Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[(2-Hydroxy-5-nitrophenyl)amino]-2-benzofuran-1(3*H*)-one monohydrate

Mustafa Odabaşoğlu[a]* and Orhan Büyükgüngör[b]

[a]Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey, and [b]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey

Correspondence e-mail: muodabas@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.115
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

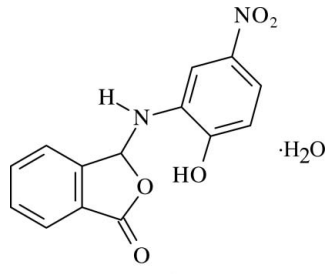
The crystal structure of the title compound, $C_{14}H_{10}N_2O_5\cdot H_2O$, is stabilized by inversion-related O—H···O and C—H···O intermolecular hydrogen bonds and also by π–π interactions. The dihedral angle between the phthalide group and the benzene ring is 51.45 (8)°.

Received 16 May 2006
Accepted 22 June 2006

3-Substituted phthalides, Part XI.

Comment

The present work is part of a structural study of compounds of 3-substituted phthalides and we report here the structure of 3-(2-hydroxy-5-nitrophenyl)aminoisobenzofuran-1(3*H*)-one, (I) (Fig. 1 and Table 1).



(I)

The phthalide group (C1–C8/O2) is planar, the largest deviation from the mean plane being 0.037 (1) Å for atom O2. The dihedral angle between the mean planes of the phthalide group and the benzene ring is 51.45 (8)°; that between the nitro group and the benzene ring is 2.52 (16)°.

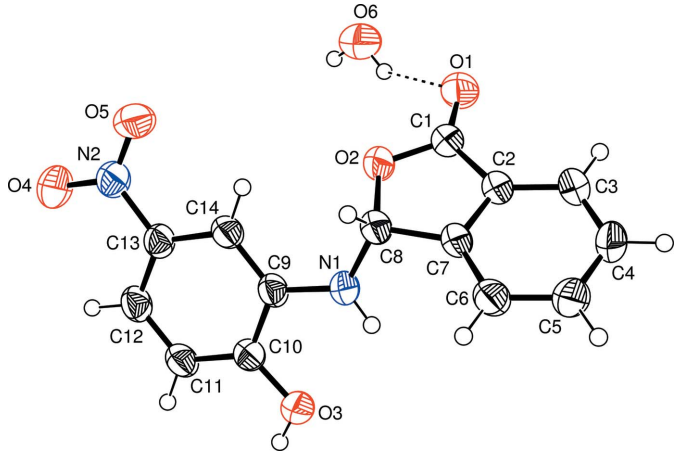


Figure 1
The asymmetric unit of (I), showing the atomic numbering scheme, with displacement ellipsoids drawn at the 50% probability level and a hydrogen bond shown as a dashed line.

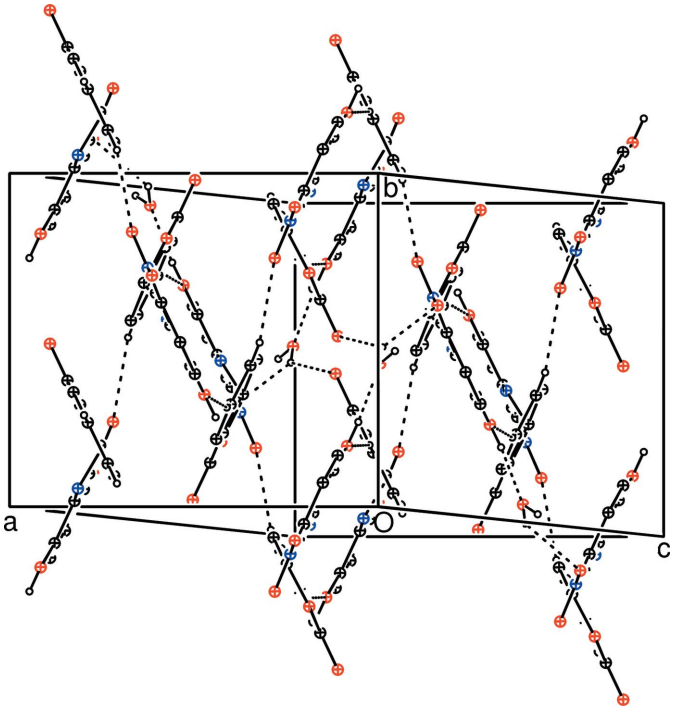


Figure 2
A packing diagram for (I), with hydrogen-bond interactions drawn as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

The crystal packing is stabilized by inversion-related O—H···O and C—H···O intermolecular hydrogen bonds (Fig. 2 and Table 2) and π–π interactions [$Cg1\cdots Cg1^{vi}$ = 3.563 (1) Å; symmetry code: (vi) $2-x, 1-y, 1-z$; perpendicular distance = 3.400 (12) Å; $Cg1$ is the centroid of the C9–C14 ring].

Experimental

The title compound was prepared as described by Odabaşoğlu & Büyükgüngör (2006), using phthalaldehydic acid and 2-hydroxy-4-nitroaniline as starting materials (yield 83%, m.p. 526–527 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol (95%) solution at room temperature.

Crystal data

$C_{14}H_{10}N_2O_5\cdot H_2O$
M_r = 304.26
Monoclinic, $P2_1/c$
a = 10.8202 (7) Å
b = 9.8139 (4) Å
c = 15.1475 (10) Å
β = 123.588 (4)°
V = 1339.93 (14) Å^3
Z = 4
D_x = 1.508 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
Prism, light brown
0.45 × 0.34 × 0.14 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.952, T_{max} = 0.984
18837 measured reflections
2628 independent reflections
2077 reflections with $I > 2\sigma(I)$
R_{int} = 0.090
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 1.02
2628 reflections
207 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.1854P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.207 (2)	C7—C8	1.501 (2)
C1—O2	1.3502 (19)	C9—N1	1.3949 (19)
C2—C7	1.377 (2)	C13—N2	1.450 (2)
O1—C1—O2	121.19 (14)	N1—C8—O2	112.19 (13)
O1—C1—C2	130.00 (15)		
C14—C13—N2—O4	179.99 (15)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O6^{i}	0.82	1.84	2.643 (2)	166
O6—H6A···O1	0.790 (19)	2.34 (4)	2.936 (3)	133 (4)
O6—H6A···O4^{ii}	0.790 (19)	2.36 (4)	3.011 (2)	141 (5)
O6—H6B···O6^{iii}	0.794 (19)	2.34 (4)	2.937 (4)	133 (5)
C4—H4···O3^{iv}	0.93	2.60	3.500 (2)	164
C5—H5···O5^{v}	0.93	2.54	3.367 (2)	148

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

The water H atoms were refined with distance restraints O—H = 0.83 (2) Å and H6A···H6B = 1.20 (2) Å. All other H atoms were refined using the riding model approximation, with C—H = 0.93 Å for aromatic and 0.98 Å for methine, N—H = 0.86 Å, and O—H = 0.82 Å, with U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{eq}(O).

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).

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

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Odabaşoğlu, M. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o1879–o1881.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.