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1*H*-Indole-3-carbaldehyde 2-nitrobenzoylhydrazone hemihydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.174
Data-to-parameter ratio = 14.9

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

In the title structure, $C_{16}H_{12}N_4O_3\cdot0.5H_2O$, the uncoordinated water molecule lies on a special position of site symmetry 2; O—H···O and N—H···O hydrogen bonds link the water and organic molecules into a layer structure.

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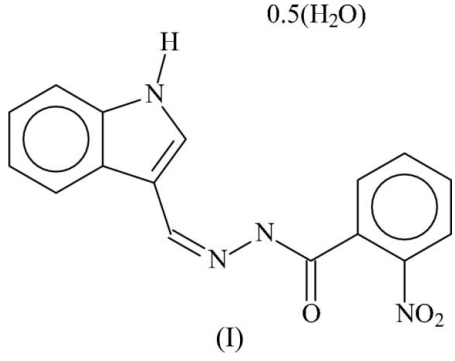
Comment

A recent study (Ali *et al.*, 2005) reports the structure of the bromo-substituted derivative of the title Schiff base, (I), the condensation product of indole-3-carbaldehyde and 2-nitrobenzoylhydrazine. The crystal structures of both the bromo derivative and the title compound exist as hemihydrates whose water molecules lie on twofold rotation axes. There are no significant differences in the bond lengths of the previously reported and the title molecules (Fig. 1) and in both structures weak O—H···O and N—H···O hydrogen bonds link molecules into a two-dimensional network (Table 1).



(I)

Experimental

Indole-3-carbaldehyde (0.50 g, 3.5 mmol) and 2-nitrobenzoylhydrazine (0.62 g, 3.5 mol) were heated in ethanol (50 ml) for 2 h. Orange crystals separated from the cooled solution after a day.

Crystal data

$C_{16}H_{12}N_4O_3\cdot0.5H_2O$
M_r = 317.30
Monoclinic, $C2/c$
a = 28.983 (2) Å
b = 6.0809 (5) Å
c = 16.559 (1) Å
β = 95.397 (2)°
V = 2905.5 (4) Å^3
Z = 8
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1977 reflections
θ = 2.3–23.7°
μ = 0.11 mm^{-1}
T = 295 (2) K
Block, orange–red
0.26 × 0.23 × 0.16 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
8682 measured reflections
3167 independent reflections
1720 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.1°
$h = -30 \rightarrow 36$
$k = -7 \rightarrow 7$
$l = -21 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.0931P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.174$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
3167 reflections	$\Delta\rho_{min} = -0.54$ e Å^{-3}
213 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2n···O3^{i}	0.86	2.11	2.961 (3)	169
N4—H4n···O1^{ii}	0.86	2.35	3.087 (3)	144
N4—H4n···O1w^{iii}	0.86	2.40	2.985 (3)	126
O1w—H1w···O2	0.87	2.31	3.066 (3)	146

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

H atoms bonded to C and N atoms were included in 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). The unique water H atom was placed in a chemically sensible position on the basis of hydrogen bonding but it was not refined and U_{iso}(H) was set at $1.2U_{eq}$(O).

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*.

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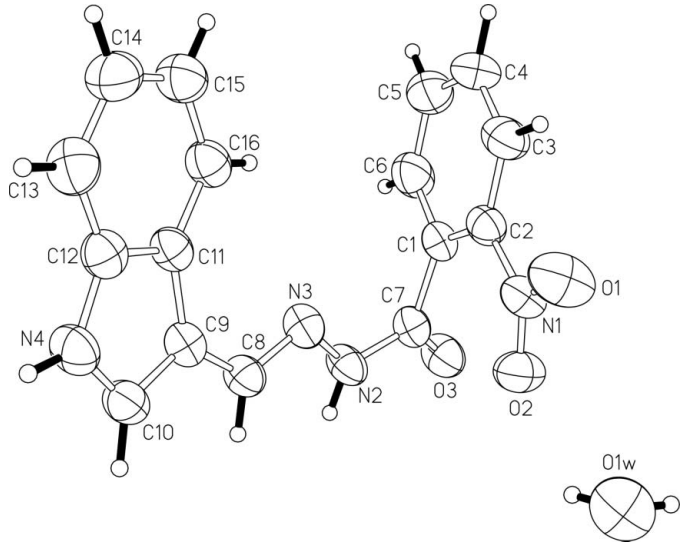

Figure 1
ORTEPII (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

References

Ali, M. H., Abdul Halim, S. N. & Ng, S. W. (2005). *Acta Cryst.* E**61**, o2308–o2309.
Bruker (2000). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.