3-(4-Hydroxyanilino)isobenzofuran-1(3*H*)-one

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The title compound, $C_{14}H_{11}NO_3$, is stabilized by inter-molecular O—H···O, N—H···O and C—H···O hydrogen bonds, and by three C—H···π intermolecular interactions. The N—H···O and O—H···O hydrogen bonds generate edge-fused $R_4^4(26)$ rings. The phthalide ring system of the molecule is almost planar and forms a dihedral angle of 63.02 (7)° with the second benzene ring.

Comment

Phthalides (isobenzofuranones) are five-membered lactones found in plants and are known to show diverse biological activities as hormones, pheromones and antibiotics. These compounds possess several important properties, such as fungicidal (Aoki *et al.*, 1973; Lacova, 1973), bactericidal and herbicidal (Lacova, 1973), analgesic (Elderfield, 1951), and hypotensive 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 phthal-ides by X-ray diffraction methods and present here the first of these, the title compound, (I).
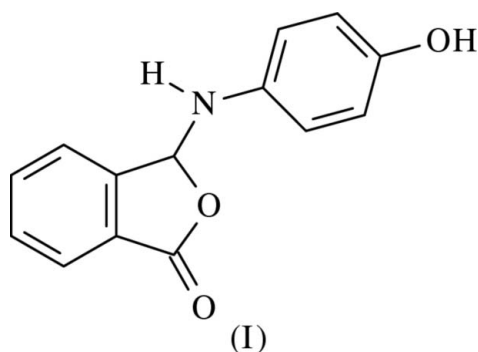
The overall view and atom-labelling of the molecule of (I) are displayed in Fig. 1. Bond lengths and angles are presented in Table 1, hydrogen-bonding parameters are given in Table 2 and the packing arrangement of the molecules is illustrated in Fig. 2. The phthalide ring system (O1/C1–C8) is essentially planar, the largest deviation from the mean plane being 0.016 (2) Å for atom C1. The dihedral angle between the five-membered ring and the fused six-membered ring is 1.86 (10)°, whereas the dihedral angle between the second benzene ring and the mean plane of the phthalide ring system is 63.02 (7)°.
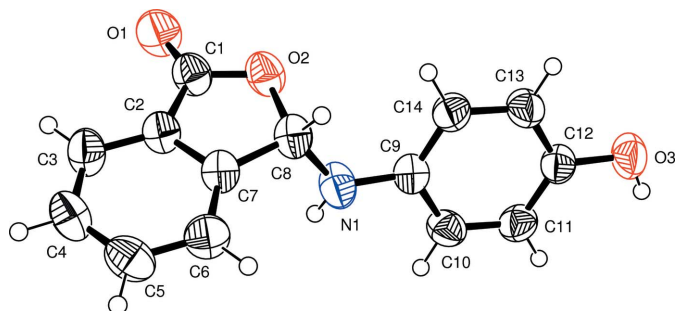
Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
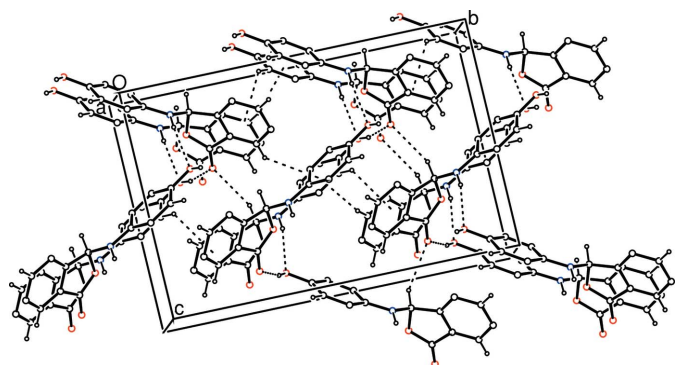

Figure 2
A packing diagram of (I), showing the hydrogen-bonding scheme and C—H···π stacking interactions as dashed lines. H atoms not involved in intermolecular interactions have been omitted for clarity.

The molecular dimensions in (I) are comparable with those observed in diisopropyl phthalide-3-phosphonate [Slowi-kowska *et al.*, 1998; refcode BEYQIF in the Cambridge Structural Database (CSD, Version 5.26; Allen, 2002)], the most significant differences being in the values of the C1—O2 and C8—O2 bond distances [1.337 (2) and 1.519 (2) Å in (I), and 1.370 (6) and 1.456 (5) Å in BEYQIF, respectively] and the O1—C1—C2 bond angle [127.58 (17)° in (I) and 131.5 (5)° in BEYQIF]. These discrepancies are obviously a structural response to the different substituent at C8.

Molecules of (I) are linked into zigzag chains running parallel to the *c* axis by intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds (Table 2). The N—H···O and O—H···O hydrogen-bond interactions generate edge-fused $R_4^4(26)$ rings (Etter, 1990). There are no direction-specific aromatic π–π interactions between adjacent chains, but there are three C—H···π interactions (Table 2).

Experimental

The title compound was prepared by a similar method to that described by Wheeler *et al.* (1957). A solution of phthalaldehydic acid (1.50 g, 0.01 mol) and *p*-hydroxyaniline (1.09 g, 0.01 mol) in 75% ethanol (100 ml) was refluxed for 30 min. The crystalline solid that separated was isolated from the cooled mixture (yield 90%; mp. 459–460 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a solution in 95% ethanol at room temperature.

$C_{14}H_{11}NO_3$	$Z = 4$
$M_r = 241.24$	$D_x = 1.337$ Mg m^{-3}
Monoclinic, Cc	Mo $K\alpha$ radiation
$a = 5.9049 (4)$ Å	$\mu = 0.10$ mm^{-1}
$b = 17.4841 (16)$ Å	$T = 296 (2)$ K
$c = 11.7553 (8)$ Å	Prism, brown
$\beta = 99.113 (6)°$	$0.60 \times 0.52 \times 0.39$ mm
$V = 1198.32 (16)$ Å^3	

Data collection

Stoe IPDS-2 diffractometer	8507 measured reflections
ω scans	1183 independent reflections
Absorption correction: integration	1138 reflections with $I > 2\sigma(I)$
(X-$RED32$; Stoe & Cie, 2002)	$R_{int} = 0.031$
$T_{min} = 0.940$, $T_{max} = 0.971$	$\theta_{max} = 26.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$+ 0.0931P]$
$wR(F^2) = 0.067$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
1183 reflections	$\Delta\rho_{max} = 0.10$ e Å^{-3}
168 parameters	$\Delta\rho_{min} = -0.10$ e Å^{-3}
H atoms treated by a mixture of	Extinction correction: $SHELXL97$
independent and constrained	(Sheldrick, 1997)
refinement	Extinction coefficient: 0.025 (4)

Table 1
Selected geometric parameters (Å, °).

C2—C7	1.371 (2)	C8—O2	1.519 (2)
C2—C1	1.457 (2)	C1—O1	1.216 (2)
C7—C8	1.501 (2)	C9—N1	1.401 (2)
C8—N1	1.387 (2)	C12—O3	1.368 (2)
C7—C2—C1	108.56 (15)	C7—C8—O2	101.75 (14)
C2—C7—C6	120.33 (17)	O1—C1—O2	122.81 (17)
C2—C7—C8	109.81 (16)	O1—C1—C2	127.58 (17)
N1—C8—C7	113.02 (17)	C8—N1—C9	124.65 (16)
N1—C8—O2	113.01 (15)	C1—O2—C8	110.18 (13)
C3—C2—C1—O1	−1.0 (3)	O1—C1—O2—C8	−177.37 (19)
C14—C9—N1—C8	0.1 (3)		

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

$Cg1$ and $Cg2$ are the centroids of the C2–C7 and C9–C14 benzene rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O1^i	0.82	1.87	2.673 (2)	165
N1—H1···O3^{ii}	0.85 (3)	2.23 (3)	2.972 (2)	147 (2)
C8—H8···O1^{iii}	0.98	2.59	3.417 (2)	143
C3—H3···$Cg2^{iv}$	0.93	3.00	3.777 (2)	142
C5—H5···$Cg2^v$	0.93	2.70	3.513 (2)	147
C11—H11···$Cg1^{vi}$	0.93	3.05	3.722 (2)	130

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

The N-bound H atom was located in a difference Fourier map and refined freely. All other H atoms were refined using the riding-model approximation, with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$. In the absence of significant anomalous scattering effects, Friedel pairs were merged in the final refinement.

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, 1990); 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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