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(*Z*)-3-Hydrazinylidene-1-phenylindolin-2-one

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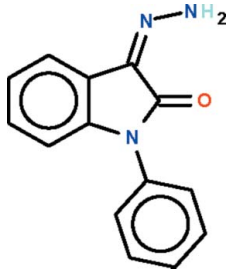
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.092; data-to-parameter ratio = 8.4.

The indoline fused-ring system of the title Schiff base, $C_{14}H_{11}N_3O$, is planar (r.m.s. deviation = 0.005 Å); the phenyl substituent is aligned at 66.5 (1)° with respect to the ring system. The amino $-NH_2$ unit forms an intramolecular hydrogen bond with the carbonyl O atom. Molecules are connected by an intermolecular N—H···N hydrogen bond, generating a zigzag chain that runs along the short c axis of the unit cell.

Related literature

For the synthesis of the title compound, see: de Diesbach & Heppner (1949).



Experimental

Crystal data

$C_{14}H_{11}N_3O$
M_r = 237.26
Orthorhombic, *Fdd*2
a = 19.328 (3) Å
b = 41.612 (5) Å
c = 5.6288 (7) Å
V = 4527 (1) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.35 × 0.04 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
10569 measured reflections
1430 independent reflections
1188 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.092
S = 1.03
1430 reflections
171 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: 1138 Friedel pairs were merged

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1···O1	0.88 (1)	2.06 (2)	2.772 (3)	137 (3)
N3—H2···N2^{i}	0.89 (1)	2.22 (1)	3.102 (3)	177 (3)

Symmetry code: (i) $-x+\frac{1}{2}, -y, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5393).

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supporting information

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(*Z*)-3-Hydrazinylidene-1-phenylindolin-2-one

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S1. Comment

Isatin derivatives such as phenylisatin have been studied in the context of its biological properties. We have synthesized the condensation product of phenylisatin with hydrazine for evaluation as a chemotherapeutic agent. The title hydrazone (Scheme I) is only mentioned once in the chemical literature (de Diesbach & Heppner, 1949). The indolinyl fused-ring is twisted with respect to the phenyl substituent by 66.5 (1)° (Fig. 1). The amino –NH2 unit forms an intramolecular hydrogen bond with the carbonyl O atom; the unit uses its other H atom for intermolecular hydrogen bonding to the two-coordinate N atom (Fig. 2). The intramolecular N—H···N interaction generates a zigzag chain that runs along the short *c* axis of the unit cell.

S2. Experimental

1-Phenylindoline-2,3-dione (0.220 g, 1 mmol) and hydrazine hydrate (0.055 g, 1.1 mmol) were dissolved in methanol (25 ml) and the solution heated for 1 h. The solvent was evaporated and the product recrystallized from ethanol to give yellow prismatic crystals; yield 70%.

S3. Refinement

Carbon-bound H atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 times U_{eq}(C).

The amino H atoms were located in a difference Fourier map, and were refined isotropically with a distance restraint of N—H 0.88 (1) Å.

As the structure has no anomalous scatterer, 1138 Friedel pairs were merged.

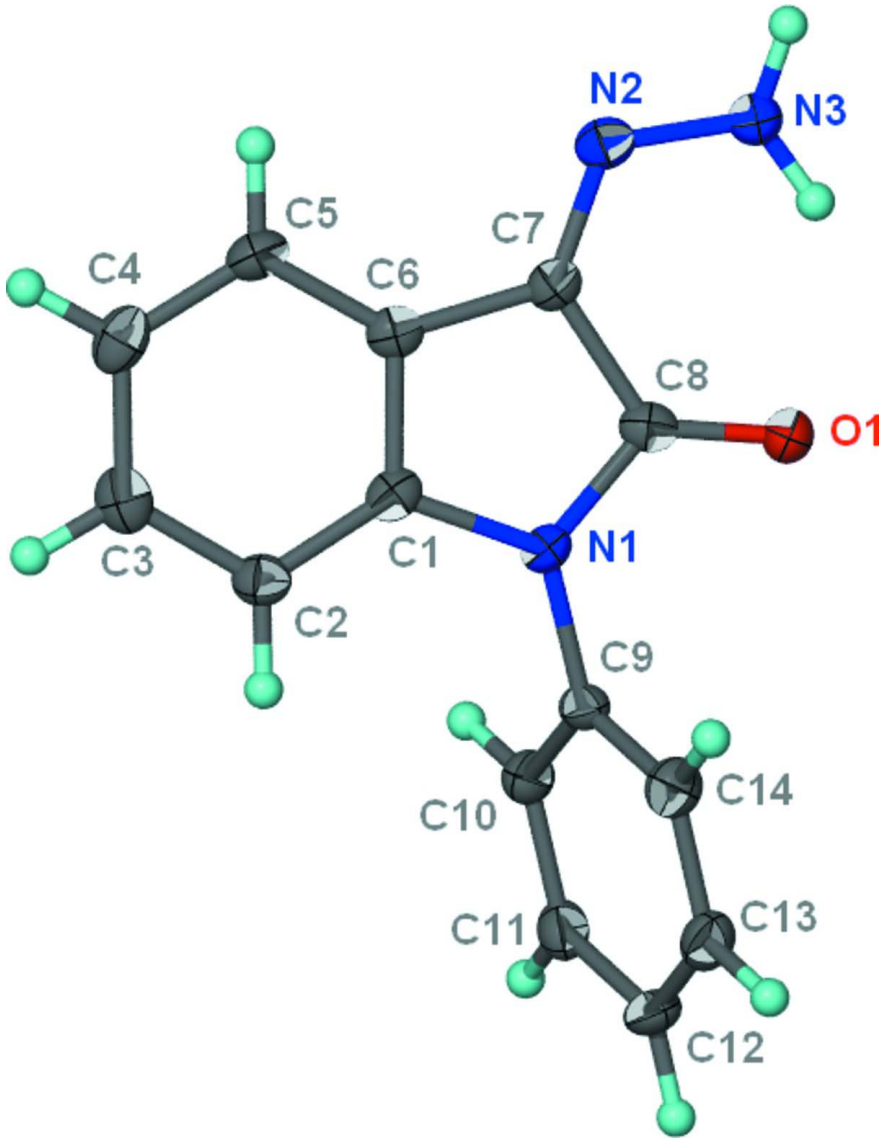

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{14}H_{11}N_3O$ at the 70% probability level; H atoms are drawn as spheres of arbitrary radius.

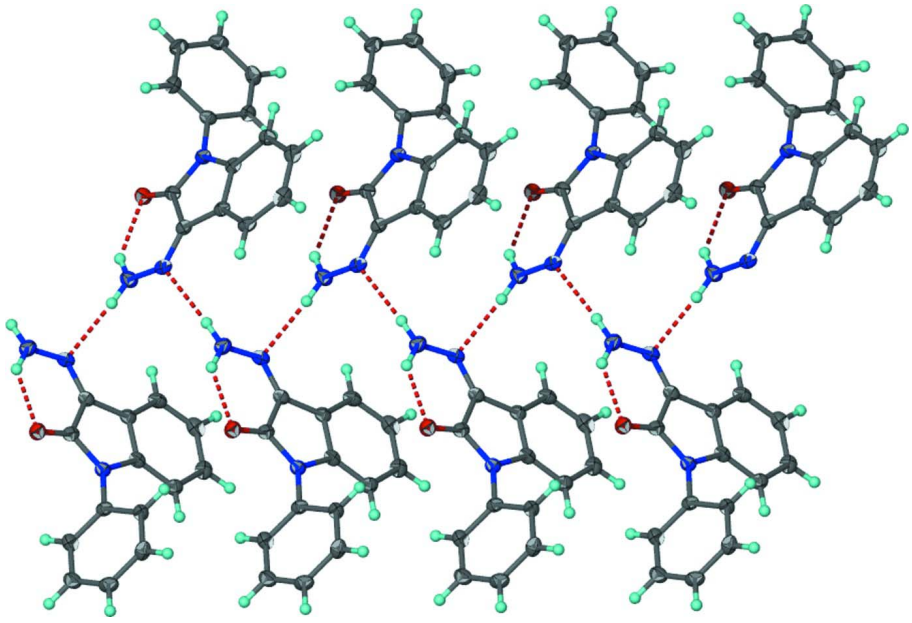

Figure 2

Hydrogen-bonded chain structure.

(*Z*)-3-Hydrazinylidene-1-phenylindolin-2-one

Crystal data

$C_{14}H_{11}N_3O$
$M_r = 237.26$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 19.328\ (3)$ Å
$b = 41.612\ (5)$ Å
$c = 5.6288\ (7)$ Å
$V = 4527\ (1)$ Å^3
$Z = 16$
$F(000) = 1984$
$D_x = 1.392$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1516 reflections
$\theta = 2.3–27.8°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Prism, yellow
0.35 × 0.04 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
10569 measured reflections
1430 independent reflections
1188 reflections with $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -24\rightarrow24$
$k = -54\rightarrow54$
$l = -7\rightarrow7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.092$
$S = 1.03$
1430 reflections
171 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Absolute structure: 1138 Friedel pairs were merged

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27334 (9)	0.09714 (4)	0.4984 (3)	0.0193 (4)
N1	0.34809 (10)	0.10293 (4)	0.1772 (4)	0.0161 (5)
N2	0.28770 (10)	0.02682 (5)	0.3752 (4)	0.0177 (5)
N3	0.24626 (11)	0.03226 (5)	0.5601 (4)	0.0201 (5)
C1	0.37995 (12)	0.08200 (6)	0.0120 (5)	0.0158 (6)
C2	0.42392 (12)	0.08987 (6)	−0.1705 (5)	0.0179 (6)
H2A	0.4368	0.1115	−0.2002	0.022*
C3	0.44890 (12)	0.06483 (6)	−0.3103 (5)	0.0206 (6)
H3	0.4788	0.0695	−0.4396	0.025*
C4	0.43087 (13)	0.03306 (6)	−0.2640 (5)	0.0213 (6)
H4	0.4485	0.0164	−0.3621	0.026*
C5	0.38720 (12)	0.02553 (6)	−0.0752 (5)	0.0192 (6)
H5	0.3753	0.0038	−0.0428	0.023*
C6	0.36135 (12)	0.05005 (6)	0.0645 (5)	0.0160 (5)
C7	0.31593 (12)	0.05137 (6)	0.2694 (4)	0.0158 (6)
C8	0.30841 (12)	0.08581 (6)	0.3372 (5)	0.0162 (5)
C9	0.35079 (12)	0.13736 (5)	0.1607 (5)	0.0158 (5)
C10	0.32204 (13)	0.15233 (6)	−0.0360 (5)	0.0181 (5)
H10	0.3004	0.1400	−0.1570	0.022*
C11	0.32528 (13)	0.18547 (6)	−0.0538 (5)	0.0207 (6)
H11	0.3055	0.1960	−0.1872	0.025*
C12	0.35737 (13)	0.20340 (6)	0.1227 (5)	0.0210 (6)
H12	0.3597	0.2261	0.1097	0.025*
C13	0.38601 (14)	0.18805 (6)	0.3177 (5)	0.0217 (6)
H13	0.4078	0.2004	0.4386	0.026*
C14	0.38314 (13)	0.15489 (6)	0.3378 (5)	0.0198 (6)
H14	0.4031	0.1444	0.4710	0.024*
H1	0.2435 (15)	0.0522 (4)	0.612 (6)	0.031 (8)*
H2	0.2355 (14)	0.0151 (5)	0.646 (5)	0.036 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0229 (9)	0.0166 (8)	0.0185 (11)	0.0001 (7)	0.0040 (9)	−0.0017 (8)
N1	0.0184 (10)	0.0129 (9)	0.0170 (12)	−0.0001 (8)	0.0014 (9)	−0.0021 (9)
N2	0.0164 (10)	0.0159 (10)	0.0207 (13)	−0.0012 (8)	−0.0024 (10)	−0.0009 (9)
N3	0.0242 (11)	0.0158 (11)	0.0203 (13)	−0.0006 (9)	0.0045 (11)	−0.0002 (10)
C1	0.0153 (11)	0.0148 (11)	0.0173 (15)	0.0024 (9)	−0.0039 (11)	−0.0006 (10)
C2	0.0176 (12)	0.0160 (11)	0.0202 (14)	0.0004 (9)	−0.0008 (12)	0.0017 (11)
C3	0.0150 (11)	0.0274 (13)	0.0194 (14)	0.0013 (10)	−0.0011 (12)	0.0005 (12)
C4	0.0183 (12)	0.0227 (12)	0.0229 (16)	0.0056 (10)	−0.0027 (12)	−0.0055 (11)

C5	0.0206 (12)	0.0138 (11)	0.0232 (15)	−0.0005 (9)	−0.0026 (13)	−0.0018 (11)
C6	0.0137 (11)	0.0163 (11)	0.0179 (14)	0.0000 (9)	−0.0036 (11)	0.0000 (11)
C7	0.0145 (12)	0.0154 (12)	0.0175 (15)	0.0002 (9)	−0.0020 (11)	−0.0014 (10)
C8	0.0147 (11)	0.0155 (11)	0.0183 (14)	−0.0006 (9)	−0.0028 (11)	0.0025 (11)
C9	0.0157 (12)	0.0125 (11)	0.0192 (14)	0.0003 (9)	0.0033 (10)	0.0003 (11)
C10	0.0211 (12)	0.0175 (12)	0.0159 (14)	−0.0013 (10)	0.0003 (11)	−0.0013 (11)
C11	0.0246 (13)	0.0204 (12)	0.0172 (14)	0.0034 (10)	0.0039 (12)	0.0011 (11)
C12	0.0246 (13)	0.0115 (10)	0.0268 (16)	−0.0012 (10)	0.0058 (13)	−0.0023 (11)
C13	0.0213 (13)	0.0216 (12)	0.0222 (15)	−0.0037 (10)	0.0025 (12)	−0.0066 (12)
C14	0.0202 (12)	0.0212 (12)	0.0181 (14)	0.0001 (10)	−0.0020 (12)	−0.0016 (11)

Geometric parameters (Å, º)

O1—C8	1.227 (3)	C5—C6	1.381 (4)
N1—C8	1.381 (3)	C5—H5	0.9500
N1—C1	1.415 (3)	C6—C7	1.451 (3)
N1—C9	1.436 (3)	C7—C8	1.491 (3)
N2—C7	1.302 (3)	C9—C14	1.385 (4)
N2—N3	1.332 (3)	C9—C10	1.387 (4)
N3—H1	0.883 (10)	C10—C11	1.384 (3)
N3—H2	0.888 (10)	C10—H10	0.9500
C1—C2	1.373 (4)	C11—C12	1.388 (4)
C1—C6	1.408 (3)	C11—H11	0.9500
C2—C3	1.392 (4)	C12—C13	1.386 (4)
C2—H2A	0.9500	C12—H12	0.9500
C3—C4	1.392 (4)	C13—C14	1.385 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.393 (4)	C14—H14	0.9500
C4—H4	0.9500		
C8—N1—C1	110.66 (19)	N2—C7—C6	126.0 (2)
C8—N1—C9	125.2 (2)	N2—C7—C8	126.6 (2)
C1—N1—C9	123.8 (2)	C6—C7—C8	107.4 (2)
C7—N2—N3	118.4 (2)	O1—C8—N1	126.3 (2)
N2—N3—H1	117 (2)	O1—C8—C7	127.8 (2)
N2—N3—H2	115 (2)	N1—C8—C7	105.9 (2)
H1—N3—H2	124 (3)	C14—C9—C10	121.3 (2)
C2—C1—C6	122.7 (2)	C14—C9—N1	119.7 (2)
C2—C1—N1	127.9 (2)	C10—C9—N1	119.0 (2)
C6—C1—N1	109.4 (2)	C11—C10—C9	119.2 (2)
C1—C2—C3	117.3 (2)	C11—C10—H10	120.4
C1—C2—H2A	121.3	C9—C10—H10	120.4
C3—C2—H2A	121.3	C10—C11—C12	120.3 (3)
C4—C3—C2	121.2 (3)	C10—C11—H11	119.9
C4—C3—H3	119.4	C12—C11—H11	119.9
C2—C3—H3	119.4	C11—C12—C13	119.8 (2)
C5—C4—C3	120.5 (2)	C11—C12—H12	120.1
C5—C4—H4	119.7	C13—C12—H12	120.1

C3—C4—H4	119.7	C14—C13—C12	120.5 (3)
C6—C5—C4	119.2 (2)	C14—C13—H13	119.7
C6—C5—H5	120.4	C12—C13—H13	119.7
C4—C5—H5	120.4	C9—C14—C13	119.0 (3)
C5—C6—C1	119.0 (2)	C9—C14—H14	120.5
C5—C6—C7	134.4 (2)	C13—C14—H14	120.5
C1—C6—C7	106.6 (2)		

C8—N1—C1—C2	−178.8 (2)	C1—N1—C8—O1	−178.4 (2)
C9—N1—C1—C2	7.9 (4)	C9—N1—C8—O1	−5.2 (4)
C8—N1—C1—C6	−0.4 (3)	C1—N1—C8—C7	0.3 (3)
C9—N1—C1—C6	−173.7 (2)	C9—N1—C8—C7	173.5 (2)
C6—C1—C2—C3	1.7 (4)	N2—C7—C8—O1	−2.1 (4)
N1—C1—C2—C3	179.9 (2)	C6—C7—C8—O1	178.5 (2)
C1—C2—C3—C4	−1.1 (4)	N2—C7—C8—N1	179.2 (2)
C2—C3—C4—C5	−0.1 (4)	C6—C7—C8—N1	−0.1 (2)
C3—C4—C5—C6	0.7 (4)	C8—N1—C9—C14	70.8 (3)
C4—C5—C6—C1	0.0 (4)	C1—N1—C9—C14	−116.8 (3)
C4—C5—C6—C7	179.9 (2)	C8—N1—C9—C10	−110.5 (3)
C2—C1—C6—C5	−1.2 (4)	C1—N1—C9—C10	61.8 (3)
N1—C1—C6—C5	−179.7 (2)	C14—C9—C10—C11	−0.6 (4)
C2—C1—C6—C7	178.8 (2)	N1—C9—C10—C11	−179.2 (2)
N1—C1—C6—C7	0.3 (3)	C9—C10—C11—C12	0.4 (4)
N3—N2—C7—C6	−179.1 (2)	C10—C11—C12—C13	−0.3 (4)
N3—N2—C7—C8	1.8 (4)	C11—C12—C13—C14	0.3 (4)
C5—C6—C7—N2	0.6 (4)	C10—C9—C14—C13	0.6 (4)
C1—C6—C7—N2	−179.4 (2)	N1—C9—C14—C13	179.2 (2)
C5—C6—C7—C8	179.9 (3)	C12—C13—C14—C9	−0.4 (4)
C1—C6—C7—C8	−0.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H1···O1	0.88 (1)	2.06 (2)	2.772 (3)	137 (3)
N3—H2···N2^{i}	0.89 (1)	2.22 (1)	3.102 (3)	177 (3)

Symmetry code: (i) −*x*+1/2, −*y*, *z*+1/2.