Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Isatin 3-benzoylhydrazone: an orthorhombic form

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.058
wR factor = 0.191
Data-to-parameter ratio = 8.1

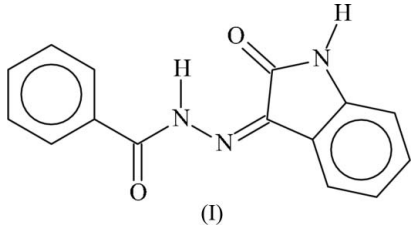
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 September 2005
Accepted 12 September 2005
Online 17 September 2005

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), $C_{15}H_{11}N_3O_2$, forms a hydrogen bond with the amide O atom of the benzoylhydrazone portion of a symmetry-related molecule to give rise to a hydrogen-bonded chain structure that propagates by a twofold screw axis along the *a* axis of the orthorhombic unit cell.

Comment

The preceeding report describes the structure of the monoclinic form of isatin 3-benzoylhydrazone (Ali *et al.*, 2005). In the orthorhombic form, the chain formed by hydrogen-bonded molecules propagates by twofold screw axial translations (Fig. 1), whereas in the monoclinic form the chain formed by hydrogen-bonded molecules propagates by glide planes. The orthorhombic form is significantly denser (density = 1.467 Mg m^{-3}) than the monoclinic form (density = 1.392 Mg m^{-3}), as the polymeric chain is more flexible owing to a more adaptable, helical construction.



(I)

Experimental

Isatin 3-benzoylhydrazone (0.50 g, 1.9 mmol) and zinc acetate (0.20 g, 0.9 mmol) were refluxed for 5 h. The yellow solid that precipitated was collected and recrystallized from pyridine to give yellow–orange blocks. The color is distinctly different from that of the monoclinic polymorph.

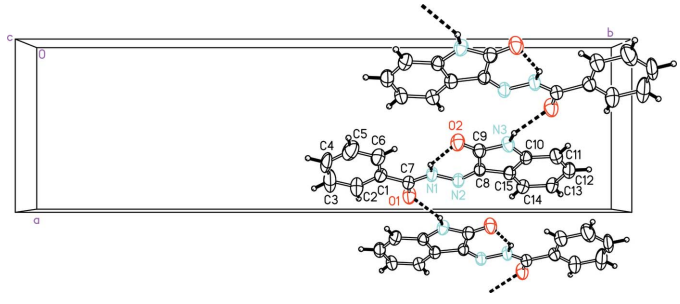


Figure 1
ORTEPII (Johnson, 1976) plot of $C_{15}H_{11}N_3O_2$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

Crystal data

$C_{15}H_{11}N_3O_2$
$M_r = 265.27$
Orthorhombic, $Pna2_1$
$a = 8.0023$ (9) Å
$b = 28.549$ (3) Å
$c = 5.2574$ (6) Å
$V = 1201.1$ (2) Å^3
$Z = 4$
$D_x = 1.467$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 928 reflections
$\theta = 2.5$–25.7°
$\mu = 0.10$ mm^{-1}
$T = 295$ (2) K
Block, yellow–orange
0.34 × 0.21 × 0.15 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scan
Absorption correction: none
6999 measured reflections
1463 independent reflections
1176 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.1^\circ$
$h = -8 \rightarrow 10$
$k = -36 \rightarrow 36$
$l = -6 \rightarrow 5$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.191$
$S = 1.08$
1463 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1317P)^2 + 0.2812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1n···O2	0.86	2.01	2.699 (5)	137
N3—H3n···O1^{i}	0.86	2.12	2.862 (5)	145

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

The phenyl ring comprising atoms C1–C6 was refined with the C—C distances restrained to 1.390 (5) Å; the 1,4-distances were restrained to 2.780 (5) Å. As there are no heavy scatterers, Friedel pairs were merged. H atoms were placed at calculated positions (C—H = 0.93 Å and N—H = 0.86 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) set to $1.2U_{eq}$(C,N).

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*.

The authors thank the Scientific Advancement Grant Allocation (No. 66-02-03-0046/Oracle 815-0046) and the University of Malaya for supporting this study. We also thank Mr Xiao-Long Feng of Sun Yat-Sen University for the diffraction measurements.

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