

Isatin 3-benzoylhydrazone: a monoclinic form

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The amine N atom of the isatin portion of the title compound (systematic name: 2-oxo-2,3-dihydro-1*H*-indole-3-carbaldehyde benzoylhydrazone), C₁₅H₁₁N₃O₂, forms a hydrogen bond with the amide O atom of the benzoylhydrazone portion of a symmetry-related molecule, giving rise to a hydrogen-bonded chain structure that propagates by a glide plane along the *c* axis of the monoclinic unit cell.

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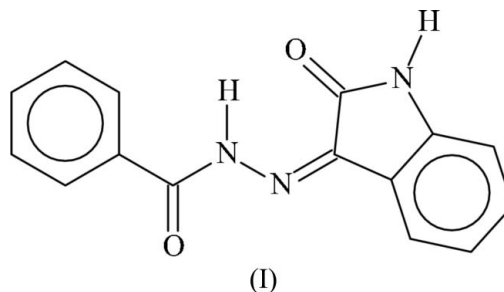
Key indicators

Single-crystal X-ray study
T = 295 K
 Mean σ (C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.125
 Data-to-parameter ratio = 14.6

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

Comment

There are a large number of structurally authenticated Schiff bases that are the condensation products of an aroylhydrazone and a carbonyl compound, as noted from the Cambridge Structural Database (Version 5.26; Allen, 2002). Among this number is a sole example of an isatin derivative, the 3-picolinoylhydrazone. Isatin aroylhydrazones are expected to function as a terdentate chelates to metals; however, in nickel bis(1-methylisatin 3-picolinoylhydrazonate), the pyridyl N atom coordinates in place of the amide O atom (Rodríguez-Argüelles *et al.*, 2004).



Isatin 3-benzoylhydrazone, (I) (Fig. 1), is a nearly planar molecule. The 3-picolinoylhydrazone mentioned above is also planar, and the planarity of the two compounds can be attributed to delocalization of the π electrons over the entire molecule through the –N–NH–C(O)– portion. Other Schiff-base derivatives of isatin that have been crystallographically characterized include (4-hexylphenyl)imino-1*H*-indol-2(3*H*-one) (Öztürk *et al.*, 2003), indole-2,3-dione 3-(trimethylammonioacetyl)hydrazone chloride (Sun *et al.*, 2001), indole-2,3-dione 3-(hexamethyleneiminyl)thiosemicarbazone (Bain *et al.*, 1997), 2,3-bis(thiosemicarbazone) (Casas *et al.*, 2000) and indole-2,3-dione β -4-(4-tolyl)thiocarbazone (Revenko *et al.*, 1994). Useful biological properties (Öztürk *et al.*, 2003) are expected of the title compound and other Schiff bases. The title compound features an intramolecular hydrogen bond; adjacent molecules are linked by another hydrogen bond to form a chain. The following paper (Ali *et al.*, 2005) describes an orthorhombic polymorph of the title compound.

Experimental

Benzhydrazide (0.50 g, 3.7 mmol) and isatin (0.54 g, 3.7 mol) were refluxed in ethanol (50 ml) for 2 h. The solid that was formed was filtered off and recrystallized from pyridine to give well defined orange-colored blocks.

Crystal data

$C_{15}H_{11}N_3O_2$
 $M_r = 265.27$
 Monoclinic, $P2_1/c$
 $a = 8.9279$ (6) Å
 $b = 12.1099$ (9) Å
 $c = 11.7567$ (9) Å
 $\beta = 95.117$ (1)°
 $V = 1266.0$ (2) Å³
 $Z = 4$

$D_x = 1.392$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2811 reflections
 $\theta = 2.4$ – 26.3 °
 $\mu = 0.10$ mm⁻¹
 $T = 295$ (2) K
 Block, orange
 $0.48 \times 0.32 \times 0.26$ mm

Data collection

Bruker SMART area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 7680 measured reflections
 2762 independent reflections

1878 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.026$
 $\theta_{max} = 27.1$ °
 $h = -11 \rightarrow 10$
 $k = -15 \rightarrow 15$
 $l = -15 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.125$
 $S = 1.01$
 2762 reflections
 189 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.1615P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.17$ e Å⁻³
 $\Delta\rho_{min} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1n \cdots O2$	0.86 (1)	2.00 (1)	2.712 (2)	139 (2)
$N3-H3n \cdots O1^i$	0.87 (1)	2.15 (1)	2.918 (2)	148 (2)

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

The C-bound H atoms were placed at calculated positions ($C-H = 0.93$ Å), and they were included in the refinement in the riding-model approximation with $U_{iso}(H)$ set to $1.2U_{eq}(C)$. The N-bound H atoms were located in a difference Fourier map and refined with a distance restraint of $N-H = 0.86$ (1) Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

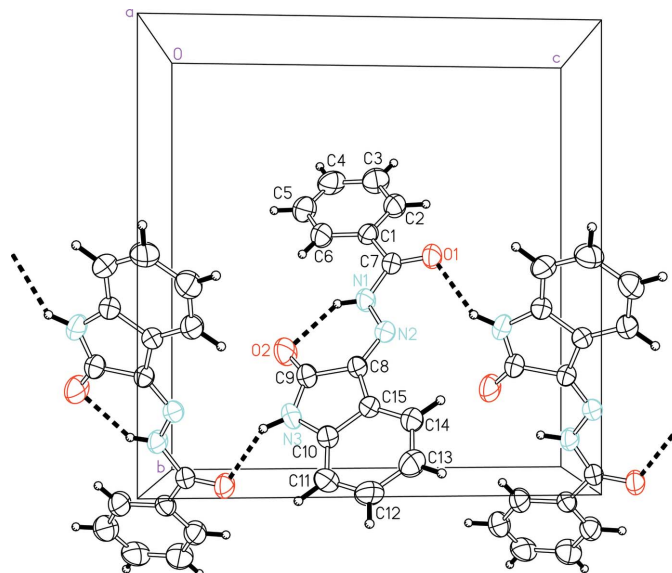


Figure 1

ORTEPII (Johnson, 1976) plot of $C_{15}H_{11}N_3O_2$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

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