831 (2)	0.77270 (8)	0.0175 (3)	
C14	0.79444 (7)	0.7395 (2)	0.70949 (8)	0.0172 (3)	
H14	0.7767	0.8657	0.7066	0.021*	
Cl1	0.873014 (18)	0.85790 (6)	0.84450 (2)	0.02270 (12)	
N1	0.72613 (6)	0.64456 (19)	0.58204 (7)	0.0165 (3)	
H1	0.7192	0.5522	0.5477	0.020*	
O1	0.66836 (5)	0.50302 (16)	0.45048 (6)	0.0219 (3)	
O2	0.51359 (5)	1.16195 (17)	0.39490 (6)	0.0229 (3)	
O3	0.77355 (5)	0.28754 (18)	0.59629 (7)	0.0236 (3)	
H3A	0.7990 (11)	0.196 (3)	0.5898 (15)	0.055 (8)*	
O4	0.5000	0.3573 (10)	0.2500	0.0253 (14)	0.25
H4A	0.500 (4)	0.286 (5)	0.2880 (13)	0.03 (2)*	0.25

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0144 (7)	0.0192 (7)	0.0150 (6)	-0.0005 (6)	0.0028 (5)	-0.0001 (6)
C2	0.0154 (7)	0.0190 (7)	0.0166 (7)	0.0000 (6)	0.0033 (5)	-0.0010 (6)
C3	0.0184 (7)	0.0226 (8)	0.0155 (7)	-0.0012 (6)	0.0023 (6)	-0.0039 (6)
C4	0.0166 (7)	0.0262 (8)	0.0127 (7)	-0.0004 (6)	0.0012 (5)	0.0000 (6)
C5	0.0158 (7)	0.0202 (7)	0.0167 (7)	0.0021 (6)	0.0043 (5)	0.0017 (6)
C6	0.0173 (7)	0.0187 (7)	0.0147 (6)	0.0001 (6)	0.0037 (5)	-0.0016 (5)
C7	0.0221 (8)	0.0180 (7)	0.0213 (7)	0.0030 (6)	0.0017 (6)	-0.0009 (6)
C8	0.0142 (7)	0.0192 (7)	0.0161 (7)	-0.0004 (6)	0.0045 (5)	-0.0018 (6)
C9	0.0119 (6)	0.0217 (7)	0.0139 (6)	0.0004 (6)	0.0031 (5)	0.0008 (6)
C10	0.0165 (7)	0.0194 (7)	0.0180 (7)	0.0003 (6)	0.0031 (6)	-0.0021 (6)
C11	0.0178 (7)	0.0196 (7)	0.0220 (7)	0.0035 (6)	0.0030 (6)	0.0025 (6)
C12	0.0153 (7)	0.0245 (8)	0.0158 (7)	0.0016 (6)	0.0011 (5)	0.0043 (6)
C13	0.0143 (7)	0.0235 (8)	0.0146 (7)	-0.0019 (6)	0.0035 (5)	-0.0017 (6)
C14	0.0158 (7)	0.0183 (7)	0.0166 (7)	0.0018 (6)	0.0021 (5)	0.0009 (6)
Cl1	0.0219 (2)	0.0258 (2)	0.01674 (19)	0.00025 (14)	-0.00266 (14)	-0.00406 (14)
N1	0.0151 (6)	0.0184 (6)	0.0142 (6)	0.0007 (5)	0.0001 (5)	-0.0018 (5)
O1	0.0205 (5)	0.0206 (6)	0.0221 (5)	0.0040 (4)	0.0002 (4)	-0.0053 (4)
O2	0.0226 (6)	0.0233 (6)	0.0189 (5)	0.0078 (4)	-0.0030 (4)	-0.0015 (4)
O3	0.0206 (6)	0.0228 (6)	0.0238 (6)	0.0049 (5)	-0.0016 (4)	-0.0077 (5)
O4	0.036 (4)	0.021 (3)	0.016 (3)	0.000	-0.001 (3)	0.000

Geometric parameters (Å, °)

C1—C8	1.412 (2)	C8—H8	0.9300
C1—C6	1.426 (2)	C9—C14	1.389 (2)
C1—C2	1.433 (2)	C9—C10	1.404 (2)
C2—O1	1.2924 (18)	C9—N1	1.4050 (18)
C2—C3	1.427 (2)	C10—O3	1.3459 (18)
C3—C4	1.359 (2)	C10—C11	1.395 (2)
C3—H3	0.9300	C11—C12	1.387 (2)
C4—C5	1.415 (2)	C11—H11	0.9300
C4—H4	0.9300	C12—C13	1.386 (2)
C5—C6	1.366 (2)	C12—H12	0.9300
C5—O2	1.3720 (18)	C13—C14	1.385 (2)
C6—H6	0.9300	C13—Cl1	1.7438 (15)
C7—O2	1.4229 (19)	C14—H14	0.9300
C7—H7A	0.9600	N1—H1	0.8600
C7—H7B	0.9600	O3—H3A	0.852 (17)
C7—H7C	0.9600	O4—H4A	0.831 (19)
C8—N1	1.3122 (19)		
C8—C1—C6	118.69 (13)	N1—C8—H8	119.2
C8—C1—C2	120.36 (14)	C1—C8—H8	119.2
C6—C1—C2	120.91 (13)	C14—C9—C10	120.98 (13)
O1—C2—C3	121.57 (14)	C14—C9—N1	123.82 (13)
O1—C2—C1	121.51 (13)	C10—C9—N1	115.19 (13)
C3—C2—C1	116.90 (14)	O3—C10—C11	124.28 (14)
C4—C3—C2	120.85 (14)	O3—C10—C9	116.60 (13)
C4—C3—H3	119.6	C11—C10—C9	119.10 (14)
C2—C3—H3	119.6	C12—C11—C10	120.35 (14)
C3—C4—C5	121.89 (13)	C12—C11—H11	119.8
C3—C4—H4	119.1	C10—C11—H11	119.8
C5—C4—H4	119.1	C13—C12—C11	119.30 (13)
C6—C5—O2	125.86 (14)	C13—C12—H12	120.3
C6—C5—C4	119.79 (14)	C11—C12—H12	120.3
O2—C5—C4	114.34 (12)	C14—C13—C12	121.88 (14)
C5—C6—C1	119.64 (14)	C14—C13—Cl1	118.58 (12)
C5—C6—H6	120.2	C12—C13—Cl1	119.49 (11)
C1—C6—H6	120.2	C13—C14—C9	118.37 (14)
O2—C7—H7A	109.5	C13—C14—H14	120.8
O2—C7—H7B	109.5	C9—C14—H14	120.8
H7A—C7—H7B	109.5	C8—N1—C9	128.60 (13)
O2—C7—H7C	109.5	C8—N1—H1	115.7
H7A—C7—H7C	109.5	C9—N1—H1	115.7
H7B—C7—H7C	109.5	C5—O2—C7	116.07 (11)
N1—C8—C1	121.66 (14)	C10—O3—H3A	114.0 (18)
C8—C1—C2—O1	-2.1 (2)	C14—C9—C10—C11	-0.7 (2)
C6—C1—C2—O1	-179.69 (14)	N1—C9—C10—C11	177.86 (14)
C8—C1—C2—C3	176.39 (14)	O3—C10—C11—C12	-178.82 (15)
C6—C1—C2—C3	-1.2 (2)	C9—C10—C11—C12	-0.1 (2)

supplementary materials

O1—C2—C3—C4	178.48 (14)	C10—C11—C12—C13	0.5 (2)
C1—C2—C3—C4	0.0 (2)	C11—C12—C13—C14	0.0 (2)
C2—C3—C4—C5	0.6 (2)	C11—C12—C13—C11	-177.44 (12)
C3—C4—C5—C6	0.0 (2)	C12—C13—C14—C9	-0.9 (2)
C3—C4—C5—O2	-179.47 (14)	C11—C13—C14—C9	176.63 (11)
O2—C5—C6—C1	178.19 (14)	C10—C9—C14—C13	1.2 (2)
C4—C5—C6—C1	-1.2 (2)	N1—C9—C14—C13	-177.27 (14)
C8—C1—C6—C5	-175.82 (14)	C1—C8—N1—C9	-176.94 (14)
C2—C1—C6—C5	1.8 (2)	C14—C9—N1—C8	-5.5 (2)
C6—C1—C8—N1	175.38 (14)	C10—C9—N1—C8	175.95 (14)
C2—C1—C8—N1	-2.3 (2)	C6—C5—O2—C7	-10.6 (2)
C14—C9—C10—O3	178.07 (14)	C4—C5—O2—C7	168.79 (13)
N1—C9—C10—O3	-3.3 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O1	0.86	1.84	2.5511 (16)	140
N1—H1 \cdots O3	0.86	2.19	2.6063 (17)	109
C3—H3 \cdots O4	0.93	2.43	3.279 (5)	151
O4—H4A \cdots O2 ⁱ	0.831 (19)	2.029 (19)	2.842 (3)	166 (6)
O3—H3A \cdots O1 ⁱⁱ	0.852 (17)	1.743 (18)	2.5652 (16)	162 (3)
C12—H12 \cdots O2 ⁱⁱⁱ	0.93	2.56	3.4372 (18)	157
C7—H7A \cdots Cg1 ^{iv}	0.96	2.83	3.644 (2)	143

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+3/2, -y+1/2, -z+1$; (iii) $x+1/2, -y+3/2, z+1/2$; (iv) $x+1/2, y+3/2, z$.

Fig. 1

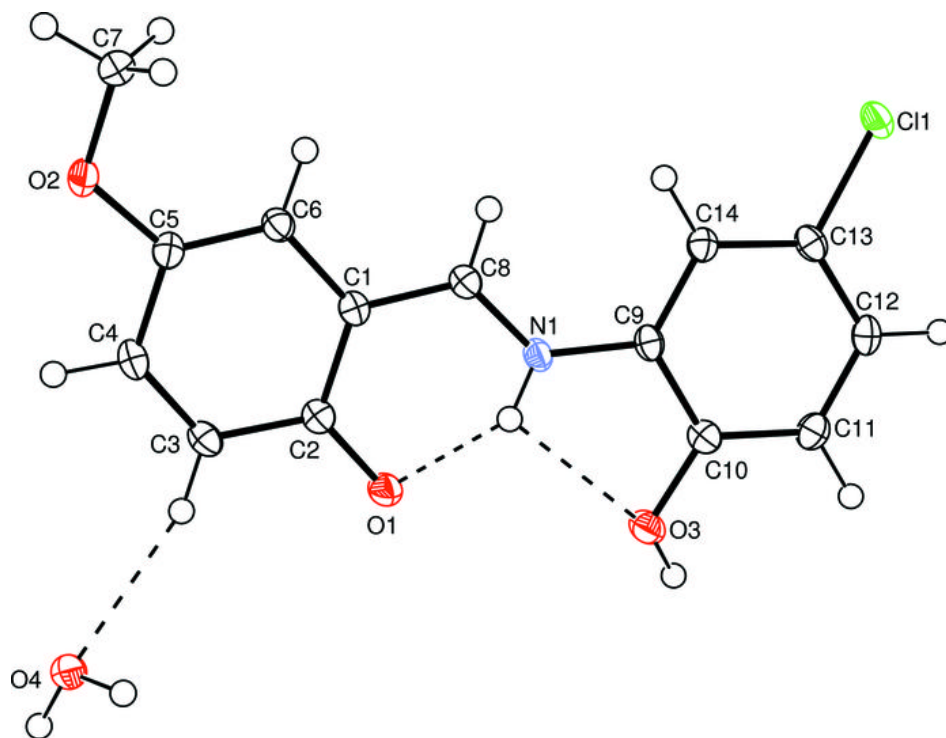


Fig. 2

