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3-[(3-Oxo-1,3-dihydroisobenzofuran-1-yl)amino]benzoic acid

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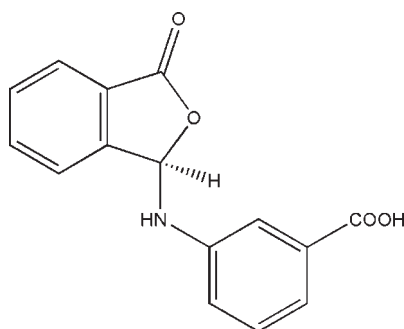
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 12.0.

In the title compound, $\text{C}_{15}\text{H}_{11}\text{NO}_4$, the dihedral angle formed by the benzene ring and isobenzofuran ring system is $67.82(5)^\circ$. The crystal structure is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.

Related literature

For general background to isobenzofuran derivatives, see: Landge *et al.* (2008); Paradkar *et al.* (1998); Joseph (1998). Odabaşoğlu & Büyükgüngör (2008).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{11}\text{NO}_4$
 $M_r = 269.25$

 Monoclinic, $P2_1/n$
 $a = 10.9025(15)$ Å

 $b = 8.1595(12)$ Å
 $c = 14.2654(18)$ Å
 $\beta = 103.463(1)^\circ$
 $V = 1234.2(3)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298$ K
 $0.27 \times 0.19 \times 0.17$ mm

Data collection

 Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.972$, $T_{\max} = 0.982$

 6011 measured reflections
 2171 independent reflections
 1206 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.107$
 $S = 0.90$
 2171 reflections

 181 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³
Table 1
 Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O4}^i$	0.82	1.91	2.712 (2)	166
$\text{N1}-\text{H1}\cdots\text{O2}^{ii}$	0.86	2.16	2.956 (2)	154

 Symmetry codes: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{5}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2162).

References

- Joseph, A. R. (1998). *J. Chem. Res. (S)*, pp. 332–333.
 Landge, S. M., Berryman, M. & Törk, B. (2008). *Tetrahedron Lett.* **49**, 4505–4508.
 Odabaşoğlu, M. & Büyükgüngör, O. (2008). *Acta Cryst.* **E64**, o752–o753.
 Paradkar, M. V., Ranade, A. A., Kulkarni, M. S., Godbole, H. M. & Joseph, A. R. (1998). *J. Chem. Res. (S)*, pp. 332–333.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

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3-[(3-Oxo-1,3-dihydroisobenzofuran-1-yl)amino]benzoic acid

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Comment

Phthalides (isobenzofuran-1(3*H*)-ones) are well known for their interesting biological properties (Paradkar *et al.*, 1998; Joseph, 1998). In addition, 3-substituted phthalides are vital heterocyclic motifs in many bioactive compounds such as isocoumarins, anthraquinones, anthracyclines, and several alkaloids (Landge *et al.*, 2008). In view of this, various methods have been reported for their synthesis. Herein, the crystal structure of the title compound is presented.

The title compound, (I), (Fig. 1) is a chirality compound with a chiral center at C₉. The dihedral angle between the benzene ring and isobenzofuran ring system is 67.82 (5) Å indicating that the two ring systems are not coplanar. The crystal structure is stabilized by intermolecular O—H...O and N—H...O hydrogen-bonding interactions (Fig. 2, Table. 1).

Experimental

To a ethanol solution (30 ml) of 3-aminobenzoic acid (3.00 mmol) added 3.00 mmol 2-formylbenzoic acid. The mixture solution was stirred at 343 K for 2.5 h. Then, sodium ethoxide (6.6 mmol) was added to the reactor and stirring for 0.5 h. Bis(tributyltin)oxide (0.3 mmol) was then added to the reactor and the reaction mixture was stirred for 6 h. The resulting clear solution was evaporated under vacuum. The product was crystallized from a solution of dichloromethane/methanol (1:1) yielding the title compound unexpectedly. Anal. Calcd (%) for C₁₅H₁₁N₁O₄ (Mr = 269.25): C, 66.91; H, 4.12; N, 5.20; O, 23.77. Found (%): C, 66.87; H, 4.13; N, 5.21; O, 23.79.

Refinement

All the H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93 (Ar—H), 0.86 (N—H) and 0.82 (O—H) Å.

Figures

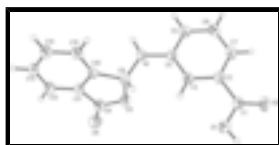


Fig. 1. The asymmetric unit of (I), showing 50% probability displacement ellipsoids.



Fig. 2. Crystal packing of (I) with hydrogen bonding as dashed lines.

3-[(3-Oxo-1,3-dihydroisobenzofuran-1-yl)amino]benzoic acid

Crystal data

$C_{15}H_{11}NO_4$	$F_{000} = 560$
$M_r = 269.25$	$D_x = 1.449 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yn	Cell parameters from 1143 reflections
$a = 10.9025 (15) \text{ \AA}$	$\theta = 2.7\text{--}21.4^\circ$
$b = 8.1595 (12) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 14.2654 (18) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 103.4630 (10)^\circ$	Block, colorless
$V = 1234.2 (3) \text{ \AA}^3$	$0.27 \times 0.19 \times 0.17 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	2171 independent reflections
Radiation source: fine-focus sealed tube	1206 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.038$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 12$
$T_{\text{min}} = 0.972$, $T_{\text{max}} = 0.982$	$k = -9 \rightarrow 9$
6011 measured reflections	$l = -16 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$
$S = 0.90$	where $P = (F_o^2 + 2F_c^2)/3$
2171 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
181 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.77236 (15)	0.2945 (2)	0.94157 (13)	0.0511 (5)

H1	0.7596	0.2454	0.8868	0.061*
O1	0.99795 (14)	0.5570 (2)	1.25378 (11)	0.0745 (6)
H1A	1.0238	0.6014	1.3062	0.112*
O2	1.19133 (13)	0.45300 (19)	1.28827 (10)	0.0592 (5)
O3	0.62455 (14)	0.2734 (2)	1.04292 (10)	0.0614 (5)
O4	0.44272 (16)	0.1671 (2)	1.06184 (12)	0.0788 (6)
C1	1.0886 (2)	0.4649 (3)	1.23355 (15)	0.0469 (6)
C2	1.05166 (19)	0.3785 (3)	1.14008 (14)	0.0407 (5)
C3	0.92786 (18)	0.3817 (3)	1.08532 (15)	0.0425 (5)
H3	0.8673	0.4427	1.1063	0.051*
C4	0.89469 (18)	0.2949 (3)	1.00014 (15)	0.0402 (6)
C5	0.9866 (2)	0.2051 (3)	0.96999 (16)	0.0494 (6)
H5	0.9656	0.1478	0.9121	0.059*
C6	1.1080 (2)	0.2000 (3)	1.02457 (17)	0.0522 (6)
H6	1.1682	0.1378	1.0039	0.063*
C7	1.1418 (2)	0.2860 (3)	1.10972 (17)	0.0491 (6)
H7	1.2243	0.2821	1.1465	0.059*
C8	0.5015 (2)	0.2389 (3)	1.01183 (17)	0.0555 (7)
C9	0.67070 (19)	0.3691 (3)	0.96714 (15)	0.0478 (6)
H9	0.6952	0.4794	0.9916	0.057*
C10	0.55418 (18)	0.3801 (3)	0.88699 (15)	0.0422 (6)
C11	0.45572 (18)	0.3033 (3)	0.91465 (15)	0.0436 (6)
C12	0.3365 (2)	0.2979 (3)	0.85393 (16)	0.0558 (7)
H12	0.2700	0.2468	0.8729	0.067*
C13	0.3193 (2)	0.3700 (3)	0.76518 (16)	0.0592 (7)
H13	0.2400	0.3681	0.7232	0.071*
C14	0.4181 (2)	0.4451 (3)	0.73746 (16)	0.0590 (7)
H14	0.4042	0.4926	0.6766	0.071*
C15	0.53674 (19)	0.4520 (3)	0.79711 (16)	0.0514 (6)
H15	0.6029	0.5032	0.7777	0.062*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0355 (11)	0.0706 (14)	0.0439 (11)	0.0046 (9)	0.0027 (8)	-0.0129 (10)
O1	0.0506 (10)	0.1082 (15)	0.0594 (11)	0.0126 (10)	0.0017 (8)	-0.0300 (10)
O2	0.0406 (9)	0.0749 (12)	0.0529 (10)	-0.0040 (8)	-0.0077 (7)	0.0057 (9)
O3	0.0450 (10)	0.0930 (13)	0.0429 (10)	0.0050 (9)	0.0033 (7)	0.0062 (9)
O4	0.0656 (12)	0.1171 (17)	0.0559 (11)	-0.0037 (10)	0.0183 (9)	0.0242 (11)
C1	0.0398 (13)	0.0544 (15)	0.0446 (14)	-0.0052 (12)	0.0062 (11)	0.0065 (12)
C2	0.0365 (12)	0.0426 (13)	0.0410 (13)	-0.0027 (10)	0.0050 (10)	0.0029 (11)
C3	0.0359 (12)	0.0450 (14)	0.0450 (13)	0.0024 (10)	0.0065 (10)	-0.0004 (11)
C4	0.0333 (12)	0.0438 (14)	0.0412 (13)	-0.0018 (10)	0.0043 (10)	0.0009 (11)
C5	0.0443 (14)	0.0548 (15)	0.0499 (15)	-0.0004 (12)	0.0125 (11)	-0.0056 (12)
C6	0.0403 (14)	0.0549 (16)	0.0630 (16)	0.0059 (11)	0.0155 (12)	-0.0003 (13)
C7	0.0329 (13)	0.0529 (15)	0.0589 (16)	0.0003 (11)	0.0057 (10)	0.0071 (13)
C8	0.0464 (15)	0.0747 (19)	0.0448 (15)	0.0040 (13)	0.0091 (12)	0.0034 (13)
C9	0.0400 (13)	0.0575 (15)	0.0439 (14)	0.0017 (11)	0.0058 (10)	-0.0028 (12)

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C10	0.0331 (12)	0.0499 (14)	0.0421 (13)	0.0039 (10)	0.0054 (10)	-0.0064 (11)
C11	0.0365 (13)	0.0559 (15)	0.0378 (13)	0.0021 (11)	0.0075 (10)	-0.0010 (11)
C12	0.0373 (13)	0.0786 (19)	0.0522 (15)	-0.0055 (12)	0.0120 (11)	-0.0035 (13)
C13	0.0375 (14)	0.0906 (19)	0.0448 (15)	0.0049 (13)	0.0002 (11)	-0.0022 (14)
C14	0.0477 (15)	0.0826 (19)	0.0454 (14)	0.0101 (13)	0.0082 (12)	0.0117 (14)
C15	0.0402 (13)	0.0630 (16)	0.0509 (15)	0.0002 (11)	0.0108 (11)	0.0046 (13)

Geometric parameters (Å, °)

N1—C9	1.386 (3)	C6—C7	1.377 (3)
N1—C4	1.399 (2)	C6—H6	0.9300
N1—H1	0.8600	C7—H7	0.9300
O1—C1	1.326 (2)	C8—C11	1.458 (3)
O1—H1A	0.8200	C9—C10	1.501 (3)
O2—C1	1.211 (2)	C9—H9	0.9800
O3—C8	1.341 (3)	C10—C11	1.378 (3)
O3—C9	1.512 (3)	C10—C15	1.382 (3)
O4—C8	1.215 (3)	C11—C12	1.384 (3)
C1—C2	1.479 (3)	C12—C13	1.369 (3)
C2—C7	1.386 (3)	C12—H12	0.9300
C2—C3	1.394 (3)	C13—C14	1.375 (3)
C3—C4	1.380 (3)	C13—H13	0.9300
C3—H3	0.9300	C14—C15	1.374 (3)
C4—C5	1.388 (3)	C14—H14	0.9300
C5—C6	1.371 (3)	C15—H15	0.9300
C5—H5	0.9300		
C9—N1—C4	123.46 (18)	O4—C8—C11	128.4 (2)
C9—N1—H1	118.3	O3—C8—C11	109.5 (2)
C4—N1—H1	118.3	N1—C9—C10	114.38 (18)
C1—O1—H1A	109.5	N1—C9—O3	112.41 (18)
C8—O3—C9	110.22 (17)	C10—C9—O3	102.25 (17)
O2—C1—O1	122.0 (2)	N1—C9—H9	109.2
O2—C1—C2	124.1 (2)	C10—C9—H9	109.2
O1—C1—C2	113.96 (17)	O3—C9—H9	109.2
C7—C2—C3	120.0 (2)	C11—C10—C15	120.71 (18)
C7—C2—C1	118.49 (19)	C11—C10—C9	109.41 (19)
C3—C2—C1	121.4 (2)	C15—C10—C9	129.9 (2)
C4—C3—C2	120.3 (2)	C10—C11—C12	121.0 (2)
C4—C3—H3	119.8	C10—C11—C8	108.61 (18)
C2—C3—H3	119.8	C12—C11—C8	130.3 (2)
C3—C4—C5	118.93 (18)	C13—C12—C11	118.2 (2)
C3—C4—N1	123.01 (19)	C13—C12—H12	120.9
C5—C4—N1	118.05 (19)	C11—C12—H12	120.9
C6—C5—C4	120.8 (2)	C12—C13—C14	120.6 (2)
C6—C5—H5	119.6	C12—C13—H13	119.7
C4—C5—H5	119.6	C14—C13—H13	119.7
C5—C6—C7	120.7 (2)	C15—C14—C13	121.9 (2)
C5—C6—H6	119.7	C15—C14—H14	119.1
C7—C6—H6	119.7	C13—C14—H14	119.1

C6—C7—C2	119.3 (2)	C14—C15—C10	117.6 (2)
C6—C7—H7	120.3	C14—C15—H15	121.2
C2—C7—H7	120.3	C10—C15—H15	121.2
O4—C8—O3	122.1 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1A \cdots O4 ⁱ	0.82	1.91	2.712 (2)	166
N1—H1 \cdots O2 ⁱⁱ	0.86	2.16	2.956 (2)	154

Symmetry codes: (i) $-x+3/2, y+1/2, -z+5/2$; (ii) $x-1/2, -y+1/2, z-1/2$.

Fig. 1

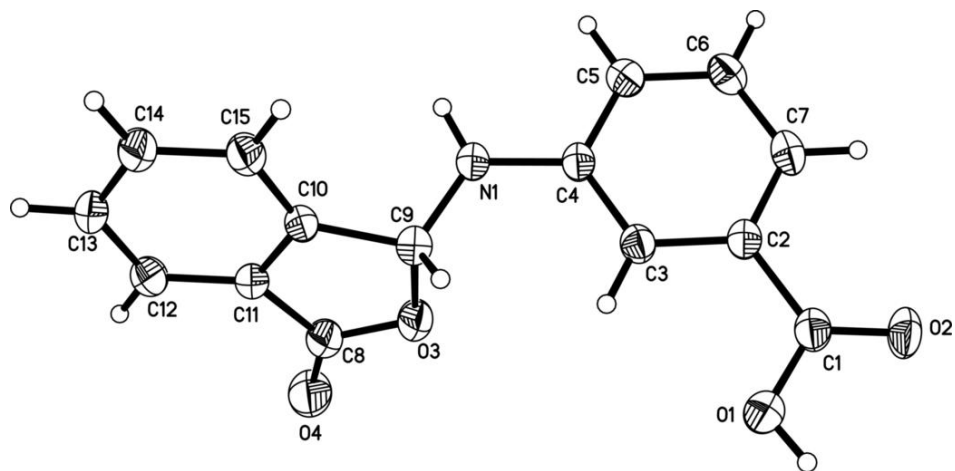


Fig. 2

