organic compounds

3-(2-Bromoanilino)isobenzofuran-1(3H)-one[1]

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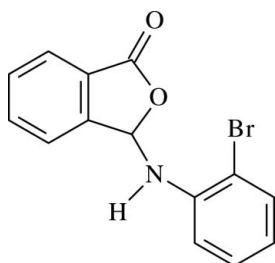Correspondence e-mail: muodabas@omu.edu.tr

The title compound, $C_{14}H_{10}BrNO_2$, contains N—H···O, N—H···Br, C—H···O and C—H···Br hydrogen-bond interactions which generate $S(5)$, $R_4^4(27)$ and $R_6^6(38)$ ring motifs. The phthalide unit is planar and is inclined to the benzene ring of the aminophenyl group at an angle of 58.24 (18)°.

Related literature

For related structures (3-halogenophenyl phthalides, halogens = I, Br, Cl, F), see: Büyükgüngör & Odabaşoğlu (2006, 2007); Odabaşoğlu & Büyükgüngör (2007, 2006a,b,c). For ring motif details, see: Bernstein et al. (1995); Etter (1990). For general background, see: Aoki et al. (1973, 1974); Lacova (1973, 1974); Elderfield (1951); Tsi & Tan (1997); Bellasio (1974, 1975); Roy & Sarkar (2005).

Experimental

Crystal data

$C_{14}H_{10}BrNO_2$	V = 1261.71 (19) Å³
M_r = 304.14	Z = 4
Monoclinic, Cc	Mo $K\alpha$ radiation
a = 9.2494 (9) Å	μ = 3.25 mm⁻¹
b = 22.9431 (17) Å	T = 296 K
c = 7.1850 (6) Å	0.76 × 0.44 × 0.14 mm
β = 124.157 (6)°	

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[1] 3-Substituted phthalides. XXXI. For Part XXX, see Odabaşoğlu & Büyükgüngör (2007).

Data collection

STOE IPDS II diffractometer	9973 measured reflections
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	1381 independent reflections
	1309 reflections with $I > 2\sigma(I)$
T_{min} = 0.205, T_{max} = 0.614	R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027	2 restraints
$wR(F^2)$ = 0.058	H-atom parameters constrained
S = 1.11	$\Delta\rho_{max}$ = 0.38 e Å⁻³
1381 reflections	$\Delta\rho_{min}$ = −0.32 e Å⁻³
164 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.86	2.49	3.130 (5)	132
C4—H4···O2^ii	0.93	2.54	3.394 (5)	153
C11—H11···Br1^iii	0.93	2.99	3.845 (4)	154
N1—H1···Br1	0.86	2.60	3.058 (3)	114

Symmetry codes: (i) $x, -y + 1, z - \frac{1}{2}$; (ii) $x + 1, -y + 1, z + \frac{1}{2}$; (iii) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$.

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 materials

3-(2-Bromoanilino)isobenzofuran-1(3*H*)-one

M. Odabasoglu and O. Büyükgüngör

Comment

We report here the structure of 3-(2-bromoanilino)isobenzofuran-1(3*H*)-one, (I) (Fig. 1), and we briefly compare this with the structure of the 4-bromo analogue, which have been previously reported (Odabaşoğlu & Büyükgüngör, 2006*b*). The dihedral angle between the *o*-bromophenyl ring and the mean plane of the phthalide group is 58.24 (18)°; for comparison, this angles is 62.2 (2)° in 3-(4-bromoanilino)phthalide.

The phthalide group (C1–C8/O2) is essentially planar, with the largest deviation from the mean plane being −0.025 (4)Å for atom C7. In (I), the crystal packing is stabilized by N—H⋯Br intramolecular, N—H⋯O, C—H⋯O and C—H⋯Br intermolecular hydrogen bonds which generate S(5), $R_4^4(27)$ and $R_6^6(38)$ ring motifs (Fig. 1, 2, 3 and Table 1) in three-dimensional network (Fig. 4). In its *p*-bromo analogue there was only N—H⋯O intermolecular hydrogen bonds which generate C(6) chains and $R_3^4(21)$ ring motifs (Odabaşoğlu & Büyükgüngör, 2006*b*).

Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2006*b*), using phthalaldehydic acid and 2-bromoaniline as starting materials (yield 87%; m.p. 443–445 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol-DMF (*v/v*, 1/1) solution at room temperature.

Refinement

The H atoms were included in calculated positions and refined using a riding model approximation. Constrained C—H and N—H bond lengths and isotropic U parameters: 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for Csp^2—H; 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methine C—H; 0.86 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for N—H. The absolute structure could not be determined, and 1366 Friedel pairs were averaged before the last refinement.
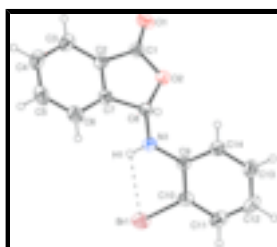
Figures

Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
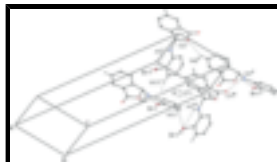
Fig. 2. Part of the crystal structure of (I), showing the formation of $R_4^4(27)$ ring with four edge-fussed S(5) hydrogen bonded motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $x + 1/2, y, z + 1/2$; (ii) $x − 1/2, y + 1/2, z$; (iii) $x − 1/2, y, z − 1/2$].
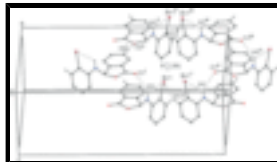

Fig. 3. Part of the crystal structure of (I), showing the formation of $R_6^6(38)$ ring motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $x, y − 1/2, z − 1/2$; (ii) $x + 1, y, z$; (iii) $x + 1/2, y, z − 1/2$; (iv) $x + 1/2, y + 1/2, z$; (v) $x + 1/2, y, z + 1/2$].

Fig. 4. A packing diagram of (I), with hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

3-(2-Bromoanilino)isobenzofuran-1(3*H*)-one

Crystal data

$C_{14}H_{10}BrNO_2$	$F_{000} = 608$
$M_r = 304.14$	$D_x = 1.601$ Mg m^{-3}
Monoclinic, Cc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: C -2yc	Cell parameters from 9973 reflections
$a = 9.2494 (9)$ Å	$\theta = 2.8–27.3°$
$b = 22.9431 (17)$ Å	$\mu = 3.25$ mm^{-1}
$c = 7.1850 (6)$ Å	$T = 296$ K
$\beta = 124.157 (6)°$	Prism, colourless
$V = 1261.71 (19)$ Å^3	$0.76 \times 0.44 \times 0.14$ mm
$Z = 4$	

Data collection

STOE IPDS 2 diffractometer	1381 independent reflections
Monochromator: plane graphite	1309 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.061$
$T = 296$ K	$\theta_{max} = 27.0°$
w–scan rotation method	$\theta_{min} = 2.8°$
Absorption correction: integration	$h = −11 \rightarrow 11$

(X-RED32; Stoe & Cie, 2002)

$T_{min} = 0.205$, $T_{max} = 0.614$

$k = -29 \rightarrow 29$

9973 measured reflections

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0224P)^2 + 0.5946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.027$	$(\Delta/\sigma)_{max} = <0.001$
$wR(F^2) = 0.058$	$\Delta\rho_{max} = 0.38$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.32$ e Å^{-3}
1381 reflections	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
164 parameters	Extinction coefficient: 0.0108 (9)
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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	0.3791 (5)	0.48218 (16)	0.5796 (6)	0.0423 (8)
C2	0.5519 (5)	0.50483 (16)	0.6507 (6)	0.0405 (7)
C3	0.7000 (6)	0.47466 (19)	0.7055 (7)	0.0524 (9)
H3	0.7002	0.4342	0.6972	0.063*
C4	0.8469 (6)	0.5068 (2)	0.7729 (8)	0.0617 (12)
H4	0.9489	0.4878	0.8107	0.074*
C5	0.8453 (6)	0.5667 (3)	0.7854 (8)	0.0641 (12)
H5	0.9469	0.5874	0.8330	0.077*
C6	0.6968 (5)	0.5969 (2)	0.7293 (8)	0.0554 (10)
H6	0.6964	0.6374	0.7368	0.066*
C7	0.5485 (5)	0.56462 (15)	0.6614 (6)	0.0399 (7)

C8	0.3724 (5)	0.58397 (15)	0.6007 (6)	0.0398 (7)
H8	0.3845	0.6026	0.7315	0.048*
C9	0.1359 (4)	0.65174 (14)	0.3574 (6)	0.0371 (7)
C10	0.0647 (5)	0.69323 (16)	0.1867 (6)	0.0421 (8)
C11	−0.0816 (6)	0.7249 (2)	0.1282 (7)	0.0572 (11)
H11	−0.1251	0.7528	0.0150	0.069*
C12	−0.1633 (6)	0.7155 (2)	0.2361 (9)	0.0642 (12)
H12	−0.2629	0.7365	0.1959	0.077*
C13	−0.0957 (6)	0.6742 (2)	0.4062 (8)	0.0562 (10)
H13	−0.1499	0.6676	0.4810	0.067*
C14	0.0507 (5)	0.64281 (17)	0.4649 (7)	0.0467 (8)
H14	0.0938	0.6151	0.5786	0.056*
N1	0.2874 (4)	0.62233 (13)	0.4174 (5)	0.0425 (7)
H1	0.3305	0.6278	0.3395	0.051*
O1	0.3280 (4)	0.43296 (12)	0.5509 (6)	0.0588 (7)
O2	0.2769 (3)	0.52800 (11)	0.5530 (5)	0.0465 (6)
Br1	0.17541 (10)	0.708652 (17)	0.03497 (12)	0.05955 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0436 (19)	0.045 (2)	0.0384 (18)	−0.0001 (16)	0.0230 (16)	0.0042 (15)
C2	0.0369 (17)	0.047 (2)	0.0341 (17)	0.0034 (15)	0.0175 (14)	0.0058 (14)
C3	0.049 (2)	0.060 (2)	0.044 (2)	0.0153 (19)	0.0236 (17)	0.0062 (17)
C4	0.042 (2)	0.087 (4)	0.052 (2)	0.017 (2)	0.0236 (19)	0.010 (2)
C5	0.037 (2)	0.093 (4)	0.057 (2)	−0.015 (2)	0.0228 (18)	−0.001 (2)
C6	0.047 (2)	0.059 (2)	0.055 (2)	−0.0107 (19)	0.0253 (18)	0.0002 (19)
C7	0.0362 (17)	0.0424 (19)	0.0378 (17)	−0.0012 (14)	0.0188 (14)	−0.0007 (14)
C8	0.0402 (18)	0.0373 (17)	0.0419 (18)	−0.0011 (15)	0.0231 (15)	0.0032 (14)
C9	0.0369 (17)	0.0322 (16)	0.0395 (17)	−0.0009 (13)	0.0198 (14)	−0.0041 (13)
C10	0.050 (2)	0.0370 (18)	0.0379 (17)	0.0005 (15)	0.0240 (16)	−0.0018 (14)
C11	0.059 (3)	0.056 (3)	0.047 (2)	0.019 (2)	0.0239 (19)	0.0099 (18)
C12	0.059 (3)	0.065 (3)	0.065 (3)	0.028 (2)	0.033 (2)	0.006 (2)
C13	0.053 (2)	0.060 (3)	0.066 (3)	0.0067 (19)	0.040 (2)	−0.003 (2)
C14	0.054 (2)	0.0404 (19)	0.051 (2)	0.0038 (16)	0.0323 (18)	0.0030 (16)
N1	0.0458 (17)	0.0412 (16)	0.0467 (16)	0.0079 (13)	0.0298 (14)	0.0089 (13)
O1	0.071 (2)	0.0399 (14)	0.0715 (19)	−0.0112 (13)	0.0435 (16)	−0.0005 (13)
O2	0.0397 (13)	0.0407 (14)	0.0613 (16)	0.0001 (10)	0.0298 (12)	0.0056 (12)
Br1	0.0784 (3)	0.0595 (2)	0.0511 (2)	0.0075 (3)	0.04267 (19)	0.0116 (3)

Geometric parameters (Å, °)

C1—O1	1.196 (5)	C8—O2	1.486 (4)
C1—O2	1.354 (5)	C8—H8	0.9800
C1—C2	1.472 (5)	C9—N1	1.388 (5)
C2—C7	1.375 (5)	C9—C10	1.392 (5)
C2—C3	1.380 (5)	C9—C14	1.394 (5)
C3—C4	1.373 (7)	C10—C11	1.378 (6)
C3—H3	0.9300	C10—Br1	1.901 (4)

C4—C5	1.379 (8)	C11—C12	1.371 (7)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.381 (7)	C12—C13	1.387 (7)
C5—H5	0.9300	C12—H12	0.9300
C6—C7	1.385 (5)	C13—C14	1.374 (6)
C6—H6	0.9300	C13—H13	0.9300
C7—C8	1.497 (5)	C14—H14	0.9300
C8—N1	1.401 (5)	N1—H1	0.8600
O1—C1—O2	122.1 (3)	O2—C8—H8	109.3
O1—C1—C2	129.8 (4)	C7—C8—H8	109.3
O2—C1—C2	108.1 (3)	N1—C9—C10	120.1 (3)
C7—C2—C3	122.3 (4)	N1—C9—C14	122.9 (3)
C7—C2—C1	108.5 (3)	C10—C9—C14	117.0 (3)
C3—C2—C1	129.2 (4)	C11—C10—C9	121.8 (4)
C4—C3—C2	117.3 (4)	C11—C10—Br1	118.7 (3)
C4—C3—H3	121.3	C9—C10—Br1	119.5 (3)
C2—C3—H3	121.3	C12—C11—C10	120.2 (4)
C3—C4—C5	120.9 (4)	C12—C11—H11	119.9
C3—C4—H4	119.5	C10—C11—H11	119.9
C5—C4—H4	119.5	C11—C12—C13	119.1 (4)
C4—C5—C6	121.8 (4)	C11—C12—H12	120.4
C4—C5—H5	119.1	C13—C12—H12	120.4
C6—C5—H5	119.1	C14—C13—C12	120.5 (4)
C5—C6—C7	117.4 (4)	C14—C13—H13	119.7
C5—C6—H6	121.3	C12—C13—H13	119.7
C7—C6—H6	121.3	C13—C14—C9	121.3 (4)
C2—C7—C6	120.3 (4)	C13—C14—H14	119.4
C2—C7—C8	109.6 (3)	C9—C14—H14	119.4
C6—C7—C8	130.1 (3)	C9—N1—C8	122.2 (3)
N1—C8—O2	112.3 (3)	C9—N1—H1	118.9
N1—C8—C7	113.9 (3)	C8—N1—H1	118.9
O2—C8—C7	102.7 (3)	C1—O2—C8	111.1 (3)
N1—C8—H8	109.3		
O1—C1—C2—C7	−179.6 (4)	N1—C9—C10—C11	−177.5 (4)
O2—C1—C2—C7	−1.1 (4)	C14—C9—C10—C11	1.1 (5)
O1—C1—C2—C3	−1.0 (7)	N1—C9—C10—Br1	0.6 (4)
O2—C1—C2—C3	177.5 (4)	C14—C9—C10—Br1	179.1 (3)
C7—C2—C3—C4	0.1 (6)	C9—C10—C11—C12	−1.1 (6)
C1—C2—C3—C4	−178.3 (4)	Br1—C10—C11—C12	−179.1 (4)
C2—C3—C4—C5	0.2 (7)	C10—C11—C12—C13	0.7 (7)
C3—C4—C5—C6	−0.6 (7)	C11—C12—C13—C14	−0.4 (7)
C4—C5—C6—C7	0.6 (7)	C12—C13—C14—C9	0.5 (7)
C3—C2—C7—C6	−0.1 (6)	N1—C9—C14—C13	177.7 (4)
C1—C2—C7—C6	178.6 (4)	C10—C9—C14—C13	−0.8 (5)
C3—C2—C7—C8	−177.8 (3)	C10—C9—N1—C8	174.3 (3)
C1—C2—C7—C8	0.9 (4)	C14—C9—N1—C8	−4.1 (5)
C5—C6—C7—C2	−0.3 (6)	O2—C8—N1—C9	73.7 (4)
C5—C6—C7—C8	176.9 (4)	C7—C8—N1—C9	−170.1 (3)

C2—C7—C8—N1	−122.0 (3)	O1—C1—O2—C8	179.5 (4)
C6—C7—C8—N1	60.6 (6)	C2—C1—O2—C8	0.9 (4)
C2—C7—C8—O2	−0.4 (4)	N1—C8—O2—C1	122.4 (3)
C6—C7—C8—O2	−177.8 (4)	C7—C8—O2—C1	−0.3 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^i	0.86	2.49	3.130 (5)	132
C4—H4···O2^{ii}	0.93	2.54	3.394 (5)	153
C11—H11···Br1^{iii}	0.93	2.99	3.845 (4)	154
N1—H1···Br1	0.86	2.60	3.058 (3)	114

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

Fig. 1

Fig. 2

Fig. 3

Fig. 4