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6-Amino-3*H*-isobenzofuran-1-one

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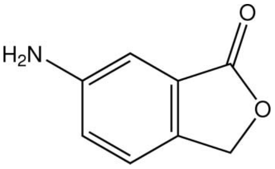
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.105; data-to-parameter ratio = 13.5.

The molecule of the title compound, $C_8H_7NO_2$, also known as 6-aminophthalide, is essentially planar. In the crystal structure, the molecules are connected into supramolecular double chains *via* N—H⋯O hydrogen bonds that participate in eight-membered (⋯HNH⋯O)$_2$ synthons.

Related literature

For related literature, see: Yathirajan *et al.* (2005).



Experimental

Crystal data

$C_8H_7NO_2$	V = 695.5 (4) Å^3
M_r = 149.15	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 8.396 (2) Å	μ = 0.10 mm^{-1}
b = 5.990 (2) Å	T = 153 (2) K
c = 14.012 (4) Å	0.40 × 0.20 × 0.10 mm
β = 99.242 (7)°	

Data collection

Rigaku AFC 12κ/SATURN 724 diffractometer	1436 independent reflections
Absorption correction: none	1370 reflections with $I > 2\sigma(I)$
4579 measured reflections	R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042	2 restraints
$wR(F^2)$ = 0.105	H-atom parameters constrained
S = 1.13	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
1436 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
106 parameters	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯O1^{i}	0.88	2.17	2.9937 (19)	156
N1—H2N⋯O1^{ii}	0.88	2.27	3.134 (2)	167
C2—H2A⋯O1^{iii}	0.99	2.51	3.477 (2)	164

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z+2$; (iii) $x, y-1, z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2488).

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supplementary materials

Acta Cryst. (2007). E**63**, o3634 [**doi:10.1107/S1600536807035763**]

6-Amino-3*H*-isobenzofuran-1-one

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Comment

The title compound (I), Fig. 1, was synthesized as a precursor for the corresponding polyamide. The maximum deviation from the least-squares plane through the 11 non-hydrogen atoms in (I) is 0.020 (1) Å, for the C5 atom. The geometric parameters in (I) match closely those reported for the isomeric 5-aminophthalide compound (Yathirajan *et al.*, 2005). The primary intermolecular interactions in the crystal structure of (I) are of the type N—H···O (Table 1). The presence of eight-membered $\{\cdots HNH\cdots O\}_2$ synthons leads to the formation of supramolecular double-chains aligned along the a direction (Fig. 2). This arrangement brings π-systems in close proximity and allows for the formation of π···π interactions that provide additional stability to the chains. The closest π···π contact of 3.7360 (15) Å occurs between *Cg*(O2, C1—C3, C8) and *Cg*(C3—C8) for symmetry operation: 1 − *x*, −*y*, 2 − *z*. The chains are linked by C—H···O interactions (Table 1) to form layers that stack along the c direction. Contacts between layers are of the type C—H···π so that C4—H4···*Cg*(C3—C8) = 2.74 Å, C4···*Cg*(C3—C8) = 3.4663 (18) Å with an angle at H = 134° for symmetry operation: 1/2 − *x*, −1/2 + *y*, 3/2 − *z*.

Experimental

The reagents were purchased as indicated and used without further purification: phthalide (98%, Alfa Aesar), *N*-bromosuccinimide (NBS) (99%, Alfa Aesar), 2,2'-Azobis(2-methylpropionitrile) (AIBN) (98%, Aldrich), benzene (99%, EM Science), sulfuric acid (98%, Mallinckrodt), nitric acid (70%, EMD), acetic acid (99.7%, Mallinckrodt), benzylamine (99%, Aldrich), dichloroethane (Aldrich), ethyl acetate (EMD), hexane (EMD), and methanol (Fisher).

The title compound (I) was obtained from two step nitration-reduction sequence. To a 250 ml round bottom flask, fitted with magnetic stirbar and an ice/water cooling bath, phthalide (21.74 g, 162 mmol) was added. An ice-cold mixture of 70% nitric acid and concentrated sulfuric acid were added at such a rate that the temperature remained below 20°C. The reaction mixture was allowed to stand overnight at room temperature. The clear yellow solution was poured into ice-cold water (300 ml). The yellow precipitate was collected and recrystallized from glacial acetic acid to give 6-nitrophthalide (81%). In a 250 round bottom flask equipped with a stirbar, 6-nitrophthalide (4.90 g, 27.4 mmol) was dissolved in ethanol (100 ml) with heating. At the same time, ammonium chloride (14.78 g, 0.276 mol) was completely dissolved in water (40 ml). To an ethanol solution of iron powder (9.26 g, 0.166 mol) was added the 6-nitrophthalide solution, followed immediately by the ammonium chloride solution. This resulted in an exotherm and the reaction turned brown immediately. The reaction mixture was heated under reflux overnight, and cooled to room temperature. Ethyl acetate was then used to extract the product, and the organic extracts were then washed with sodium bicarbonate and water give a white powder as the product (65%). Light-yellow crystals of (I) were obtained by recrystallization from a methanol solution.

TLC R_f = 0.38 (50/50 hexane/ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ (p.p.m.) 5.23 (s, 2H, α-CH–), 6.99 (dd, 1H, aromatic 7-H, J_1 = 1.96 Hz, J_2 = 8.30 Hz), 7.14 (d, 1H, aromatic 5-H, J = 2.44 Hz), 7.25 (d, 1H, aromatic 4-H, J = 8.30 Hz). IR (cm^{-1}) 3500 (–NH_2), 3300 (–NH_2), 1727 (–COO), 1630, 1615, 1505, 1491, 1465, 1329, 1187, 1056, 989.

Refinement

All the H atoms were included in the riding-model approximation, with C—H = 0.95–0.99 Å, N—H = 0.88 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) and 1.5$U_{\rm eq}$(N).

Figures

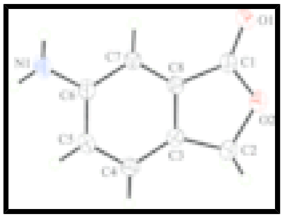

Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level (arbitrary spheres for the H atoms).

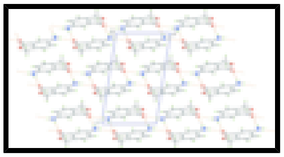

Fig. 2. View of the crystal packing in (I) highlighting the supramolecular double-chains mediated by hydrogen bonds, shown as orange-dashed lines. Colour code: red (oxygen), blue (nitrogen), grey (carbon) and green (hydrogen).

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Crystal data

$C_8H_7NO_2$

M_r = 149.15

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

a = 8.396 (2) Å

b = 5.990 (2) Å

c = 14.012 (4) Å

β = 99.242 (7)°

V = 695.5 (4) Å^3

Z = 4

F_{000} = 312

D_x = 1.424 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71070 Å

Cell parameters from 1986 reflections

θ = 2.7–30.2°

μ = 0.10 mm^{-1}

T = 153 (2) K

Prism, light yellow

0.40 × 0.20 × 0.10 mm

Data collection

Rigaku AFC12κ/SATURN724 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 153(2) K

ω scans

Absorption correction: none

4579 measured reflections

1436 independent reflections

1370 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.020

$\theta_{\rm max}$ = 26.5°

$\theta_{\rm min}$ = 2.7°

$h = -10 \rightarrow 10$

$k = -7 \rightarrow 6$

$l = -14 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.2023P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{max} < 0.001$
1436 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
106 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
O1	0.74553 (11)	0.30158 (17)	0.91253 (7)	0.0408 (3)
O2	0.71640 (11)	−0.03455 (17)	0.84229 (7)	0.0393 (3)
N1	0.10635 (15)	0.3245 (3)	0.94360 (11)	0.0529 (4)
H1N	0.0067	0.2770	0.9418	0.079*
H2N	0.1367	0.4437	0.9785	0.079*
C1	0.65841 (15)	0.1465 (2)	0.88321 (9)	0.0321 (3)
C2	0.58839 (16)	−0.1970 (2)	0.81381 (11)	0.0386 (3)
H2A	0.6135	−0.3405	0.8479	0.046*
H2B	0.5726	−0.2240	0.7432	0.046*
C3	0.44184 (15)	−0.0911 (2)	0.84286 (9)	0.0301 (3)
C4	0.28242 (16)	−0.1620 (2)	0.83663 (9)	0.0330 (3)
H4	0.2494	−0.3028	0.8091	0.040*
C5	0.17340 (15)	−0.0233 (2)	0.87133 (9)	0.0337 (3)
H5	0.0648	−0.0714	0.8677	0.040*
C6	0.21809 (15)	0.1874 (2)	0.91198 (9)	0.0339 (3)
C7	0.37826 (15)	0.2566 (2)	0.91822 (9)	0.0318 (3)
H7	0.4125	0.3972	0.9455	0.038*

C8	0.48546 (14)	0.1144 (2)	0.88350 (9)	0.0285 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0281 (5)	0.0427 (6)	0.0506 (6)	−0.0048 (4)	0.0037 (4)	−0.0033 (5)
O2	0.0287 (5)	0.0412 (6)	0.0494 (6)	0.0035 (4)	0.0102 (4)	−0.0034 (4)
N1	0.0270 (6)	0.0604 (9)	0.0721 (9)	0.0014 (6)	0.0100 (6)	−0.0306 (7)
C1	0.0268 (6)	0.0359 (7)	0.0334 (7)	0.0028 (5)	0.0044 (5)	0.0026 (5)
C2	0.0338 (7)	0.0351 (7)	0.0479 (8)	0.0029 (5)	0.0094 (6)	−0.0051 (6)
C3	0.0305 (7)	0.0299 (6)	0.0301 (6)	0.0021 (5)	0.0055 (5)	0.0010 (5)
C4	0.0340 (7)	0.0307 (6)	0.0339 (7)	−0.0030 (5)	0.0037 (5)	−0.0018 (5)
C5	0.0264 (6)	0.0399 (7)	0.0347 (7)	−0.0031 (5)	0.0049 (5)	−0.0005 (5)
C6	0.0275 (6)	0.0406 (7)	0.0331 (7)	0.0032 (5)	0.0035 (5)	−0.0053 (5)
C7	0.0274 (6)	0.0339 (7)	0.0330 (7)	0.0012 (5)	0.0018 (5)	−0.0062 (5)
C8	0.0259 (6)	0.0315 (6)	0.0276 (6)	0.0006 (5)	0.0024 (5)	0.0018 (5)

Geometric parameters (Å, °)

O1—C1	1.2123 (16)	C3—C8	1.3808 (18)
O2—C1	1.3535 (17)	C3—C4	1.3937 (18)
O2—C2	1.4570 (17)	C4—C5	1.3810 (19)
N1—C6	1.3726 (18)	C4—H4	0.9500
N1—H1N	0.8801	C5—C6	1.4104 (19)
N1—H2N	0.8801	C5—H5	0.9500
C1—C8	1.4654 (17)	C6—C7	1.3965 (18)
C2—C3	1.4976 (18)	C7—C8	1.3830 (17)
C2—H2A	0.9900	C7—H7	0.9500
C2—H2B	0.9900		
C1—O2—C2	110.57 (10)	C5—C4—C3	118.60 (12)
C6—N1—H1N	119.5	C5—C4—H4	120.7
C6—N1—H2N	120.9	C3—C4—H4	120.7
H1N—N1—H2N	118	C4—C5—C6	122.15 (12)
O1—C1—O2	121.26 (12)	C4—C5—H5	118.9
O1—C1—C8	130.08 (12)	C6—C5—H5	118.9
O2—C1—C8	108.67 (11)	N1—C6—C7	120.22 (13)
O2—C2—C3	104.17 (11)	N1—C6—C5	120.95 (12)
O2—C2—H2A	110.9	C7—C6—C5	118.82 (12)
C3—C2—H2A	110.9	C8—C7—C6	118.02 (12)
O2—C2—H2B	110.9	C8—C7—H7	121.0
C3—C2—H2B	110.9	C6—C7—H7	121.0
H2A—C2—H2B	108.9	C3—C8—C7	123.33 (12)
C8—C3—C4	119.09 (12)	C3—C8—C1	108.25 (11)
C8—C3—C2	108.34 (11)	C7—C8—C1	128.42 (12)
C4—C3—C2	132.58 (13)		
C2—O2—C1—O1	179.64 (12)	C5—C6—C7—C8	0.33 (19)
C2—O2—C1—C8	−0.42 (14)	C4—C3—C8—C7	−0.49 (19)
C1—O2—C2—C3	0.01 (14)	C2—C3—C8—C7	−179.98 (12)

O2—C2—C3—C8	0.42 (14)	C4—C3—C8—C1	178.81 (11)
O2—C2—C3—C4	−178.97 (13)	C2—C3—C8—C1	−0.68 (14)
C8—C3—C4—C5	0.13 (19)	C6—C7—C8—C3	0.26 (19)
C2—C3—C4—C5	179.47 (13)	C6—C7—C8—C1	−178.90 (12)
C3—C4—C5—C6	0.5 (2)	O1—C1—C8—C3	−179.37 (13)
C4—C5—C6—N1	178.26 (14)	O2—C1—C8—C3	0.70 (14)
C4—C5—C6—C7	−0.7 (2)	O1—C1—C8—C7	−0.1 (2)
N1—C6—C7—C8	−178.63 (13)	O2—C1—C8—C7	179.96 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1^{i}	0.88	2.17	2.9937 (19)	156
N1—H2N···O1^{ii}	0.88	2.27	3.134 (2)	167
C2—H2A···O1^{iii}	0.99	2.51	3.477 (2)	164

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z+2$; (iii) $x, y-1, z$.

Fig. 1

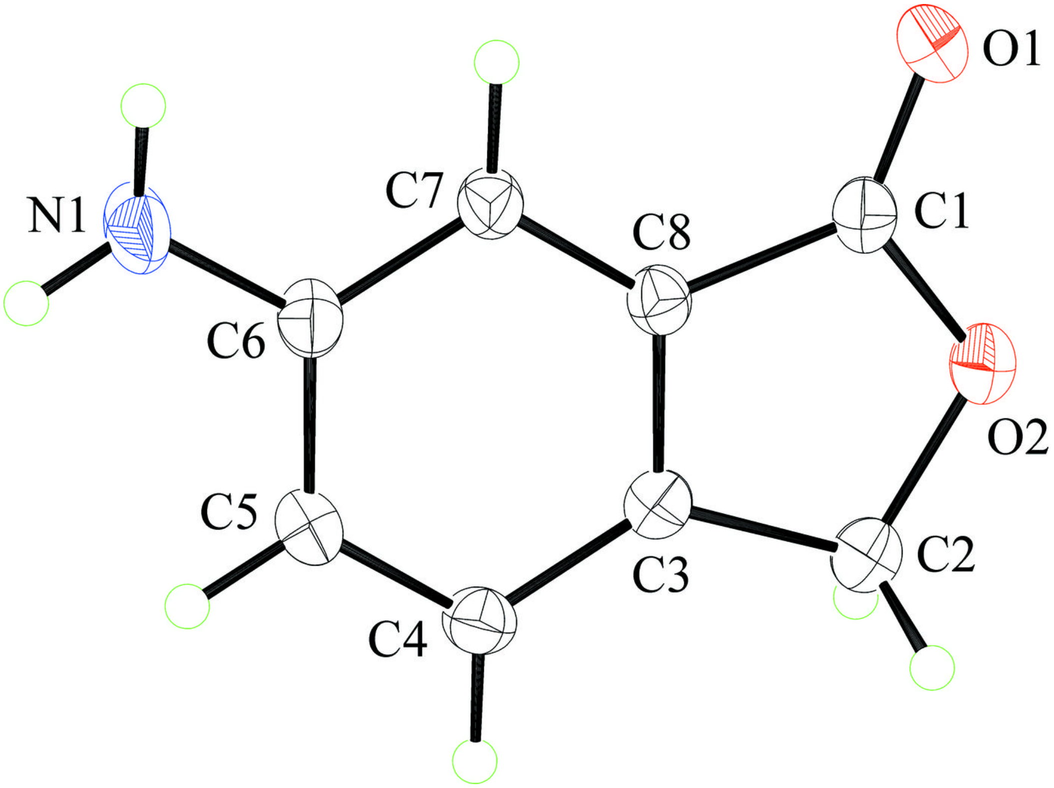

Fig. 2