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5-Amino-3*H*-isobenzofuran-1-one (5-aminophthalide)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.067
Data-to-parameter ratio = 7.3

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

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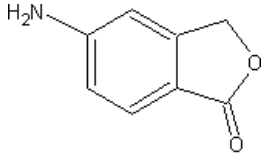
The title compound, $C_8H_7NO_2$, serves as an intermediate for the synthesis of citalopram. The packing of the planar molecules is stabilized by N—H···O and N—H···N hydrogen bonds.

Comment

Phthalide is a versatile synthetic building block, particularly for the synthesis of carbocyclic and heterocyclic compounds (Bradley *et al.*, 1997). The title compound, (I), is an intermediate for the synthesis of citalopram, which is a versatile antidepressant (Liechti *et al.*, 2000). A perspective view is shown in Fig. 1.



(I)

Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002). They agree with the values determined for *o*-phthalaldehyde (Majeed *et al.*, 1998; Mendenhall *et al.*, 2003), 6-nitrophthalide (Bradley *et al.*, 1997), 3-hydroxyphthalide (Khoo & Hazell, 1999) and 5-bromophthalide (Yathirajan *et al.*, 2005). All non-H atoms are coplanar (r.m.s. deviation = 0.017 Å). The crystal packing (Fig. 2) is stabilized by N—H···O and N—H···N hydrogen bonds. The NH vector of the donor group is almost perpendicular (85.8°) to the plane formed by the acceptor NH_2 group and the adjacent C atom.

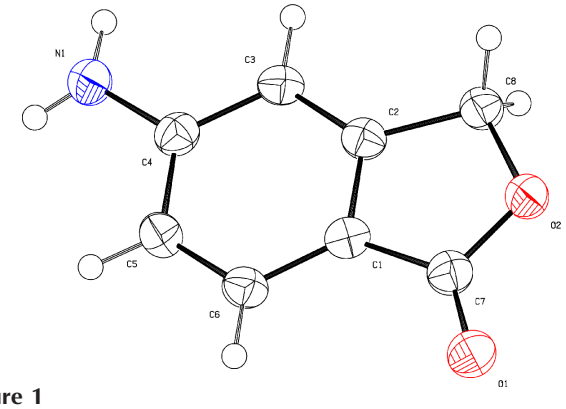


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are shown at the 50% probability level.

Experimental

5-Aminoisoindole-1,3-dione (1 g, 6.17 mmol) was heated at 353 K with zinc dust (1 g, 15.38 mmol) in 30% NaOH solution (10 ml) for 4 h. The residue was filtered off, the filtrate was acidified with concentrated HCl (20 ml) and the mass was heated at 353 K for 2 h. It was then cooled; the pH was adjusted to neutral using liquid NH_3, and the resulting solid was filtered off and recrystallized from acetonitrile (m.p. 463–466 K).

Crystal data

$C_8H_7NO_2$
M_r = 149.15
Orthorhombic, $P2_12_12_1$
a = 4.6858 (7) Å
b = 8.2573 (9) Å
c = 17.627 (2) Å
V = 682.02 (15) Å^3
Z = 4
D_x = 1.453 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 413 reflections
θ = 4.0–25.7°
μ = 0.11 mm^{-1}
T = 173 (2) K
Block, light brown
0.42 × 0.38 × 0.36 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: none
8516 measured reflections
789 independent reflections
676 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.7°
$h = -5 \rightarrow 5$
$k = -10 \rightarrow 9$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.067
S = 1.04
789 reflections
108 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.96 (2)	2.03 (3)	2.953 (2)	161 (2)
N1—H1B···N1^{ii}	0.82 (2)	2.40 (3)	3.200 (2)	166 (2)

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

H atoms were located in a difference map. Those bonded to carbon were positioned geometrically and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using a riding model, with C—H = 0.99 and 0.95 Å for methylene and aromatic CH groups, respectively. H atoms bonded to nitrogen were refined isotropically. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

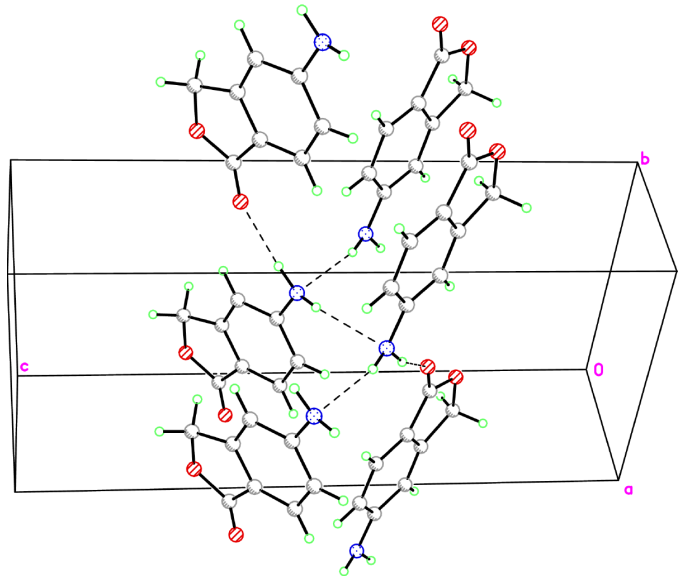


Figure 2
Packing diagram of the title compound; hydrogen bonds are shown as dashed lines.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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