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5-Bromo-1*H*-indole-3-carbaldehyde 2-nitrophenylhydrazone hemihydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.110
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 compound, $C_{16}H_{11}BrN_4O_3\cdot0.5H_2O$, the water molecule lies on a twofold rotation axis. Symmetry-related molecules are linked to form an N—H···O and O—H···O hydrogen-bonded layer structure.

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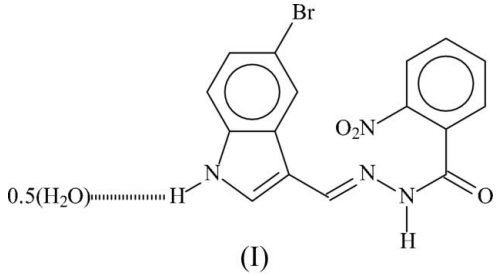
Comment

A previous study on the Schiff base derived from 5-bromoindole-3-carbaldehyde details the structure of a thienoylhydrazone derivative (Ali *et al.*, 2005). Replacing the thienyl ring by the 2-nitrophenyl group leads to no significant differences in bonds connecting the two rings; the title compound, (I) (Fig. 1), crystallizes as a hemihydrate, and adjacent molecules are linked by hydrogen bonds (Table 1) into a layer structure.



(I)

Experimental

5-Bromoindole-3-carboxaldehyde (0.50 g, 2.23 mmol) and 2-nitrobenzhydrazide (0.37 g, 2.23 mmol) were heated in ethanol for 2 h. The solvent was removed to give the crude product, which was then purified by recrystallization from ethyl acetate.

Crystal data

$C_{16}H_{11}BrN_4O_3\cdot0.5H_2O$
M_r = 396.21
Monoclinic, $P2/a$
a = 16.547 (4) Å
b = 6.053 (2) Å
c = 16.924 (4) Å
β = 111.93 (2)°
V = 1572.3 (8) Å^3
Z = 4
D_x = 1.674 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9035 reflections
θ = 3.4–27.5°
μ = 2.64 mm^{-1}
T = 295 (2) K
Block, orange
0.29 × 0.21 × 0.16 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.306, T_{max} = 0.677
14182 measured reflections
3491 independent reflections
2481 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°
$h = -21 \rightarrow 21$
$k = -7 \rightarrow 7$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.110$
$S = 1.05$
3491 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.5445P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.63$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2n···O3^{i}	0.85 (1)	2.09 (1)	2.937 (3)	173 (3)
N4—H4n···O1w	0.85 (1)	2.37 (3)	2.973 (3)	128 (3)
N4—H4n···O1^{ii}	0.85 (1)	2.43 (2)	3.142 (3)	142 (3)
O1w—H1w···O2^{ii}	0.85 (1)	2.22 (2)	3.061 (3)	171 (7)

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

The C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) set at 1.2U_{eq}(C). The water and amine H atoms were located in a difference Fourier map and were refined with a distance restraint of 0.85 (1) Å.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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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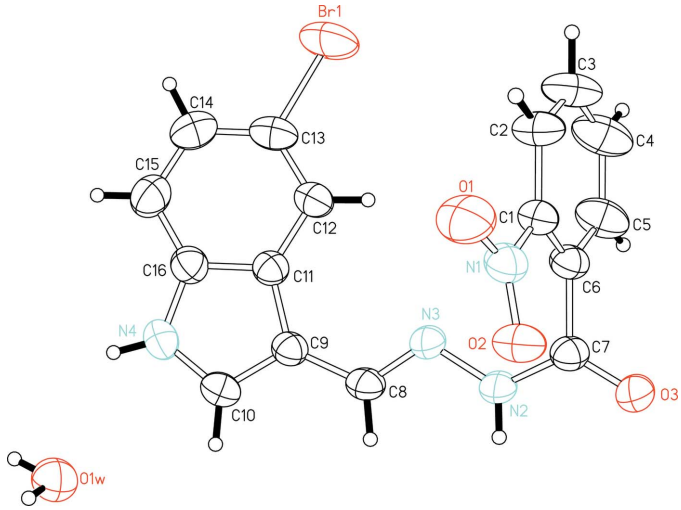

Figure 1
ORTEPII plot (Johnson, 1976) of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

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