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3-(5-Chloro-2-hydroxyanilino)isobenzofuran-1(3*H*)-one[1]

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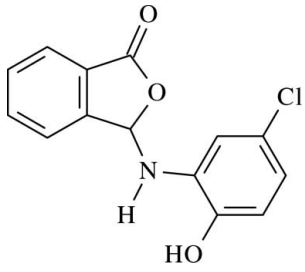
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.052; wR factor = 0.147; data-to-parameter ratio = 11.2.

The crystal structure of the title compound, $C_{14}H_{10}ClNO_3$, is stabilized by inversion-related O—H···O and C—H···O intermolecular hydrogen bonds and also by C—H···π and π–π interactions (centroid-to-centroid distance 3.681 Å and plane-to plane separation 3.618 Å). The O—H···O and C—H···O hydrogen bonds generate edge-fused $R_2^1(6)R_4^4(27)R_2^1(6)$ ring motifs. The phthalide part of the molecule is planar and is inclined at 76.8 (2)° to the benzene ring of the aminophenyl group.

Related literature

For related structures, see: Büyükgüngör & Odabaşoğlu (2006*a*,*b*, 2007); Odabaşoğlu & Büyükgüngör (2006*a*,*b*, 2007*a*,*b*). For general background, see: Aoki *et al.* (1973); Lacova (1973); Elderfield (1951); Tsi & Tan (1997); Bellasio (1974); Roy & Sarkar (2005); Etter (1990).



Experimental

Crystal data

$C_{14}H_{10}ClNO_3$
M_r = 275.68
Monoclinic, $P2_1/c$
a = 9.0439 (6) Å
b = 11.5363 (10) Å
c = 12.6602 (9) Å
β = 112.593 (5)°
V = 1219.51 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 296 K
0.53 × 0.39 × 0.13 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.731, T_{max} = 0.955
11707 measured reflections
2392 independent reflections
1898 reflections with $I > 2\sigma(I)$
R_{int} = 0.134

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.147
S = 1.03
2392 reflections
213 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C9–C14 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O3—H10···O1^{i}	0.83 (4)	2.03 (4)	2.820 (3)	160
C11—H2···O1^{i}	0.92 (3)	2.70 (3)	3.265 (3)	121
C4—H8···O2^{ii}	0.92 (4)	2.70 (4)	3.237 (3)	117
C5—H5···*Cg*1^{iii}	0.93 (2)	2.885	3.695 (3)	146

Symmetry codes: (i) $x+1, y, z$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+2, -y+1, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); 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: SJ2371).

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[1] 3-Substituted phthalides. Part XXVII. For Part XXVI, see Odabaşoğlu & Büyükgüngör (2007*c*).

supplementary materials

Acta Cryst. (2007). E**63**, o4343 [**doi:10.1107/S1600536807049641**]

3-(5-Chloro-2-hydroxyanilino)isobenzofuran-1(3*H*)-one

M. Odabasoglu and O. Büyükgüngör

Comment

Phthalides (isobenzofuranones) possess several important properties, such as fungicidal (Aoki *et al.*, 1973; Lacova, 1973), bactericidal and herbicidal (Lacova, 1973), analgesic (Elderfield, 1951), hypertensive and vasorelaxant activities (Tsi & Tan, 1997). In addition, phthalide derivatives are useful in the treatment of circulatory and heart-related diseases (Bellasio, 1974). They are also found to be associated with pesticidal activities (Roy & Sarkar, 2005). Considering the potential interest of such phthalide-3-phosphonates in synthetic organic chemistry, and as agrochemical and pharmaceutical agents, we decided to investigate the solid-state structures of 3-substituted phthalides by *x*-ray diffraction methods. As part of a continuing study of the interplay between molecular conformation and supramolecular aggregation in 3-substituted phthalides, we now report the structure of the title compound, 3-[2-(hydroxy-5-chloro)phenylamino]isobenzofuran-1(3*H*)-one, (I) (Fig. 1).

The geometry of the molecule of (I) does not show any significant differences from the average geometry found for 3-anilinoisobenzofuran-1(3*H*)-ones (Büyükgüngör & Odabaşoğlu, 2006*a*,b, 2007; Odabaşoğlu & Büyükgüngör, 2006*a*,b, 2007*a*,b). The phthalide group (C1–C8/O2) is planar, the largest deviation from the mean plane being −0.009 (1) Å for atom C7. The dihedral angle between the mean planes of the phthalide group and the phenyl ring is 76.8 (2)°.

In (I), the crystal packing is stabilized by C—H···O and N—H···O intermolecular hydrogen bonds (Fig. 2, Table 1), which generate $R_2^1(6)R_4^4(27)R_2^1(6)$ ring motifs (Etter, 1990). These motifs also generate a three dimensional network by C5—H5···*Cg*1 (*Cg*1 is the C9–C14 ring centroid), Table 1, and π···π interactions. - The π···π interaction occurs between the C2–C7 and C9–C14 rings and their symmetry-related counterparts at (x, 1/2 − y, 1/2 + z), with a centroid-to-centroid distance of 3.681 Å and a plane-to plane separation of 3.618 Å.

Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2006*a*), using phthalaldehydic acid and 2-hydroxy-5-chloroaniline as starting materials (yield 80%; m.p. 478–479 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol (95%) solution at room temperature.

Refinement

All H atoms were located in a difference Fourier map and were refined freely with isotropic displacement parameters.

Figures

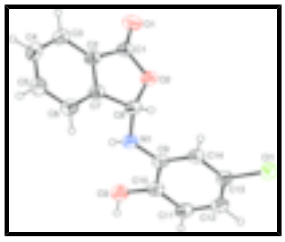

Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

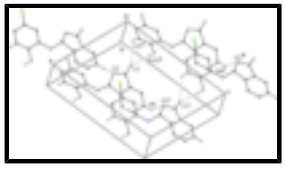

Fig. 2. Part of the crystal structure of (I), showing the formation of $R_2^1(6)R_4^4(27)R_2^1(6)$ motifs with hydrogen bonds shown as dashed lines. [Symmetry codes: (i) x, 1/2 − y, z + 1/2; (ii) x − 1, y, z; (iii) 1 − x, 1/2 − y, z + 1/2].

3-(5-Chloro-2-hydroxy-5-chloroanilino)isobenzofuran-1(3*H*)-one

Crystal data

$C_{14}H_{10}ClNO_3$	F_{000} = 568
M_r = 275.68	D_x = 1.502 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 11707 reflections
a = 9.0439 (6) Å	θ = 2.4–27.9º
b = 11.5363 (10) Å	μ = 0.32 mm^{-1}
c = 12.6602 (9) Å	T = 296 K
β = 112.593 (5)º	Plate, colourless
V = 1219.51 (16) Å^3	0.53 × 0.39 × 0.13 mm
Z = 4	

Data collection

Stoe IPDS 2 diffractometer	2392 independent reflections
Monochromator: plane graphite	1898 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	R_{int} = 0.134
T = 296 K	θ_{max} = 26.0º
ω scans	θ_{min} = 2.4º
Absorption correction: integration (X-RED; Stoe & Cie, 2002)	h = −11→11
T_{min} = 0.731, T_{max} = 0.955	k = −14→14
11707 measured reflections	l = −15→15

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.147$

$S = 1.03$

2392 reflections

213 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0766P)^2 + 0.1818P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.32$ e Å^{-3}

$\Delta\rho_{min} = -0.37$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.008 (1)

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4753 (3)	0.2984 (2)	0.3612 (2)	0.0528 (5)
C2	0.5622 (3)	0.34482 (18)	0.4771 (2)	0.0492 (5)
C3	0.5358 (3)	0.3266 (2)	0.5766 (2)	0.0584 (6)
C4	0.6373 (4)	0.3805 (2)	0.6741 (2)	0.0619 (6)
C5	0.7624 (3)	0.4495 (2)	0.6728 (2)	0.0620 (6)
C6	0.7886 (3)	0.4667 (2)	0.5734 (2)	0.0598 (6)
C7	0.6855 (3)	0.41331 (19)	0.4756 (2)	0.0491 (5)
C8	0.6824 (3)	0.4165 (2)	0.3563 (2)	0.0520 (5)
C9	0.8505 (3)	0.37712 (19)	0.2471 (2)	0.0495 (5)
C10	0.9761 (3)	0.3099 (2)	0.2418 (2)	0.0534 (5)
C11	1.0103 (3)	0.3104 (2)	0.1448 (2)	0.0578 (6)
C12	0.9220 (3)	0.3779 (2)	0.0514 (2)	0.0575 (6)
C13	0.7988 (3)	0.4440 (2)	0.0573 (2)	0.0545 (5)
C14	0.7618 (3)	0.4443 (2)	0.1535 (2)	0.0535 (6)
N1	0.8247 (2)	0.37584 (19)	0.34941 (19)	0.0557 (5)
O1	0.3639 (2)	0.23271 (17)	0.32605 (17)	0.0680 (5)
O2	0.54328 (19)	0.34162 (16)	0.29172 (15)	0.0578 (4)
O3	1.0593 (2)	0.24668 (18)	0.33773 (18)	0.0673 (5)
Cl1	0.68573 (8)	0.53075 (6)	−0.05833 (6)	0.0703 (3)
H1	0.679 (3)	0.489 (2)	0.154 (2)	0.051 (6)*
H2	1.091 (3)	0.264 (2)	0.141 (2)	0.056 (7)*

H3	0.449 (3)	0.279 (3)	0.578 (2)	0.072 (8)*
H4	0.654 (3)	0.492 (2)	0.320 (2)	0.058 (7)*
H5	0.829 (3)	0.485 (2)	0.740 (2)	0.063 (7)*
H6	0.873 (3)	0.514 (2)	0.575 (2)	0.063 (7)*
H7	0.949 (3)	0.383 (2)	−0.017 (3)	0.071 (8)*
H8	0.626 (4)	0.371 (3)	0.743 (3)	0.086 (10)*
H9	0.864 (3)	0.320 (2)	0.390 (2)	0.052 (7)*
H10	1.147 (4)	0.226 (3)	0.339 (3)	0.097 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0449 (11)	0.0537 (12)	0.0662 (14)	0.0042 (10)	0.0283 (10)	0.0003 (10)
C2	0.0474 (11)	0.0472 (11)	0.0587 (13)	0.0058 (9)	0.0268 (10)	0.0013 (9)
C3	0.0639 (14)	0.0550 (13)	0.0691 (15)	−0.0014 (11)	0.0398 (13)	0.0052 (11)
C4	0.0826 (17)	0.0532 (13)	0.0594 (15)	0.0075 (12)	0.0377 (13)	0.0042 (11)
C5	0.0720 (15)	0.0554 (13)	0.0582 (14)	0.0000 (12)	0.0245 (12)	−0.0059 (11)
C6	0.0613 (14)	0.0575 (13)	0.0662 (15)	−0.0063 (11)	0.0307 (12)	−0.0043 (11)
C7	0.0480 (11)	0.0468 (11)	0.0584 (13)	0.0054 (9)	0.0269 (10)	0.0009 (9)
C8	0.0495 (11)	0.0543 (12)	0.0596 (13)	0.0001 (10)	0.0291 (10)	−0.0017 (10)
C9	0.0439 (11)	0.0518 (11)	0.0601 (13)	−0.0067 (9)	0.0283 (10)	−0.0028 (10)
C10	0.0464 (11)	0.0512 (12)	0.0687 (14)	−0.0044 (9)	0.0288 (11)	0.0000 (10)
C11	0.0545 (13)	0.0537 (12)	0.0776 (16)	−0.0004 (10)	0.0391 (12)	−0.0062 (11)
C12	0.0589 (13)	0.0596 (13)	0.0647 (15)	−0.0096 (11)	0.0358 (12)	−0.0084 (11)
C13	0.0520 (12)	0.0570 (12)	0.0581 (13)	−0.0087 (10)	0.0253 (10)	−0.0030 (10)
C14	0.0450 (11)	0.0577 (12)	0.0644 (14)	−0.0008 (10)	0.0286 (10)	−0.0016 (11)
N1	0.0490 (10)	0.0636 (12)	0.0632 (12)	0.0065 (9)	0.0310 (9)	0.0081 (10)
O1	0.0509 (9)	0.0766 (11)	0.0786 (13)	−0.0112 (8)	0.0274 (9)	−0.0126 (9)
O2	0.0494 (8)	0.0721 (10)	0.0587 (10)	−0.0012 (7)	0.0282 (7)	−0.0063 (8)
O3	0.0554 (10)	0.0744 (11)	0.0813 (13)	0.0143 (9)	0.0366 (9)	0.0147 (9)
Cl1	0.0659 (4)	0.0854 (5)	0.0623 (4)	0.0049 (3)	0.0278 (3)	0.0081 (3)

Geometric parameters (Å, °)

C1—O1	1.201 (3)	C8—H4	0.98 (3)
C1—O2	1.348 (3)	C9—C14	1.386 (3)
C1—C2	1.474 (3)	C9—C10	1.398 (3)
C2—C7	1.373 (3)	C9—N1	1.401 (3)
C2—C3	1.385 (3)	C10—O3	1.368 (3)
C3—C4	1.373 (4)	C10—C11	1.376 (4)
C3—H3	0.96 (3)	C11—C12	1.385 (4)
C4—C5	1.388 (4)	C11—H2	0.92 (3)
C4—H8	0.93 (4)	C12—C13	1.374 (3)
C5—C6	1.382 (4)	C12—H7	0.98 (3)
C5—H5	0.93 (3)	C13—C14	1.381 (3)
C6—C7	1.377 (3)	C13—Cl1	1.744 (3)
C6—H6	0.94 (3)	C14—H1	0.92 (3)
C7—C8	1.500 (3)	N1—H9	0.82 (3)
C8—N1	1.404 (3)	O3—H10	0.83 (4)

C8—O2	1.486 (3)		
O1—C1—O2	121.3 (2)	O2—C8—H4	104.3 (15)
O1—C1—C2	130.5 (2)	C7—C8—H4	113.7 (16)
O2—C1—C2	108.22 (19)	C14—C9—C10	118.9 (2)
C7—C2—C3	121.7 (2)	C14—C9—N1	123.3 (2)
C7—C2—C1	108.7 (2)	C10—C9—N1	117.8 (2)
C3—C2—C1	129.6 (2)	O3—C10—C11	124.0 (2)
C4—C3—C2	117.4 (2)	O3—C10—C9	115.6 (2)
C4—C3—H3	120.6 (18)	C11—C10—C9	120.4 (2)
C2—C3—H3	122.1 (18)	C10—C11—C12	120.7 (2)
C3—C4—C5	121.0 (2)	C10—C11—H2	119.8 (16)
C3—C4—H8	121 (2)	C12—C11—H2	119.5 (16)
C5—C4—H8	118 (2)	C13—C12—C11	118.6 (2)
C6—C5—C4	121.3 (3)	C13—C12—H7	119.5 (17)
C6—C5—H5	119.6 (18)	C11—C12—H7	121.8 (17)
C4—C5—H5	119.1 (18)	C12—C13—C14	121.7 (2)
C7—C6—C5	117.5 (2)	C12—C13—Cl1	119.92 (19)
C7—C6—H6	122.8 (18)	C14—C13—Cl1	118.39 (19)
C5—C6—H6	119.7 (18)	C13—C14—C9	119.7 (2)
C2—C7—C6	121.2 (2)	C13—C14—H1	119.2 (16)
C2—C7—C8	109.1 (2)	C9—C14—H1	121.1 (16)
C6—C7—C8	129.7 (2)	C9—N1—C8	122.4 (2)
N1—C8—O2	111.98 (19)	C9—N1—H9	114.0 (19)
N1—C8—C7	113.0 (2)	C8—N1—H9	114.4 (19)
O2—C8—C7	102.92 (18)	C1—O2—C8	111.01 (18)
N1—C8—H4	110.4 (16)	C10—O3—H10	111 (3)
O1—C1—C2—C7	177.5 (2)	N1—C9—C10—O3	1.6 (3)
O2—C1—C2—C7	−1.8 (2)	C14—C9—C10—C11	−0.3 (3)
O1—C1—C2—C3	−1.5 (4)	N1—C9—C10—C11	−178.0 (2)
O2—C1—C2—C3	179.2 (2)	O3—C10—C11—C12	−179.1 (2)
C7—C2—C3—C4	0.5 (3)	C9—C10—C11—C12	0.5 (4)
C1—C2—C3—C4	179.4 (2)	C10—C11—C12—C13	−0.4 (4)
C2—C3—C4—C5	−0.7 (4)	C11—C12—C13—C14	0.0 (4)
C3—C4—C5—C6	0.3 (4)	C11—C12—C13—Cl1	179.65 (18)
C4—C5—C6—C7	0.3 (4)	C12—C13—C14—C9	0.2 (4)
C3—C2—C7—C6	0.2 (3)	Cl1—C13—C14—C9	−179.39 (17)
C1—C2—C7—C6	−179.0 (2)	C10—C9—C14—C13	−0.1 (3)
C3—C2—C7—C8	−179.8 (2)	N1—C9—C14—C13	177.5 (2)
C1—C2—C7—C8	1.1 (2)	C14—C9—N1—C8	17.8 (4)
C5—C6—C7—C2	−0.6 (4)	C10—C9—N1—C8	−164.5 (2)
C5—C6—C7—C8	179.3 (2)	O2—C8—N1—C9	67.8 (3)
C2—C7—C8—N1	−121.1 (2)	C7—C8—N1—C9	−176.5 (2)
C6—C7—C8—N1	59.0 (3)	O1—C1—O2—C8	−177.6 (2)
C2—C7—C8—O2	−0.1 (2)	C2—C1—O2—C8	1.7 (2)
C6—C7—C8—O2	180.0 (2)	N1—C8—O2—C1	120.7 (2)
C14—C9—C10—O3	179.4 (2)	C7—C8—O2—C1	−1.0 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H10···O1^{i}	0.83 (4)	2.03 (4)	2.820 (3)	160
C11—H2···O1^{i}	0.92 (3)	2.70 (3)	3.265 (3)	121
C4—H8···O2^{ii}	0.92 (4)	2.70 (4)	3.237 (3)	117
C5—H5···Cg1^{iii}	0.93 (2)	2.885	3.695 (3)	146

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*+2, −*y*+1, −*z*+1.

Fig. 1

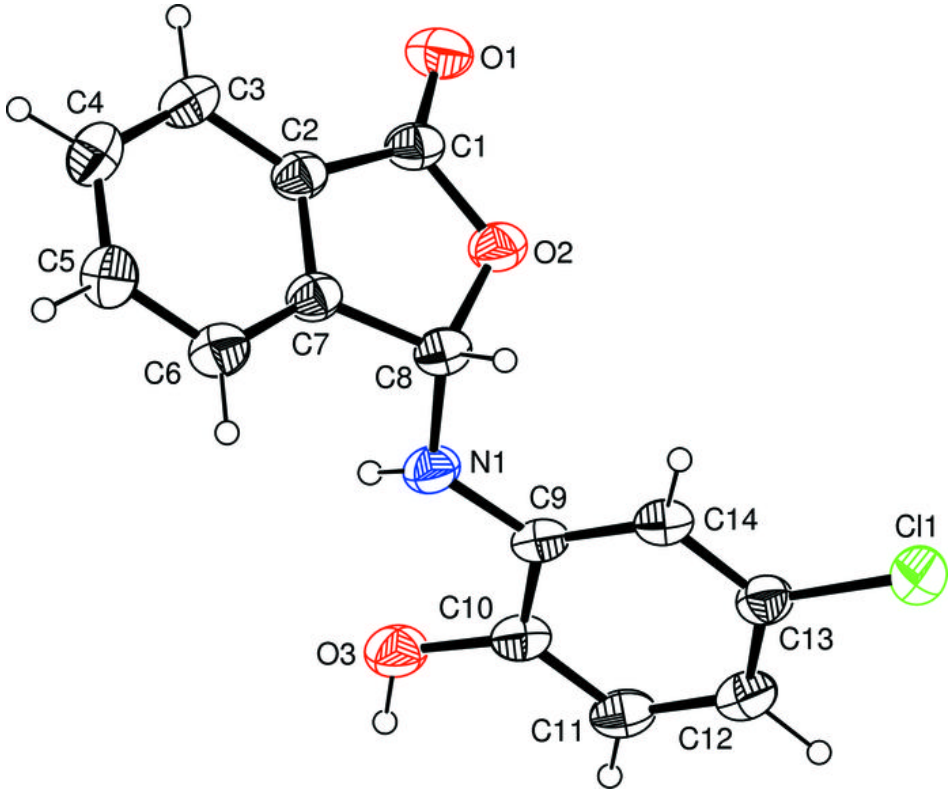

Fig. 2