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3-(2-Hydroxy-5-methylanilino)isobenzofuran-1(3H)-one¹Mustafa Odabaşoğlu^a and Orhan Büyükgüngör^{b*}

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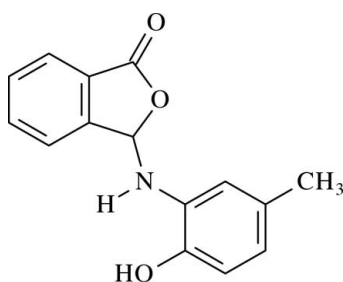
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.029; wR factor = 0.066; data-to-parameter ratio = 8.1.

In the molecule of the title compound, $\text{C}_{15}\text{H}_{13}\text{NO}_3$, the phthalide ring system is virtually planar, with a dihedral angle of $1.98(3)^\circ$ between the fused five- and six-membered rings. The substituted aromatic ring is oriented at a dihedral angle of $57.50(3)^\circ$ with respect to the phthalide ring system. In the crystal structure, intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules, forming a three-dimensional network.

Related literature

For a related structure, see: Odabaşoğlu & Büyükgüngör (2006). For ring-motif details, see: Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{13}\text{NO}_3$
 $M_r = 255.26$
 Orthorhombic, $Pna2_1$
 $a = 8.7198(5)$ Å
 $b = 15.5950(14)$ Å
 $c = 9.3992(6)$ Å
 $V = 1278.15(16)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 296$ K
 $0.64 \times 0.42 \times 0.28$ mm

Data collection

Stoe IPDSII diffractometer
 Absorption correction: integration
 ($X\text{-RED32}$; Stoe & Cie, 2002)
 $T_{\min} = 0.957$, $T_{\max} = 0.982$
 4658 measured reflections
 1418 independent reflections
 1196 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.066$
 $S = 0.99$
 1418 reflections
 175 parameters
 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.08$ e Å⁻³
 $\Delta\rho_{\min} = -0.09$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{O3}-\text{H3A}\cdots\text{O1}^i$ | 0.82 | 1.95 | 2.767 (2) | 173 |
| $\text{N1}-\text{H1}\cdots\text{O2}^{ii}$ | 0.86 | 2.78 | 3.5593 (19) | 152 |

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

Data collection: $X\text{-AREA}$ (Stoe & Cie, 2002); cell refinement: $X\text{-AREA}$; data reduction: $X\text{-RED32}$ (Stoe & Cie, 2002); 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); software used to prepare material for publication: $WinGX$ (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDSII diffractometer (purchased under grant F.279 of the University Research Fund).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2444).

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¹ 3-Substituted phthalides. Part XXXVII.

supplementary materials

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3-(2-Hydroxy-5-methylanilino)isobenzofuran-1(3H)-one

M. Odabasoglu and O. Büyükgüngör

Comment

The present work is part of a structural study of compounds of 3-substituted phthalides, and we report here the crystal structure of the title compound, (I).

The molecule of (I), (Fig. 1), is built up from a phthalimide unit connected to 2-hydroxy-5-methylphenyl group through an amino group. Rings A (C2-C7), B (C1/C2/C7/C8/O2) and C (C9-C14) are, of course, planar. The dihedral angles between them are A/B = 1.98 (3)°, A/C = 58.27 (3)° and B/C = 56.39 (3)°. So, rings A and B are also nearly coplanar. Ring C is oriented with respect to the coplanar ring system at a dihedral angle of 57.50 (3)°.

In the crystal structure, intermolecular O-H...O and N-H...O hydrogen bonds (Table 1) link the molecules by C(4) chains (Fig. 2) (Bernstein *et al.*, 1995; Etter, 1990), to form a three-dimensional network (Fig. 3), in which they may be effective in the stabilization of the structure.

Experimental

The title compound was prepared according to the method described by Odabaşođlu & Büyükgüngör (2006), using phthalaldehydic acid and 2-aminophenol as starting materials (yield; 80%). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol-DMF (1:1) solution at room temperature.

Refinement

H atoms were positioned geometrically, with O-H = 0.82 Å (for OH), N-H = 0.86 Å (for NH) and C-H = 0.93 and 0.98 Å for aromatic and methine H, respectively, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,O,N})$, where $x = 1.5$ for OH and NH H and $x = 1.2$ for all other H atoms.

Figures

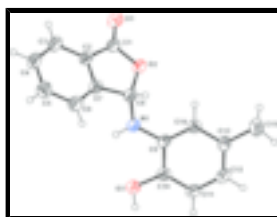


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

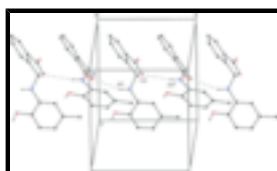


Fig. 2. A partial packing diagram of (I), showing the formation of C(4) chain along the [100] direction. Hydrogen bonds are shown as dashed lines [symmetry code: (i) 1 - x, -y, z + 1/2]. H atoms not involved in hydrogen bondings have been omitted for clarity.

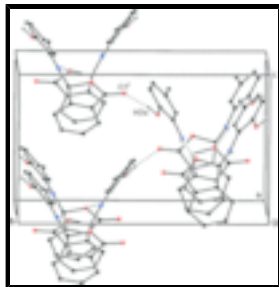


Fig. 3. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines [symmetry code: (i) $1 - x, -y, z + 1/2$]. H atoms not involved in hydrogen bondings have been omitted for clarity.

3-(2-Hydroxy-5-methylanilino)isobenzofuran-1(3H)-one

Crystal data

$C_{15}H_{13}NO_3$

$M_r = 255.26$

Orthorhombic, $Pna2_1$

Hall symbol: P 2c -2n

$a = 8.7198$ (5) Å

$b = 15.5950$ (14) Å

$c = 9.3992$ (6) Å

$V = 1278.15$ (16) Å³

$Z = 4$

$F_{000} = 536$

$D_x = 1.327$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4658 reflections

$\theta = 2.2$ – 27.2°

$\mu = 0.09$ mm⁻¹

$T = 296$ K

Prism, colorless

$0.64 \times 0.42 \times 0.28$ mm

Data collection

Stoe IPDSII
diffractometer

Monochromator: plane graphite

Detector resolution: 6.67 pixels mm⁻¹

$T = 296$ K

ω rotation method scans

Absorption correction: integration
(X-RED32; Stoe & Cie, 2002)

$T_{\min} = 0.957$, $T_{\max} = 0.982$

4658 measured reflections

1418 independent reflections

1196 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 26.7^\circ$

$\theta_{\min} = 2.5^\circ$

$h = -11 \rightarrow 10$

$k = -12 \rightarrow 19$

$l = -10 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.066$

$S = 0.99$

1418 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2]$

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

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

$\Delta\rho_{\max} = 0.08$ e Å⁻³

175 parameters

$$\Delta\rho_{\min} = -0.08 \text{ e } \text{\AA}^{-3}$$

1 restraint

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.014 (2)

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 $F^2 > 2\sigma(F^2)$ 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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| O1 | 0.40903 (18) | 0.07832 (10) | 0.36000 (19) | 0.0720 (4) |
| O2 | 0.45824 (13) | 0.20974 (10) | 0.43987 (15) | 0.0570 (4) |
| O3 | 0.20122 (14) | 0.44221 (12) | 0.70125 (18) | 0.0703 (5) |
| H3A | 0.1679 | 0.4796 | 0.7542 | 0.105* |
| N1 | 0.34673 (16) | 0.34164 (11) | 0.51886 (18) | 0.0520 (4) |
| H1 | 0.2484 | 0.3454 | 0.5210 | 0.062* |
| C1 | 0.3901 (2) | 0.15492 (14) | 0.3495 (2) | 0.0545 (5) |
| C2 | 0.3001 (2) | 0.20426 (14) | 0.2462 (2) | 0.0525 (5) |
| C3 | 0.2126 (2) | 0.17580 (17) | 0.1316 (3) | 0.0669 (6) |
| H3 | 0.2014 | 0.1177 | 0.1121 | 0.080* |
| C4 | 0.1436 (3) | 0.23701 (19) | 0.0486 (3) | 0.0749 (7) |
| H4 | 0.0837 | 0.2199 | -0.0283 | 0.090* |
| C5 | 0.1609 (3) | 0.32357 (18) | 0.0765 (3) | 0.0726 (6) |
| H5 | 0.1146 | 0.3636 | 0.0169 | 0.087* |
| C6 | 0.2468 (2) | 0.35171 (17) | 0.1926 (2) | 0.0631 (6) |
| H6 | 0.2576 | 0.4098 | 0.2129 | 0.076* |
| C7 | 0.31506 (19) | 0.28990 (15) | 0.2760 (2) | 0.0517 (5) |
| C8 | 0.41502 (19) | 0.30062 (13) | 0.4044 (2) | 0.0495 (4) |
| H8 | 0.5076 | 0.3322 | 0.3771 | 0.059* |
| C9 | 0.43056 (19) | 0.37720 (11) | 0.6310 (2) | 0.0433 (4) |
| C10 | 0.35434 (19) | 0.43126 (13) | 0.7257 (2) | 0.0491 (4) |
| C11 | 0.4335 (2) | 0.46949 (13) | 0.8349 (2) | 0.0533 (5) |
| H11 | 0.3819 | 0.5041 | 0.8997 | 0.064* |
| C12 | 0.5902 (2) | 0.45668 (13) | 0.8491 (2) | 0.0548 (5) |
| H12 | 0.6432 | 0.4842 | 0.9217 | 0.066* |
| C13 | 0.6683 (2) | 0.40359 (14) | 0.7568 (2) | 0.0511 (4) |
| C14 | 0.5859 (2) | 0.36305 (13) | 0.6500 (2) | 0.0493 (4) |
| H14 | 0.6364 | 0.3253 | 0.5895 | 0.059* |

supplementary materials

| | | | | |
|------|------------|------------|------------|------------|
| C15 | 0.8385 (2) | 0.3893 (2) | 0.7713 (3) | 0.0793 (7) |
| H15A | 0.8895 | 0.4095 | 0.6872 | 0.119* |
| H15B | 0.8584 | 0.3292 | 0.7833 | 0.119* |
| H15C | 0.8760 | 0.4201 | 0.8526 | 0.119* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| O1 | 0.0755 (9) | 0.0573 (10) | 0.0832 (11) | -0.0036 (7) | -0.0069 (9) | -0.0174 (9) |
| O2 | 0.0564 (6) | 0.0588 (9) | 0.0558 (7) | -0.0021 (6) | -0.0041 (7) | -0.0153 (7) |
| O3 | 0.0509 (7) | 0.0807 (12) | 0.0793 (10) | 0.0085 (7) | -0.0030 (8) | -0.0320 (8) |
| N1 | 0.0421 (7) | 0.0615 (10) | 0.0523 (9) | -0.0042 (7) | 0.0017 (7) | -0.0164 (8) |
| C1 | 0.0486 (9) | 0.0570 (12) | 0.0580 (11) | -0.0076 (9) | 0.0066 (9) | -0.0176 (11) |
| C2 | 0.0469 (9) | 0.0619 (13) | 0.0488 (10) | -0.0091 (8) | 0.0069 (8) | -0.0155 (10) |
| C3 | 0.0619 (11) | 0.0785 (17) | 0.0603 (12) | -0.0107 (11) | -0.0022 (10) | -0.0241 (13) |
| C4 | 0.0698 (13) | 0.095 (2) | 0.0599 (13) | -0.0043 (12) | -0.0101 (11) | -0.0271 (14) |
| C5 | 0.0718 (13) | 0.0891 (19) | 0.0571 (12) | 0.0046 (12) | -0.0071 (10) | -0.0059 (12) |
| C6 | 0.0643 (12) | 0.0654 (15) | 0.0597 (12) | -0.0035 (10) | -0.0028 (10) | -0.0063 (11) |
| C7 | 0.0454 (9) | 0.0633 (13) | 0.0465 (10) | -0.0068 (8) | 0.0053 (8) | -0.0110 (10) |
| C8 | 0.0457 (8) | 0.0528 (11) | 0.0501 (11) | -0.0061 (8) | 0.0012 (8) | -0.0103 (9) |
| C9 | 0.0503 (9) | 0.0392 (10) | 0.0405 (8) | -0.0068 (7) | 0.0015 (8) | -0.0007 (8) |
| C10 | 0.0501 (9) | 0.0467 (11) | 0.0506 (10) | -0.0026 (7) | 0.0022 (8) | -0.0032 (9) |
| C11 | 0.0655 (11) | 0.0466 (11) | 0.0480 (10) | 0.0007 (8) | -0.0011 (9) | -0.0087 (9) |
| C12 | 0.0683 (11) | 0.0502 (11) | 0.0460 (10) | -0.0103 (9) | -0.0089 (9) | -0.0013 (10) |
| C13 | 0.0526 (9) | 0.0551 (12) | 0.0456 (10) | -0.0060 (9) | -0.0040 (9) | 0.0050 (9) |
| C14 | 0.0502 (9) | 0.0523 (12) | 0.0453 (10) | -0.0004 (8) | 0.0044 (8) | -0.0021 (9) |
| C15 | 0.0531 (11) | 0.115 (2) | 0.0695 (14) | -0.0051 (12) | -0.0079 (11) | -0.0088 (15) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|-----------|-----------|-------------|
| O3—H3A | 0.8200 | C8—O2 | 1.504 (3) |
| N1—H1 | 0.8600 | C8—H8 | 0.9800 |
| C1—O1 | 1.210 (3) | C9—C14 | 1.384 (2) |
| C1—O2 | 1.344 (2) | C9—C10 | 1.394 (3) |
| C1—C2 | 1.466 (3) | C9—N1 | 1.397 (2) |
| C2—C7 | 1.371 (3) | C10—O3 | 1.366 (2) |
| C2—C3 | 1.393 (3) | C10—C11 | 1.373 (3) |
| C3—C4 | 1.372 (4) | C11—C12 | 1.387 (3) |
| C3—H3 | 0.9300 | C11—H11 | 0.9300 |
| C4—C5 | 1.383 (4) | C12—C13 | 1.379 (3) |
| C4—H4 | 0.9300 | C12—H12 | 0.9300 |
| C5—C6 | 1.395 (3) | C13—C14 | 1.387 (3) |
| C5—H5 | 0.9300 | C13—C15 | 1.506 (3) |
| C6—C7 | 1.378 (3) | C14—H14 | 0.9300 |
| C6—H6 | 0.9300 | C15—H15A | 0.9600 |
| C7—C8 | 1.498 (3) | C15—H15B | 0.9600 |
| C8—N1 | 1.386 (2) | C15—H15C | 0.9600 |
| O1—C1—O2 | 121.1 (2) | C14—C9—N1 | 123.10 (16) |

| | | | |
|---------------|--------------|-----------------|--------------|
| O1—C1—C2 | 130.19 (19) | C10—C9—N1 | 118.16 (15) |
| O2—C1—C2 | 108.75 (18) | O3—C10—C11 | 124.26 (17) |
| C7—C2—C3 | 121.4 (2) | O3—C10—C9 | 115.74 (16) |
| C7—C2—C1 | 108.97 (16) | C11—C10—C9 | 120.00 (16) |
| C3—C2—C1 | 129.7 (2) | C10—C11—C12 | 120.27 (18) |
| C4—C3—C2 | 117.3 (2) | C10—C11—H11 | 119.9 |
| C4—C3—H3 | 121.4 | C12—C11—H11 | 119.9 |
| C2—C3—H3 | 121.4 | C13—C12—C11 | 120.84 (17) |
| C3—C4—C5 | 121.5 (2) | C13—C12—H12 | 119.6 |
| C3—C4—H4 | 119.2 | C11—C12—H12 | 119.6 |
| C5—C4—H4 | 119.2 | C12—C13—C14 | 118.20 (16) |
| C4—C5—C6 | 120.9 (2) | C12—C13—C15 | 121.22 (18) |
| C4—C5—H5 | 119.5 | C14—C13—C15 | 120.58 (19) |
| C6—C5—H5 | 119.5 | C9—C14—C13 | 121.89 (17) |
| C7—C6—C5 | 117.2 (2) | C9—C14—H14 | 119.1 |
| C7—C6—H6 | 121.4 | C13—C14—H14 | 119.1 |
| C5—C6—H6 | 121.4 | C13—C15—H15A | 109.5 |
| C2—C7—C6 | 121.62 (19) | C13—C15—H15B | 109.5 |
| C2—C7—C8 | 109.19 (19) | H15A—C15—H15B | 109.5 |
| C6—C7—C8 | 129.2 (2) | C13—C15—H15C | 109.5 |
| N1—C8—C7 | 115.26 (14) | H15A—C15—H15C | 109.5 |
| N1—C8—O2 | 111.73 (16) | H15B—C15—H15C | 109.5 |
| C7—C8—O2 | 102.67 (15) | C8—N1—C9 | 122.95 (13) |
| N1—C8—H8 | 109.0 | C8—N1—H1 | 118.5 |
| C7—C8—H8 | 109.0 | C9—N1—H1 | 118.5 |
| O2—C8—H8 | 109.0 | C1—O2—C8 | 110.39 (16) |
| C14—C9—C10 | 118.71 (16) | C10—O3—H3A | 109.5 |
| O1—C1—C2—C7 | -179.2 (2) | N1—C9—C10—O3 | 1.9 (3) |
| O2—C1—C2—C7 | -0.1 (2) | C14—C9—C10—C11 | 0.2 (3) |
| O1—C1—C2—C3 | -0.4 (4) | N1—C9—C10—C11 | -178.11 (17) |
| O2—C1—C2—C3 | 178.76 (19) | O3—C10—C11—C12 | -178.1 (2) |
| C7—C2—C3—C4 | 0.8 (3) | C9—C10—C11—C12 | 2.0 (3) |
| C1—C2—C3—C4 | -177.9 (2) | C10—C11—C12—C13 | -1.9 (3) |
| C2—C3—C4—C5 | 0.5 (3) | C11—C12—C13—C14 | -0.3 (3) |
| C3—C4—C5—C6 | -1.5 (4) | C11—C12—C13—C15 | 180.0 (2) |
| C4—C5—C6—C7 | 1.0 (3) | C10—C9—C14—C13 | -2.5 (3) |
| C3—C2—C7—C6 | -1.3 (3) | N1—C9—C14—C13 | 175.70 (19) |
| C1—C2—C7—C6 | 177.66 (17) | C12—C13—C14—C9 | 2.6 (3) |
| C3—C2—C7—C8 | 179.96 (17) | C15—C13—C14—C9 | -177.7 (2) |
| C1—C2—C7—C8 | -1.11 (19) | C7—C8—N1—C9 | -162.60 (18) |
| C5—C6—C7—C2 | 0.3 (3) | O2—C8—N1—C9 | 80.7 (2) |
| C5—C6—C7—C8 | 178.81 (19) | C14—C9—N1—C8 | -10.7 (3) |
| C2—C7—C8—N1 | -120.00 (19) | C10—C9—N1—C8 | 167.55 (18) |
| C6—C7—C8—N1 | 61.4 (3) | O1—C1—O2—C8 | -179.57 (18) |
| C2—C7—C8—O2 | 1.72 (17) | C2—C1—O2—C8 | 1.21 (19) |
| C6—C7—C8—O2 | -176.92 (18) | N1—C8—O2—C1 | 122.30 (16) |
| C14—C9—C10—O3 | -179.76 (17) | C7—C8—O2—C1 | -1.79 (17) |

supplementary materials

Hydrogen-bond geometry (Å, °)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------|-------|-------------|-------------|---------------|
| $O3-H3A\cdots O1^i$ | 0.82 | 1.95 | 2.767 (2) | 173 |
| $N1-H1\cdots O2^{ii}$ | 0.86 | 2.78 | 3.5593 (19) | 152 |

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

Fig. 1

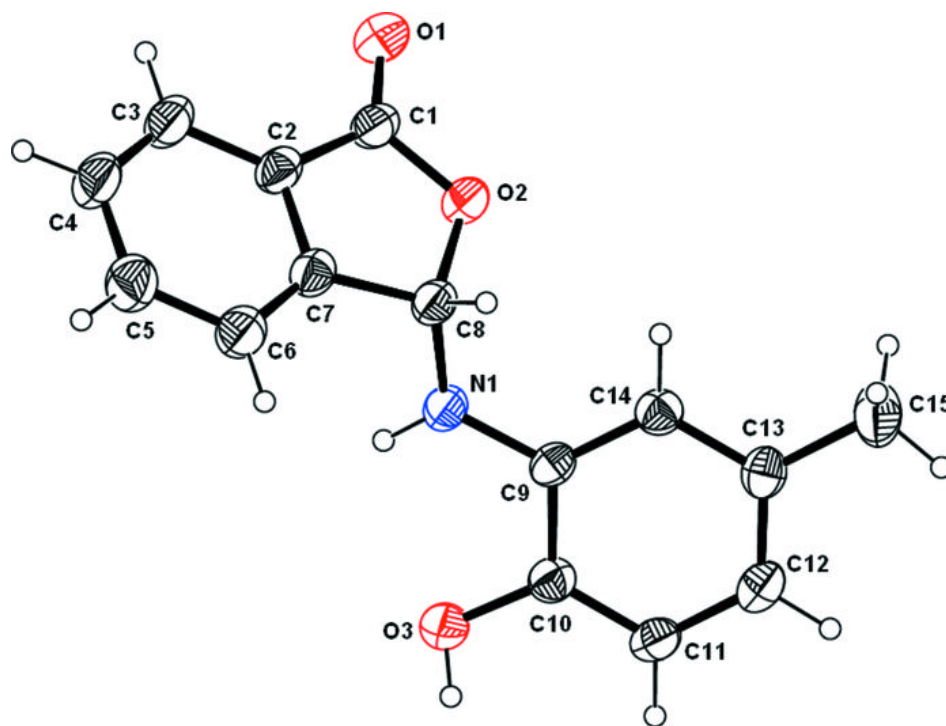


Fig. 2

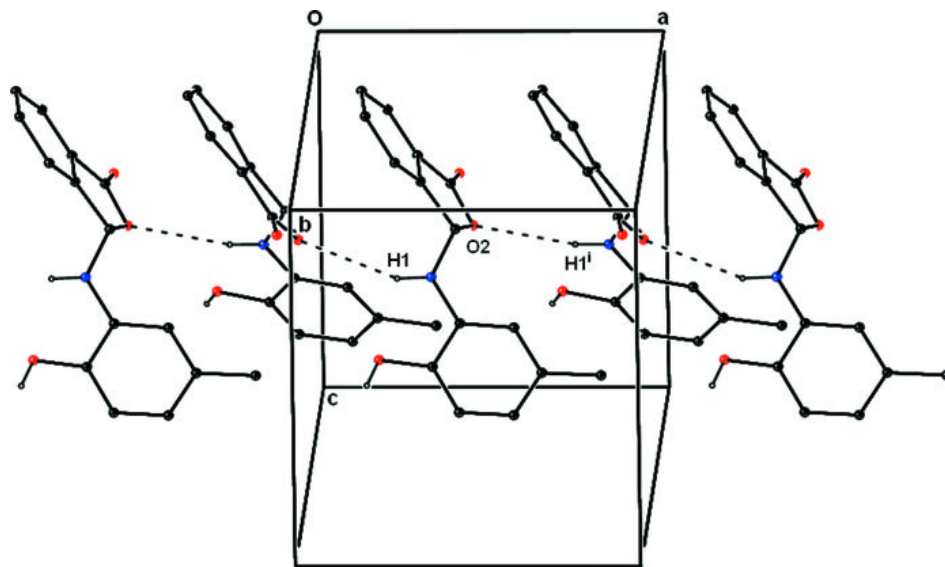


Fig. 3

