

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

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Key indicators

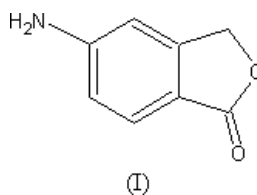
Single-crystal X-ray study
T = 173 K
Mean σ (C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.067
Data-to-parameter ratio = 7.3

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

The title compound, C₈H₇NO₂, 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.



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 NH₂ group and the adjacent C atom.

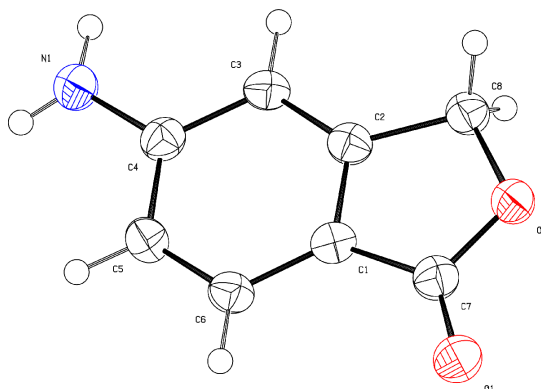


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are shown at the 50% probability level.

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 NH₃, and the resulting solid was filtered off and recrystallized from acetonitrile (m.p. 463–466 K).

Crystal data

C₈H₇NO₂
M_r = 149.15
 Orthorhombic, *P*2₁2₁2₁
a = 4.6858 (7) Å
b = 8.2573 (9) Å
c = 17.627 (2) Å
V = 682.02 (15) Å³
Z = 4
D_x = 1.453 Mg m⁻³

Mo *K*α 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* > 2σ(*I*)
*R*_{int} = 0.050
 θ_{\max} = 25.7°
h = -5 → 5
k = -10 → 9
l = -21 → 21

Refinement

Refinement on *F*²
R [*F*² > 2σ(*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 $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 $\Delta\rho_{\max} = 0.12 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.13 \text{ e } \text{Å}^{-3}$

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
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) = 1.2*U*_{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.

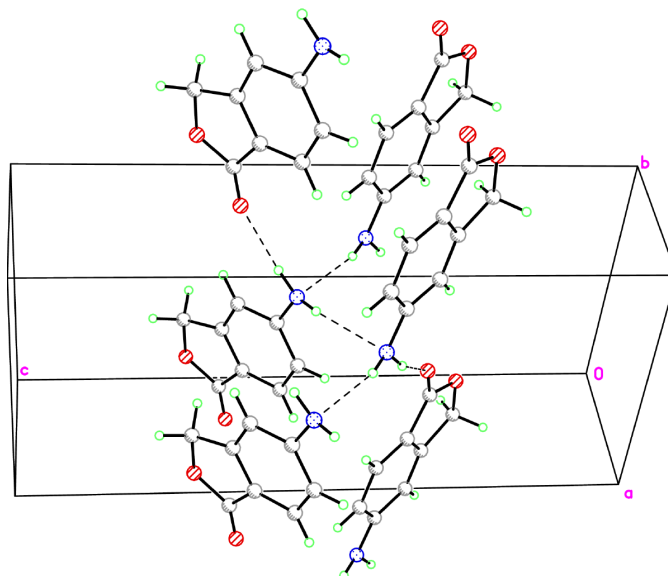


Figure 2

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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