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1-Oxo-*N*-phenylisoindoline-2-carboxamide

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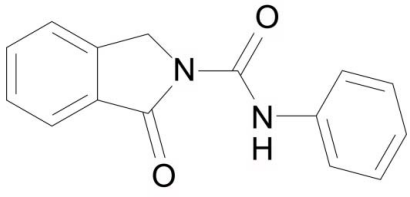
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.097; data-to-parameter ratio = 16.1.

The title molecule, $C_{15}H_{12}N_2O_2$, is essentially planar. The crystal structure is stabilized by an extensive hydrogen-bonded network. There are two intramolecular hydrogen bonds resulting in six-membered rings in graph-set patterns $S(6)$ and three intermolecular interactions. Two of these are C—H···O intermolecular hydrogen bonds that result in dimers, representing $R_2^2(10)$ graph-set patterns, and a C—H···N hydrogen bond leading to the formation of chains of molecules along the b axis.

Related literature

For related literature, see: Benati *et al.* (2003); Berger *et al.* (1999); Bernstein *et al.* (1994); Cignarella *et al.* (1981); Mancilla *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{12}N_2O_2$
M_r = 252.27
Monoclinic, $P2_1/c$
a = 12.905 (4) Å
b = 5.619 (1) Å
c = 17.161 (6) Å
β = 102.228 (17)°
V = 1216.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.35 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.968, T_{max} = 0.994
5034 measured reflections
2768 independent reflections
2119 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.097
S = 1.02
2768 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1^{i}	0.95	2.41	3.3105 (17)	159
C7—H7B···O2^{ii}	0.99	2.57	3.0746 (17)	112
C7—H7B···N2^{iii}	0.99	2.60	3.4695 (18)	147
C11—H11···O2	0.95	2.27	2.8833 (19)	122
N2—H2N···O1	0.88	1.95	2.6803 (15)	140

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2558).

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supplementary materials

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1-Oxo-*N*-phenylisoindoline-2-carboxamide

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Comment

Isoindole type compounds are important as intermediates for the synthesis of novel multidrugs resistance reversal agents (Berger *et al.*, 1999). They have also shown diuretic activity (Cignarella *et al.*, 1981). Several isoindoles have exhibited anti-inflammatory and analgesic activity (Mancilla *et al.*, 2007). In our efforts to isolate chromagens responsible for color development in the reaction of *N*-phenylurea with *ortho* phthaldehyde (OPTA), the title compound, (I), was prepared, the crystal structure of which is presented in this article.

The structure of (I) is composed of an essentially planar molecule comprised of an isoindole ring that is briged through a carboxamide group to a phenyl ring (Fig. 1). A mean-planes calculation through all non-hydrogen atoms revealed that C12 and N1 atoms lie 0.057 (1) and 0.052 (1) Å above and below, respectively, from the plane. The angle between mean-planes of the isoindole ring and the phenyl ring is 2.62 (8)°. The structure is stabilized by two intramolecular hydrogen bonds N2—H2N···O1 and C11—H11A···O2 that result in six membered rings which may be best defined in graph set patterns S(6) (Bernstein *et al.*, 1994); details of hydrogen bonding geometry have been provided in the Table. In addition, rather weak intermolecular interactions of two distinct types are present in the crystal (Fig. 2). The C—H···O type intermolecular H-bonding results in dimers forming ten-membered rings ···O1—C8—C1—C2—H2···O1^{i} and ···O2—C9—N1—C7—H7b···O2^{ii}, both representing $R_2^2(10)$ graph set patterns (Bernstein *et al.*, 1994). The C—H···N type H-bonding results in chains of molecules in a pattern ···N2—C9—N1—C7—H7B···N2^{iii} along the b-aixs. Symmetry codes as in the bydrogen bond geometry table.

The structure of 2-(bis(1-oxo-2,3-dihydroisoindol-2-yl)methyl)tetrahydrothiophene (Benati *et al.*, 2003) which is somewhat related to the title compound has been reported.

Experimental

A mixture of *o*-phthaldehyde (0.67 g, 200 mmol) and *N*-phenylurea (0.68 g, 200 mmol) in 100 ml of ethanol was refluxed for 12 hrs. The contents of flask were then left to stand for 48 hrs. at room temperature. The crystals of (I) thus formed were filtered, washed with hexane, ether and ethanol and were dried at room temperature. Crystals suitable for X-ray diffraction were grown from a solution of acetone by slow evaporation at room temperature.

Refinement

H-atoms were included in the refinements at geometrically idealized positions with C—$H_{aromatic}$ = 0.95, CH_2 = 0.99 and N—H = 0.88 Å and U_{iso} = 1.2 times U_{eq} of the atoms to which they were bonded. The final difference map was free of any chemically significant features.

Figures

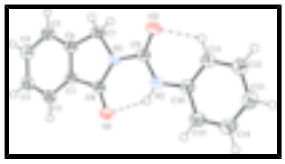

Fig. 1. The molecular structure with displacement ellipsoids plotted at 50% probability level; intermolecular interactions have been indicated by broken lines.

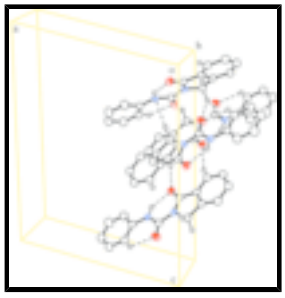

Fig. 2. H-bonding interactions in the unit cell of (I); H-atoms not involved in interactions have been ignored. H-bonds are shown as dashed lines.

1-Oxo-*N*-phenylisoindoline-2-carboxamide

Crystal data

$C_{15}H_{12}N_2O_2$	F_{000} = 528
M_r = 252.27	D_x = 1.378 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5034 reflections
a = 12.905 (4) Å	θ = 2.6–27.5º
b = 5.6190 (10) Å	μ = 0.09 mm^{-1}
c = 17.161 (6) Å	T = 173 (2) K
β = 102.228 (17)º	Needle, colorless
V = 1216.2 (6) Å^3	0.35 × 0.10 × 0.06 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	2768 independent reflections
Radiation source: fine-focus sealed tube	2119 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 173(2) K	θ_{max} = 27.5º
ω and φ scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	h = −16→16
T_{min} = 0.968, T_{max} = 0.994	k = −6→7
5034 measured reflections	l = −22→22

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.39P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
2768 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. IR spectra (nujol) showed vibrations 3425 cm^{-1} (N—H), 1714 and 1680 cm^{-1} (C=O), 1600 and 155.8 cm^{-1} (C=C), 1150 cm^{-1} (C—O). ^{1}H-NMR (DMSO-d$_6$); δ: 4.92 (s –CH$_2$–), 7.10–7.93 (aromatic ring protons, 9H), 10.72 (s, NH). ^{13}C-NMR (DMSO-d$_6$); δ: 48.59 (–CH$_2$–), 123.37 (C$_4$), 124.08 (C$_3$), 128.65 (C$_5$), 130.94 (C$_6$), 133.93 (C$_2$), 141.07 (C$_1$), 150.25 (C$_9$), 169.44 (C$_8$) and 120.06, 124.86, 129.03, 137.50 (N—Ph carbons). Anal. Calcd for $C_{15}H_{12}N_2O_2$. Required: C, 71.42; H, 4.79; N, 11.0; O, 12.68; Found: C, 71.40; H, 4.80; N, 11.07; O, 12.70. m.p. 441 K. m/*z*; 252.

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 > 2sigma(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	0.92562 (7)	−0.27832 (16)	0.39885 (5)	0.0328 (2)
O2	0.89390 (8)	0.31349 (17)	0.24855 (6)	0.0406 (3)
N1	0.97796 (8)	0.07840 (18)	0.34984 (6)	0.0263 (2)
N2	0.80821 (8)	−0.01677 (19)	0.28210 (6)	0.0297 (3)
H2N	0.8169	−0.1366	0.3159	0.036*
C1	1.09874 (10)	−0.1015 (2)	0.44905 (7)	0.0268 (3)
C2	1.14801 (11)	−0.2514 (2)	0.51004 (8)	0.0315 (3)
H2	1.1139	−0.3908	0.5234	0.038*
C3	1.24880 (11)	−0.1894 (3)	0.55062 (8)	0.0356 (3)
H3	1.2846	−0.2866	0.5932	0.043*
C4	1.29830 (11)	0.0143 (3)	0.52972 (8)	0.0380 (3)
H4	1.3676	0.0532	0.5584	0.046*

C5	1.24877 (11)	0.1619 (2)	0.46793 (8)	0.0346 (3)
H5	1.2834	0.2991	0.4535	0.041*
C6	1.14717 (10)	0.1021 (2)	0.42818 (7)	0.0281 (3)
C7	1.07353 (10)	0.2269 (2)	0.36116 (8)	0.0296 (3)
H7A	1.0587	0.3916	0.3761	0.036*
H7B	1.1030	0.2308	0.3124	0.036*
C8	0.99162 (10)	−0.1207 (2)	0.39860 (7)	0.0263 (3)
C9	0.89044 (10)	0.1374 (2)	0.28902 (7)	0.0284 (3)
C10	0.71058 (10)	−0.0067 (2)	0.22699 (8)	0.0301 (3)
C11	0.68394 (11)	0.1681 (3)	0.16870 (8)	0.0360 (3)
H11	0.7324	0.2927	0.1648	0.043*
C12	0.58577 (11)	0.1588 (3)	0.11620 (9)	0.0419 (4)
H12	0.5675	0.2784	0.0765	0.050*
C13	0.51455 (12)	−0.0206 (3)	0.12064 (9)	0.0452 (4)
H13	0.4481	−0.0259	0.0840	0.054*
C14	0.54067 (12)	−0.1927 (3)	0.17890 (10)	0.0480 (4)
H14	0.4916	−0.3158	0.1828	0.058*
C15	0.63785 (11)	−0.1869 (3)	0.23162 (9)	0.0401 (3)
H15	0.6552	−0.3067	0.2713	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0361 (5)	0.0257 (5)	0.0383 (5)	−0.0031 (4)	0.0116 (4)	0.0042 (4)
O2	0.0453 (6)	0.0332 (5)	0.0402 (6)	−0.0068 (4)	0.0016 (4)	0.0125 (4)
N1	0.0310 (6)	0.0215 (5)	0.0280 (5)	−0.0004 (4)	0.0093 (4)	0.0015 (4)
N2	0.0330 (6)	0.0276 (5)	0.0290 (6)	−0.0025 (4)	0.0080 (4)	0.0037 (4)
C1	0.0315 (6)	0.0226 (6)	0.0286 (6)	0.0032 (5)	0.0119 (5)	−0.0018 (5)
C2	0.0375 (7)	0.0267 (6)	0.0328 (7)	0.0051 (5)	0.0131 (6)	0.0026 (5)
C3	0.0383 (7)	0.0360 (7)	0.0333 (7)	0.0105 (6)	0.0094 (6)	0.0028 (6)
C4	0.0322 (7)	0.0410 (8)	0.0398 (8)	0.0035 (6)	0.0054 (6)	−0.0033 (6)
C5	0.0349 (7)	0.0296 (7)	0.0403 (8)	−0.0023 (6)	0.0105 (6)	−0.0016 (6)
C6	0.0330 (7)	0.0237 (6)	0.0296 (7)	0.0019 (5)	0.0113 (5)	−0.0023 (5)
C7	0.0336 (7)	0.0223 (6)	0.0336 (7)	−0.0033 (5)	0.0084 (5)	0.0006 (5)
C8	0.0340 (7)	0.0200 (6)	0.0281 (6)	0.0025 (5)	0.0139 (5)	−0.0004 (5)
C9	0.0347 (7)	0.0248 (6)	0.0272 (6)	0.0002 (5)	0.0097 (5)	−0.0012 (5)
C10	0.0314 (7)	0.0302 (6)	0.0302 (7)	0.0011 (5)	0.0100 (5)	−0.0036 (5)
C11	0.0335 (7)	0.0347 (7)	0.0406 (8)	−0.0003 (6)	0.0092 (6)	0.0050 (6)
C12	0.0356 (7)	0.0448 (8)	0.0444 (8)	0.0033 (6)	0.0067 (6)	0.0085 (7)
C13	0.0339 (8)	0.0493 (9)	0.0494 (9)	−0.0019 (7)	0.0024 (7)	0.0009 (7)
C14	0.0407 (8)	0.0425 (8)	0.0583 (10)	−0.0122 (7)	0.0048 (7)	0.0028 (7)
C15	0.0419 (8)	0.0341 (7)	0.0434 (8)	−0.0063 (6)	0.0070 (7)	0.0043 (6)

Geometric parameters (Å, °)

O1—C8	1.2293 (15)	C5—C6	1.3851 (19)
O2—C9	1.2148 (16)	C5—H5	0.9500
N1—C8	1.3859 (15)	C6—C7	1.5016 (18)
N1—C9	1.4057 (17)	C7—H7A	0.9900

N1—C7	1.4676 (16)	C7—H7B	0.9900
N2—C9	1.3556 (16)	C10—C11	1.3921 (19)
N2—C10	1.4073 (17)	C10—C15	1.3944 (19)
N2—H2N	0.8800	C11—C12	1.391 (2)
C1—C6	1.3865 (17)	C11—H11	0.9500
C1—C2	1.3883 (18)	C12—C13	1.377 (2)
C1—C8	1.4715 (18)	C12—H12	0.9500
C2—C3	1.384 (2)	C13—C14	1.380 (2)
C2—H2	0.9500	C13—H13	0.9500
C3—C4	1.394 (2)	C14—C15	1.383 (2)
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.391 (2)	C15—H15	0.9500
C4—H4	0.9500		
C8—N1—C9	128.19 (10)	N1—C7—H7B	111.3
C8—N1—C7	112.54 (10)	C6—C7—H7B	111.3
C9—N1—C7	118.99 (10)	H7A—C7—H7B	109.2
C9—N2—C10	127.29 (11)	O1—C8—N1	125.74 (12)
C9—N2—H2N	116.4	O1—C8—C1	128.03 (11)
C10—N2—H2N	116.4	N1—C8—C1	106.22 (10)
C6—C1—C2	122.40 (12)	O2—C9—N2	126.40 (12)
C6—C1—C8	109.11 (11)	O2—C9—N1	119.71 (12)
C2—C1—C8	128.47 (12)	N2—C9—N1	113.89 (11)
C3—C2—C1	117.27 (12)	C11—C10—C15	119.12 (13)
C3—C2—H2	121.4	C11—C10—N2	124.12 (12)
C1—C2—H2	121.4	C15—C10—N2	116.75 (12)
C2—C3—C4	120.69 (13)	C12—C11—C10	119.41 (13)
C2—C3—H3	119.7	C12—C11—H11	120.3
C4—C3—H3	119.7	C10—C11—H11	120.3
C5—C4—C3	121.61 (13)	C13—C12—C11	121.24 (14)
C5—C4—H4	119.2	C13—C12—H12	119.4
C3—C4—H4	119.2	C11—C12—H12	119.4
C6—C5—C4	117.71 (13)	C12—C13—C14	119.29 (14)
C6—C5—H5	121.1	C12—C13—H13	120.4
C4—C5—H5	121.1	C14—C13—H13	120.4
C5—C6—C1	120.30 (12)	C13—C14—C15	120.41 (14)
C5—C6—C7	130.05 (12)	C13—C14—H14	119.8
C1—C6—C7	109.66 (11)	C15—C14—H14	119.8
N1—C7—C6	102.34 (10)	C14—C15—C10	120.52 (14)
N1—C7—H7A	111.3	C14—C15—H15	119.7
C6—C7—H7A	111.3	C10—C15—H15	119.7
C6—C1—C2—C3	0.42 (18)	C2—C1—C8—O1	−2.6 (2)
C8—C1—C2—C3	−177.84 (12)	C6—C1—C8—N1	−1.61 (13)
C1—C2—C3—C4	−0.79 (19)	C2—C1—C8—N1	176.84 (12)
C2—C3—C4—C5	0.1 (2)	C10—N2—C9—O2	0.3 (2)
C3—C4—C5—C6	1.0 (2)	C10—N2—C9—N1	−179.59 (11)
C4—C5—C6—C1	−1.35 (19)	C8—N1—C9—O2	−175.71 (12)
C4—C5—C6—C7	178.64 (13)	C7—N1—C9—O2	−2.24 (17)
C2—C1—C6—C5	0.67 (19)	C8—N1—C9—N2	4.20 (17)

C8—C1—C6—C5	179.23 (11)	C7—N1—C9—N2	177.67 (10)
C2—C1—C6—C7	−179.32 (11)	C9—N2—C10—C11	0.9 (2)
C8—C1—C6—C7	−0.76 (13)	C9—N2—C10—C15	−179.93 (12)
C8—N1—C7—C6	−3.79 (13)	C15—C10—C11—C12	−0.3 (2)
C9—N1—C7—C6	−178.24 (10)	N2—C10—C11—C12	178.82 (13)
C5—C6—C7—N1	−177.34 (13)	C10—C11—C12—C13	−0.2 (2)
C1—C6—C7—N1	2.65 (13)	C11—C12—C13—C14	0.8 (2)
C9—N1—C8—O1	−3.3 (2)	C12—C13—C14—C15	−0.8 (3)
C7—N1—C8—O1	−177.10 (12)	C13—C14—C15—C10	0.3 (2)
C9—N1—C8—C1	177.28 (11)	C11—C10—C15—C14	0.2 (2)
C7—N1—C8—C1	3.46 (13)	N2—C10—C15—C14	−178.95 (13)
C6—C1—C8—O1	178.97 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{i}	0.95	2.41	3.3105 (17)	159
C7—H7B···O2^{ii}	0.99	2.57	3.0746 (17)	112
C7—H7B···N2^{iii}	0.99	2.60	3.4695 (18)	147
C11—H11···O2	0.95	2.27	2.8833 (19)	122
N2—H2N···O1	0.88	1.95	2.6803 (15)	140

Symmetry codes: (i) −*x*+2, −*y*−1, −*z*+1; (ii) −*x*+2, *y*−1/2, −*z*+1/2; (iii) −*x*+2, *y*+1/2, −*z*+1/2.

Fig. 1

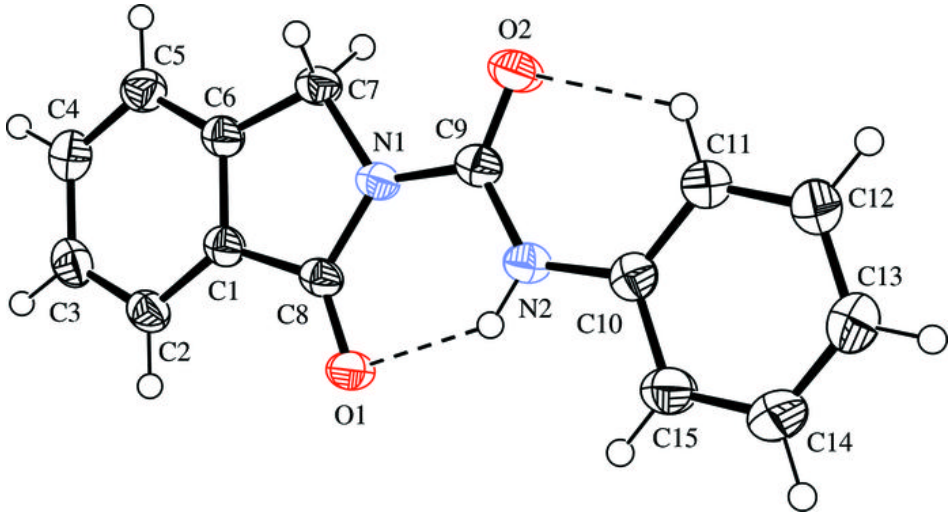

Fig. 2