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

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.100
Data-to-parameter ratio = 13.6

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

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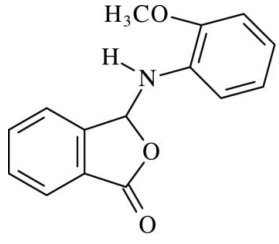
3-Substituted phthalides, Part III.

The title compound, $C_{15}H_{13}NO_3$, contains an intramolecular N—H···O hydrogen bond and the packing is stabilized by N—H···O and C—H···O intermolecular hydrogen bonds. The N—H···O hydrogen bonds generate $S(5)[R_2^2(12)]S(5)$ motifs and these motifs are bonded to each other by C—H···O intermolecular hydrogen bonds. The phthalide group is planar, showing a dihedral angle of 63.26 (8)° with the benzene ring.

Comment

Phthalides are known to show diverse biological activities as hormones, pheromones and antibiotics. As part of our ongoing research on 3-substituted phthalides, the title compound, (I), has been synthesized and its crystal structure is reported here.



(I)

The overall view and labeling of the molecule are displayed in Fig. 1. Selected bond lengths and angles are presented in Table 1, hydrogen bonding parameters are given in Table 2 and the packing arrangement of the molecules within the crystal structure is shown in Fig. 2. The phthalide part (C1 through C8) of the molecule is essentially planar, the largest deviation from the mean plane being 0.010 (2) Å for atom C1. The dihedral angle between the five-membered ring and the fused six-membered ring is 1.29 (8)°, whereas the dihedral angle between the mean plane of the phthalide system and benzene ring is 63.26 (8)°. The geometry of the molecule does not show any significant differences from the average geometry found for 3-(2-hydroxyanilino)isobenzofuran-1(3*H*)-one (Odabaşoğlu & Büyükgüngör, 2006*a*).

Two enantiomeric molecules of (I) are linked by N—H···O hydrogen bonds. These N—H···O hydrogen bonds generate $R_2^2(12)$ rings (Etter, 1990), which also have two edge-fused $S(5)$ rings, resulting in an $S(5)[R_2^2(12)]S(5)$ motif (Fig. 2 and Table 2).

Experimental

The title compound was prepared as described by Odabaşoğlu & Büyükgüngör (2006*b*), using phthalaldehydic acid and *o*-methoxy-

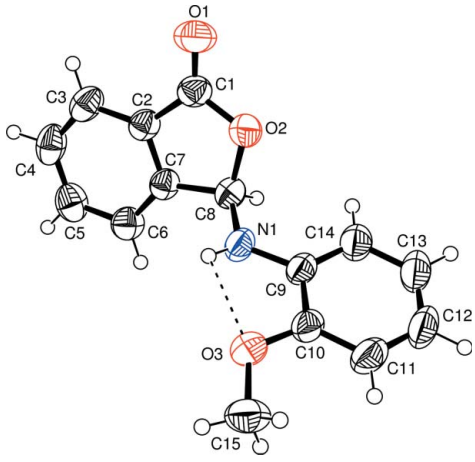


Figure 1
A view of (I), with the atomic numbering scheme; the intramolecular hydrogen bond is shown as a dashed line. Displacement ellipsoids are drawn at the 50% probability level.

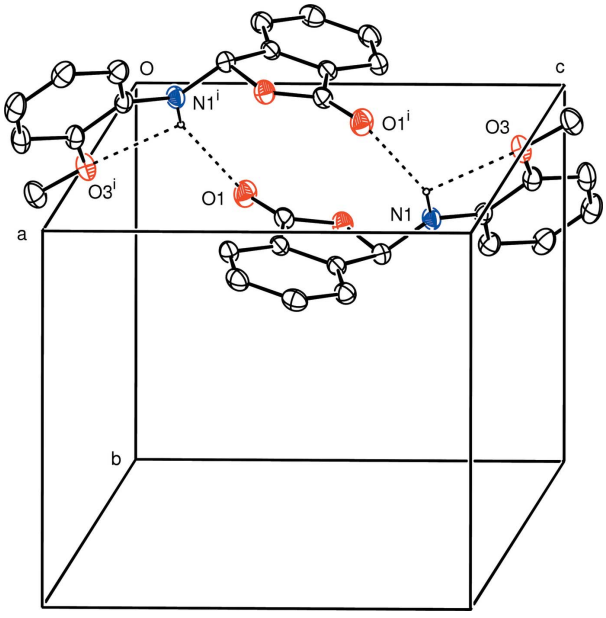


Figure 2
A view showing the formation of a dimer through N—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $1-x, -y, 1-z$]

aniline as starting materials (yield 95%; m.p. 427–428 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol (95%) solution at room temperature.

Crystal data

$C_{15}H_{13}NO_3$
$M_r = 255.26$
Triclinic, $P\overline{1}$
$a = 8.1259\ (10)$ Å
$b = 8.2644\ (11)$ Å
$c = 10.6641\ (13)$ Å
$\alpha = 87.136\ (11)^\circ$
$\beta = 78.290\ (10)^\circ$
$\gamma = 61.155\ (9)^\circ$
$V = 613.28\ (15)$ Å^3
$Z = 2$
$D_x = 1.382$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 296\ (2)$ K
Prism, brown
0.50 × 0.32 × 0.21 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.970$, $T_{max} = 0.986$
8287 measured reflections
2399 independent reflections
1774 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 26.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.100$
$S = 1.03$
2399 reflections
176 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.0674P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.2061 (19)	C8—N1	1.3913 (19)
C1—O2	1.3502 (19)	C8—O2	1.4961 (18)
C2—C7	1.376 (2)	C9—N1	1.3976 (19)
C7—C8	1.498 (2)		
O1—C1—O2	121.90 (15)	N1—C8—O2	112.46 (12)
O1—C1—C2	129.47 (15)	N1—C8—C7	114.88 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86 (2)	2.286 (18)	2.6193 (18)	103.3 (16)
N1—H1···O1^{i}	0.857 (18)	2.441 (18)	3.224 (2)	152.1 (15)
C15—H15B···O1^{ii}	0.96	2.54	3.387 (2)	147

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

All C-bound H atoms were refined using the riding-model approximation, with C—H = 0.93 Å for aromatic, 0.98 Å for methine and 0.96 Å for methyl H atoms [U_{iso}(H) = 1.2U_{eq}(parent atom)]. The N-bound H atom was located in a Fourier difference map and refined freely.

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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