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N′-(5-Bromo-1*H*-indol-3-ylmethylidene)-3,4,5-trihydroxybenzohydrazide

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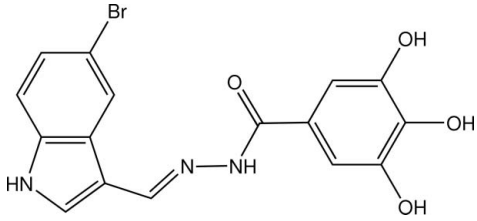
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.078; data-to-parameter ratio = 15.5.

The two aromatic parts of the title molecule, $C_{16}H_{12}BrN_3O_4$, are connected through a conjugated –CH=N—NH—C(O)– fragment to furnish an almost planar molecule. Adjacent molecules are linked by N—H···O and O—H···O hydrogen bonds into a three-dimensional network. An intramolecular O—H···O link also occurs.

Related literature

For other Schiff bases derived by condensing 5-bromo-1*H*-indole-3-carbaldehyde with aroylhydrazines, see: Ali *et al.* (2005*a*,*b*,*c*).



Experimental

Crystal data

$C_{16}H_{12}BrN_3O_4$
M_r = 390.20
Monoclinic, $P2_1/n$
a = 9.6454 (2) Å
b = 14.9694 (4) Å
c = 10.3845 (2) Å
β = 97.390 (1)°
V = 1486.92 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.79 mm^{-1}
T = 100 (2) K
0.40 × 0.25 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.401, T_{max} = 0.768
10182 measured reflections
3403 independent reflections
2786 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.078
S = 1.02
3403 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O3	0.84	2.21	2.681 (2)	116
O3—H3O···O1^{i}	0.84	1.76	2.595 (2)	173
O4—H4O···N2^{i}	0.84	2.02	2.778 (2)	150
N1—H1N···O2^{ii}	0.88	2.26	3.111 (2)	163
N3—H3N···O4^{iii}	0.88	2.11	2.932 (2)	154

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2310).

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supporting information

Acta Cryst. (2008). E**64**, o2108 [doi:10.1107/S1600536808031991]

N′-(5-Bromo-1*H*-indol-3-ylmethylidene)-3,4,5-trihydroxybenzohydrazide

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S1. Comment

The molecule of (I), Fig. 1, is almost planar with the aromatic groups connected via a conjugated –CH═N–NH–C(O)– fragment. Molecules are connected into a 3-D network via N—H···O and O—H···O hydrogen bonds, Table 1.

S2. Experimental

5-Bromoindole-3-carbaldehyde (0.34 g, 1.5 mmol) and 3,4,5-trihydroxybenzoylhydrazine (0.27 g, 1.5 mmol) were heated in ethanol (20 ml) for 3 h. About 1 ml of acetic acid also added. The solution was set aside for the growth of crystals.

S3. Refinement

Hydrogen atoms were placed at calculated positions (C—H 0.95, N—H 0.88 and O—H 0.84 Å) and were treated as riding on their parent atoms, with U(H) set to 1.2–1.5 times U_{eq}(C,N,O). For the hydroxy groups, an sp^2 type of hybridization was assumed.

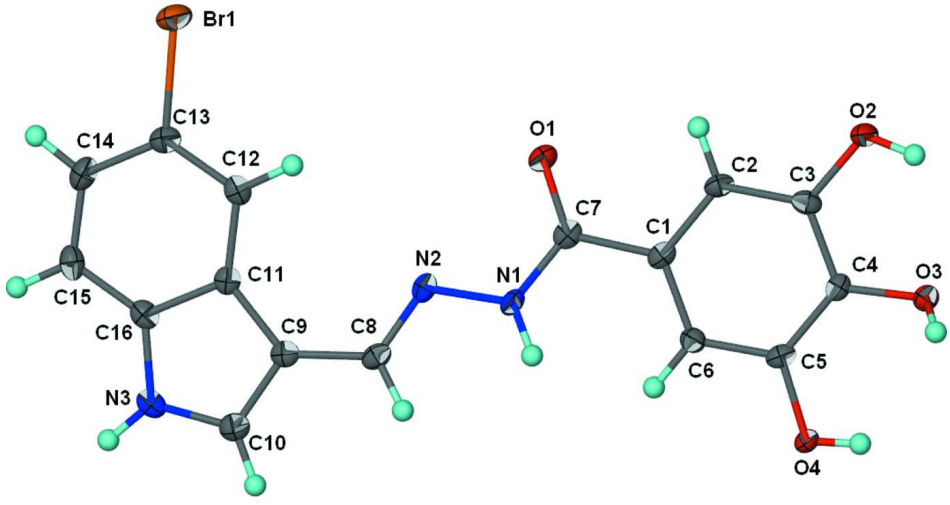

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of (I) at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

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Crystal data

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Hall symbol: -P 2yn
a = 9.6454 (2) Å
b = 14.9694 (4) Å

c = 10.3845 (2) Å
β = 97.390 (1)°
V = 1486.92 (6) Å^3
Z = 4
F(000) = 784
D_x = 1.743 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 3960 reflections
θ = 2.4–28.2°
μ = 2.79 mm^{-1}
T = 100 K
Block, orange
0.40 × 0.25 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
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Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.401, T_{max} = 0.768

10182 measured reflections
3403 independent reflections
2786 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −12→11
k = −19→19
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.078
S = 1.02
3403 reflections
220 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.9663P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.00401 (2)	1.090710 (16)	0.25205 (2)	0.02243 (8)
O1	0.69032 (15)	0.83598 (10)	0.56939 (13)	0.0157 (3)
O2	0.64359 (15)	0.62930 (10)	0.94165 (13)	0.0163 (3)
H2O	0.5987	0.5999	0.9914	0.024*
O3	0.37031 (15)	0.59355 (9)	0.93388 (13)	0.0133 (3)
H3O	0.3085	0.6178	0.9721	0.020*
O4	0.17544 (15)	0.67374 (10)	0.73325 (13)	0.0141 (3)
H4O	0.1569	0.6421	0.7956	0.021*
N1	0.46882 (18)	0.86863 (11)	0.48158 (15)	0.0134 (3)
H1N	0.3788	0.8619	0.4852	0.016*
N2	0.51457 (18)	0.92346 (11)	0.38712 (16)	0.0129 (3)
N3	0.40143 (19)	1.10565 (11)	0.03097 (16)	0.0151 (4)
H3N	0.3557	1.1326	−0.0369	0.018*

C1	0.5056 (2)	0.76400 (13)	0.66032 (18)	0.0123 (4)
C2	0.6012 (2)	0.72451 (13)	0.75620 (18)	0.0129 (4)
H2	0.6984	0.7361	0.7598	0.016*
C3	0.5519 (2)	0.66829 (13)	0.84567 (18)	0.0128 (4)
C4	0.4097 (2)	0.65084 (13)	0.84261 (18)	0.0116 (4)
C5	0.3160 (2)	0.68919 (13)	0.74520 (18)	0.0122 (4)
C6	0.3639 (2)	0.74523 (13)	0.65414 (18)	0.0124 (4)
H6	0.2996	0.7709	0.5873	0.015*
C7	0.5628 (2)	0.82603 (13)	0.56719 (18)	0.0128 (4)
C8	0.4168 (2)	0.96589 (13)	0.31563 (19)	0.0133 (4)
H8	0.3229	0.9587	0.3324	0.016*
C9	0.4446 (2)	1.02350 (13)	0.21187 (19)	0.0125 (4)
C10	0.3424 (2)	1.05580 (14)	0.11812 (19)	0.0150 (4)
H10	0.2451	1.0447	0.1152	0.018*
C11	0.5760 (2)	1.05663 (13)	0.17868 (18)	0.0121 (4)
C12	0.7151 (2)	1.05011 (13)	0.23622 (18)	0.0135 (4)
H12	0.7399	1.0177	0.3144	0.016*
C13	0.8147 (2)	1.09278 (13)	0.1744 (2)	0.0152 (4)
C14	0.7829 (2)	1.14153 (14)	0.0594 (2)	0.0172 (4)
H14	0.8558	1.1685	0.0195	0.021*
C15	0.6456 (2)	1.15045 (14)	0.00394 (19)	0.0158 (4)
H15	0.6215	1.1845	−0.0728	0.019*
C16	0.5443 (2)	1.10761 (13)	0.06493 (19)	0.0138 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01187 (12)	0.03131 (14)	0.02403 (13)	−0.00169 (9)	0.00198 (8)	0.00285 (9)
O1	0.0119 (7)	0.0181 (7)	0.0178 (7)	−0.0008 (6)	0.0047 (6)	0.0012 (6)
O2	0.0100 (7)	0.0235 (8)	0.0151 (7)	−0.0005 (6)	0.0010 (6)	0.0048 (6)
O3	0.0120 (7)	0.0162 (7)	0.0122 (6)	0.0003 (6)	0.0039 (5)	0.0011 (5)
O4	0.0101 (7)	0.0174 (7)	0.0145 (7)	−0.0035 (6)	0.0005 (5)	0.0038 (5)
N1	0.0114 (9)	0.0144 (8)	0.0152 (8)	−0.0019 (7)	0.0050 (7)	0.0021 (7)
N2	0.0149 (9)	0.0123 (8)	0.0125 (7)	−0.0020 (7)	0.0051 (7)	−0.0006 (6)
N3	0.0158 (9)	0.0156 (9)	0.0133 (8)	0.0028 (7)	−0.0004 (7)	−0.0001 (6)
C1	0.0123 (10)	0.0122 (9)	0.0133 (9)	−0.0012 (8)	0.0044 (7)	−0.0035 (7)
C2	0.0093 (9)	0.0150 (9)	0.0150 (9)	0.0001 (8)	0.0031 (7)	−0.0024 (8)
C3	0.0116 (10)	0.0149 (10)	0.0116 (8)	0.0016 (8)	−0.0002 (7)	−0.0027 (7)
C4	0.0133 (10)	0.0111 (9)	0.0109 (8)	−0.0017 (7)	0.0035 (7)	−0.0017 (7)
C5	0.0101 (9)	0.0126 (9)	0.0143 (9)	−0.0010 (8)	0.0025 (7)	−0.0042 (7)
C6	0.0119 (10)	0.0124 (9)	0.0127 (9)	0.0006 (8)	0.0009 (7)	0.0003 (7)
C7	0.0148 (10)	0.0114 (9)	0.0131 (9)	−0.0005 (8)	0.0046 (8)	−0.0044 (7)
C8	0.0117 (10)	0.0133 (9)	0.0155 (9)	−0.0012 (8)	0.0042 (7)	−0.0031 (8)
C9	0.0131 (10)	0.0104 (9)	0.0141 (9)	0.0005 (8)	0.0018 (8)	−0.0029 (7)
C10	0.0144 (10)	0.0146 (9)	0.0162 (9)	0.0008 (8)	0.0027 (8)	−0.0021 (8)
C11	0.0148 (10)	0.0099 (9)	0.0119 (8)	0.0009 (8)	0.0032 (7)	−0.0025 (7)
C12	0.0165 (10)	0.0125 (9)	0.0116 (9)	0.0023 (8)	0.0028 (7)	−0.0013 (7)
C13	0.0132 (10)	0.0146 (10)	0.0176 (9)	0.0000 (8)	0.0017 (8)	−0.0028 (8)

C14	0.0194 (11)	0.0149 (10)	0.0185 (10)	−0.0026 (9)	0.0072 (8)	−0.0002 (8)
C15	0.0233 (11)	0.0116 (9)	0.0128 (9)	0.0002 (8)	0.0035 (8)	0.0004 (7)
C16	0.0166 (10)	0.0117 (9)	0.0126 (9)	0.0021 (8)	0.0005 (8)	−0.0024 (7)

Geometric parameters (Å, º)

Br1—C13	1.899 (2)	C2—H2	0.9500
O1—C7	1.236 (3)	C3—C4	1.392 (3)
O2—C3	1.375 (2)	C4—C5	1.391 (3)
O2—H2O	0.8400	C5—C6	1.387 (3)
O3—C4	1.368 (2)	C6—H6	0.9500
O3—H3O	0.8400	C8—C9	1.432 (3)
O4—C5	1.365 (2)	C8—H8	0.9500
O4—H4O	0.8400	C9—C10	1.381 (3)
N1—C7	1.346 (3)	C9—C11	1.443 (3)
N1—N2	1.394 (2)	C10—H10	0.9500
N1—H1N	0.8800	C11—C12	1.401 (3)
N2—C8	1.290 (3)	C11—C16	1.406 (3)
N3—C10	1.354 (3)	C12—C13	1.379 (3)
N3—C16	1.378 (3)	C12—H12	0.9500
N3—H3N	0.8800	C13—C14	1.400 (3)
C1—C6	1.388 (3)	C14—C15	1.381 (3)
C1—C2	1.398 (3)	C14—H14	0.9500
C1—C7	1.497 (3)	C15—C16	1.388 (3)
C2—C3	1.382 (3)	C15—H15	0.9500
C3—O2—H2O	109.5	O1—C7—C1	120.77 (18)
C4—O3—H3O	109.5	N1—C7—C1	116.61 (18)
C5—O4—H4O	109.5	N2—C8—C9	122.40 (19)
C7—N1—N2	119.79 (17)	N2—C8—H8	118.8
C7—N1—H1N	120.1	C9—C8—H8	118.8
N2—N1—H1N	120.1	C10—C9—C8	123.76 (19)
C8—N2—N1	114.89 (17)	C10—C9—C11	106.28 (17)
C10—N3—C16	109.48 (17)	C8—C9—C11	129.92 (19)
C10—N3—H3N	125.3	N3—C10—C9	109.93 (19)
C16—N3—H3N	125.3	N3—C10—H10	125.0
C6—C1—C2	120.15 (18)	C9—C10—H10	125.0
C6—C1—C7	122.57 (18)	C12—C11—C16	119.25 (18)
C2—C1—C7	117.28 (18)	C12—C11—C9	134.17 (18)
C3—C2—C1	118.93 (18)	C16—C11—C9	106.54 (18)
C3—C2—H2	120.5	C13—C12—C11	117.12 (18)
C1—C2—H2	120.5	C13—C12—H12	121.4
O2—C3—C2	120.06 (18)	C11—C12—H12	121.4
O2—C3—C4	118.49 (17)	C12—C13—C14	123.3 (2)
C2—C3—C4	121.44 (18)	C12—C13—Br1	118.96 (15)
O3—C4—C3	117.54 (18)	C14—C13—Br1	117.63 (16)
O3—C4—C5	123.37 (18)	C15—C14—C13	119.95 (19)
C3—C4—C5	119.04 (18)	C15—C14—H14	120.0

O4—C5—C6	117.06 (17)	C13—C14—H14	120.0
O4—C5—C4	122.77 (17)	C14—C15—C16	117.31 (19)
C6—C5—C4	120.17 (19)	C14—C15—H15	121.3
C1—C6—C5	120.23 (18)	C16—C15—H15	121.3
C1—C6—H6	119.9	N3—C16—C15	129.25 (19)
C5—C6—H6	119.9	N3—C16—C11	107.77 (18)
O1—C7—N1	122.61 (18)	C15—C16—C11	122.99 (19)
C7—N1—N2—C8	−175.49 (18)	N2—C8—C9—C10	166.76 (19)
C6—C1—C2—C3	−1.3 (3)	N2—C8—C9—C11	−10.6 (3)
C7—C1—C2—C3	178.65 (17)	C16—N3—C10—C9	0.0 (2)
C1—C2—C3—O2	−179.39 (17)	C8—C9—C10—N3	−177.81 (18)
C1—C2—C3—C4	−0.4 (3)	C11—C9—C10—N3	0.1 (2)
O2—C3—C4—O3	−1.8 (3)	C10—C9—C11—C12	177.6 (2)
C2—C3—C4—O3	179.23 (17)	C8—C9—C11—C12	−4.7 (4)
O2—C3—C4—C5	−179.37 (17)	C10—C9—C11—C16	−0.1 (2)
C2—C3—C4—C5	1.7 (3)	C8—C9—C11—C16	177.60 (19)
O3—C4—C5—O4	0.9 (3)	C16—C11—C12—C13	−1.9 (3)
C3—C4—C5—O4	178.29 (17)	C9—C11—C12—C13	−179.3 (2)
O3—C4—C5—C6	−178.58 (18)	C11—C12—C13—C14	0.2 (3)
C3—C4—C5—C6	−1.2 (3)	C11—C12—C13—Br1	177.22 (14)
C2—C1—C6—C5	1.8 (3)	C12—C13—C14—C15	1.6 (3)
C7—C1—C6—C5	−178.16 (18)	Br1—C13—C14—C15	−175.44 (15)
O4—C5—C6—C1	179.97 (17)	C13—C14—C15—C16	−1.7 (3)
C4—C5—C6—C1	−0.6 (3)	C10—N3—C16—C15	−179.9 (2)
N2—N1—C7—O1	2.9 (3)	C10—N3—C16—C11	0.0 (2)
N2—N1—C7—C1	−176.32 (16)	C14—C15—C16—N3	179.8 (2)
C6—C1—C7—O1	−174.49 (18)	C14—C15—C16—C11	0.0 (3)
C2—C1—C7—O1	5.5 (3)	C12—C11—C16—N3	−178.04 (17)
C6—C1—C7—N1	4.7 (3)	C9—C11—C16—N3	0.1 (2)
C2—C1—C7—N1	−175.23 (17)	C12—C11—C16—C15	1.8 (3)
N1—N2—C8—C9	−178.50 (17)	C9—C11—C16—C15	179.91 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O3	0.84	2.21	2.681 (2)	116
O3—H3*O*···O1^{i}	0.84	1.76	2.595 (2)	173
O4—H4*O*···N2^{i}	0.84	2.02	2.778 (2)	150
N1—H1*N*···O2^{ii}	0.88	2.26	3.111 (2)	163
N3—H3*N*···O4^{iii}	0.88	2.11	2.932 (2)	154

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*+1/2; (ii) *x*−1/2, −*y*+3/2, *z*−1/2; (iii) −*x*+1/2, *y*+1/2, −*z*+1/2.