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N'-[(5-Chloro-1*H*-indol-3-yl)methylene]-3,4,5-trihydroxybenzohydrazide

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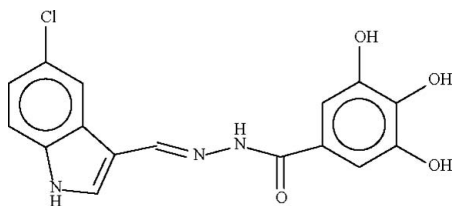
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.034; wR factor = 0.093; data-to-parameter ratio = 15.4.

The two aromatic parts of the title compound, $\text{C}_{16}\text{H}_{13}\text{ClN}_3\text{O}_4$, are connected through a conjugated $-\text{CH}=\text{N}-\text{NH}-\text{C}(\text{O})-$ fragment, giving an almost planar molecule (r.m.s. deviation 0.08 Å). In the crystal structure, adjacent molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds into a three-dimensional network.

Related literature

 For the isostructural $\text{C}_{16}\text{H}_{13}\text{BrN}_3\text{O}_4$ analog, see: Khaledi *et al.* (2008).


Experimental

Crystal data

 $\text{C}_{16}\text{H}_{12}\text{ClN}_3\text{O}_4$
 $M_r = 345.74$
 Monoclinic, $P2_1/n$
 $a = 9.6481$ (2) Å

 $b = 15.1408$ (3) Å
 $c = 10.2206$ (2) Å
 $\beta = 98.232$ (1)°
 $V = 1477.64$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 100$ (2) K
 $0.32 \times 0.22 \times 0.12$ mm

Data collection

 Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.914$, $T_{\text{max}} = 0.966$

 10185 measured reflections
 3389 independent reflections
 2907 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.093$
 $S = 1.06$
 3389 reflections

 220 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}2-\text{H}2\cdots\text{O}3$	0.84	2.20	2.6726 (14)	116
$\text{O}3-\text{H}3\cdots\text{O}1^i$	0.84	1.74	2.5818 (14)	175
$\text{O}4-\text{H}4\cdots\text{N}2^j$	0.84	2.01	2.7668 (15)	149
$\text{N}1-\text{H}1\cdots\text{O}2^{\text{ii}}$	0.88	2.23	3.0875 (16)	163
$\text{N}3-\text{H}3\cdots\text{O}4^{\text{iii}}$	0.88	2.15	2.9518 (15)	152

 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: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; 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: *pubCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2471).

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supplementary materials

Acta Cryst. (2009). E65, o169 [doi:10.1107/S160053680804258X]

***N'*-[5-Chloro-1*H*-indol-3-yl)methylene]-3,4,5-trihydroxybenzohydrazide**

H. Khaledi, H. Mohd Ali and S. W. Ng

Comment

(type here to add)

Experimental

5-Chloroindole-3-carbaldehyde (0.27 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 solid that separated out was collected, washed with water and then recrystallized from DMSO.

Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95, N–H 0.88, O–H 0.84 Å) and were treated as riding on their parent carbon atoms, with $U(H)$ set to 1.2–1.5 times $U_{eq}(C,N,O)$. The hydroxy groups were rotated to fit the electron density.

Figures

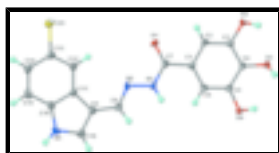


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{16}H_{12}ClN_3O_4$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

***N'*-[5-Chloro-1*H*-indol-3-yl)methylene]-3,4,5-trihydroxybenzohydrazide**

Crystal data

$C_{16}H_{12}ClN_3O_4$

$M_r = 345.74$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 9.6481$ (2) Å

$b = 15.1408$ (3) Å

$c = 10.2206$ (2) Å

$\beta = 98.232$ (1)°

$V = 1477.64$ (5) Å³

$Z = 4$

$F_{000} = 712$

$D_x = 1.554$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4113 reflections

$\theta = 2.4$ – 28.2 °

$\mu = 0.29$ mm⁻¹

$T = 100$ (2) K

Prism, orange

$0.32 \times 0.22 \times 0.12$ mm

supplementary materials

Data collection

Bruker SMART APEX diffractometer	3389 independent reflections
Radiation source: fine-focus sealed tube	2907 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 100(2)$ K	$\theta_{\text{max}} = 27.5^\circ$
ω scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 12$
$T_{\text{min}} = 0.914$, $T_{\text{max}} = 0.966$	$k = -19 \rightarrow 19$
10185 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.6635P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
3389 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
220 parameters	$\Delta\rho_{\text{max}} = 0.34 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.33 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.99034 (4)	1.07558 (3)	0.24671 (4)	0.02606 (12)
O1	0.68592 (10)	0.82901 (7)	0.56884 (10)	0.0165 (2)
O2	0.64086 (10)	0.62519 (7)	0.94667 (10)	0.0172 (2)
H2O	0.5962	0.5961	0.9968	0.026*
O3	0.36728 (11)	0.59550 (6)	0.94304 (9)	0.0140 (2)
H3O	0.3047	0.6195	0.9801	0.021*
O4	0.16964 (10)	0.67781 (7)	0.73963 (10)	0.0149 (2)
H4O	0.1521	0.6414	0.7971	0.022*
N1	0.46397 (12)	0.86816 (8)	0.48430 (11)	0.0138 (2)
H1N	0.3740	0.8638	0.4895	0.017*
N2	0.51003 (13)	0.92205 (7)	0.38882 (11)	0.0137 (2)
N3	0.39990 (13)	1.10793 (8)	0.03144 (12)	0.0162 (3)
H3N	0.3544	1.1361	-0.0369	0.019*
C1	0.50119 (15)	0.76183 (9)	0.66318 (13)	0.0130 (3)
C2	0.59758 (15)	0.72084 (9)	0.75913 (13)	0.0141 (3)
H2	0.6952	0.7305	0.7616	0.017*
C3	0.54846 (15)	0.66576 (9)	0.85061 (13)	0.0131 (3)

C4	0.40551 (15)	0.65121 (9)	0.84928 (13)	0.0123 (3)
C5	0.31087 (14)	0.69082 (9)	0.75114 (13)	0.0125 (3)
C6	0.35827 (15)	0.74599 (9)	0.65851 (13)	0.0133 (3)
H6	0.2932	0.7730	0.5919	0.016*
C7	0.55811 (15)	0.82260 (9)	0.56894 (13)	0.0131 (3)
C8	0.41317 (15)	0.96681 (9)	0.31823 (13)	0.0142 (3)
H8	0.3195	0.9615	0.3358	0.017*
C9	0.44207 (15)	1.02444 (9)	0.21398 (13)	0.0142 (3)
C10	0.33977 (16)	1.05950 (9)	0.11972 (14)	0.0162 (3)
H10	0.2419	1.0509	0.1172	0.019*
C11	0.57451 (15)	1.05429 (9)	0.18016 (13)	0.0133 (3)
C12	0.71420 (15)	1.04422 (9)	0.23767 (13)	0.0147 (3)
H12	0.7386	1.0109	0.3164	0.018*
C13	0.81494 (16)	1.08455 (10)	0.17549 (14)	0.0169 (3)
C14	0.78385 (16)	1.13505 (10)	0.06023 (14)	0.0187 (3)
H14	0.8574	1.1609	0.0206	0.022*
C15	0.64682 (16)	1.14724 (9)	0.00445 (14)	0.0172 (3)
H15	0.6235	1.1823	-0.0727	0.021*
C16	0.54321 (16)	1.10616 (9)	0.06539 (13)	0.0146 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01421 (19)	0.0351 (2)	0.0286 (2)	-0.00330 (15)	0.00218 (15)	0.00450 (16)
O1	0.0125 (5)	0.0194 (5)	0.0187 (5)	-0.0004 (4)	0.0054 (4)	0.0023 (4)
O2	0.0113 (5)	0.0246 (6)	0.0156 (5)	0.0001 (4)	0.0013 (4)	0.0057 (4)
O3	0.0132 (5)	0.0167 (5)	0.0131 (5)	0.0012 (4)	0.0055 (4)	0.0018 (4)
O4	0.0101 (5)	0.0191 (5)	0.0155 (5)	-0.0022 (4)	0.0017 (4)	0.0035 (4)
N1	0.0124 (6)	0.0149 (6)	0.0152 (6)	-0.0011 (5)	0.0059 (4)	0.0021 (4)
N2	0.0159 (6)	0.0135 (6)	0.0129 (5)	-0.0014 (5)	0.0060 (4)	-0.0005 (4)
N3	0.0185 (6)	0.0160 (6)	0.0135 (5)	0.0028 (5)	0.0003 (5)	0.0009 (4)
C1	0.0145 (7)	0.0119 (6)	0.0134 (6)	-0.0007 (5)	0.0050 (5)	-0.0029 (5)
C2	0.0110 (7)	0.0166 (7)	0.0154 (6)	-0.0016 (5)	0.0039 (5)	-0.0022 (5)
C3	0.0122 (7)	0.0143 (6)	0.0125 (6)	0.0024 (5)	0.0009 (5)	-0.0020 (5)
C4	0.0140 (7)	0.0126 (6)	0.0110 (6)	-0.0013 (5)	0.0038 (5)	-0.0024 (5)
C5	0.0109 (6)	0.0137 (6)	0.0135 (6)	-0.0009 (5)	0.0034 (5)	-0.0038 (5)
C6	0.0134 (7)	0.0137 (6)	0.0125 (6)	0.0010 (5)	0.0014 (5)	-0.0007 (5)
C7	0.0151 (7)	0.0121 (6)	0.0131 (6)	-0.0004 (5)	0.0049 (5)	-0.0040 (5)
C8	0.0132 (7)	0.0149 (6)	0.0153 (6)	-0.0012 (5)	0.0046 (5)	-0.0039 (5)
C9	0.0153 (7)	0.0136 (6)	0.0139 (6)	0.0004 (5)	0.0029 (5)	-0.0029 (5)
C10	0.0164 (7)	0.0155 (7)	0.0167 (7)	0.0003 (6)	0.0020 (5)	-0.0032 (5)
C11	0.0173 (7)	0.0102 (6)	0.0130 (6)	-0.0005 (5)	0.0041 (5)	-0.0028 (5)
C12	0.0167 (7)	0.0134 (7)	0.0139 (6)	-0.0003 (5)	0.0024 (5)	-0.0005 (5)
C13	0.0143 (7)	0.0173 (7)	0.0193 (7)	-0.0007 (6)	0.0024 (6)	-0.0022 (5)
C14	0.0224 (8)	0.0159 (7)	0.0192 (7)	-0.0037 (6)	0.0082 (6)	-0.0005 (5)
C15	0.0255 (8)	0.0133 (6)	0.0137 (6)	-0.0004 (6)	0.0058 (6)	-0.0002 (5)
C16	0.0191 (7)	0.0122 (6)	0.0125 (6)	0.0003 (5)	0.0022 (5)	-0.0028 (5)

supplementary materials

Geometric parameters (Å, °)

C11—C13	1.7491 (15)	C2—H2	0.9500
O1—C7	1.2371 (17)	C3—C4	1.3948 (19)
O2—C3	1.3731 (16)	C4—C5	1.3919 (19)
O2—H2O	0.8400	C5—C6	1.3883 (19)
O3—C4	1.3664 (16)	C6—H6	0.9500
O3—H3O	0.8400	C8—C9	1.4350 (19)
O4—C5	1.3650 (16)	C8—H8	0.9500
O4—H4O	0.8400	C9—C10	1.383 (2)
N1—C7	1.3506 (18)	C9—C11	1.443 (2)
N1—N2	1.3927 (16)	C10—H10	0.9500
N1—H1N	0.8800	C11—C12	1.399 (2)
N2—C8	1.2884 (19)	C11—C16	1.4080 (19)
N3—C10	1.3558 (19)	C12—C13	1.378 (2)
N3—C16	1.3768 (19)	C12—H12	0.9500
N3—H3N	0.8800	C13—C14	1.400 (2)
C1—C6	1.3936 (19)	C14—C15	1.375 (2)
C1—C2	1.397 (2)	C14—H14	0.9500
C1—C7	1.4925 (19)	C15—C16	1.397 (2)
C2—C3	1.3860 (19)	C15—H15	0.9500
C3—O2—H2O	109.5	O1—C7—C1	120.52 (13)
C4—O3—H3O	109.5	N1—C7—C1	116.90 (12)
C5—O4—H4O	109.5	N2—C8—C9	122.22 (13)
C7—N1—N2	119.69 (12)	N2—C8—H8	118.9
C7—N1—H1N	120.2	C9—C8—H8	118.9
N2—N1—H1N	120.2	C10—C9—C8	123.75 (14)
C8—N2—N1	114.96 (12)	C10—C9—C11	106.32 (12)
C10—N3—C16	109.38 (12)	C8—C9—C11	129.87 (13)
C10—N3—H3N	125.3	N3—C10—C9	109.91 (13)
C16—N3—H3N	125.3	N3—C10—H10	125.0
C6—C1—C2	120.24 (13)	C9—C10—H10	125.0
C6—C1—C7	122.53 (13)	C12—C11—C16	119.31 (13)
C2—C1—C7	117.23 (13)	C12—C11—C9	134.24 (13)
C3—C2—C1	118.90 (13)	C16—C11—C9	106.41 (13)
C3—C2—H2	120.5	C13—C12—C11	117.29 (13)
C1—C2—H2	120.5	C13—C12—H12	121.4
O2—C3—C2	120.11 (12)	C11—C12—H12	121.4
O2—C3—C4	118.44 (12)	C12—C13—C14	123.35 (14)
C2—C3—C4	121.44 (13)	C12—C13—C11	118.55 (11)
O3—C4—C5	123.80 (13)	C14—C13—C11	118.07 (11)
O3—C4—C3	117.16 (12)	C15—C14—C13	119.96 (14)
C5—C4—C3	118.99 (12)	C15—C14—H14	120.0
O4—C5—C6	116.76 (12)	C13—C14—H14	120.0
O4—C5—C4	122.94 (12)	C14—C15—C16	117.53 (13)
C6—C5—C4	120.30 (13)	C14—C15—H15	121.2
C5—C6—C1	120.07 (13)	C16—C15—H15	121.2
C5—C6—H6	120.0	N3—C16—C15	129.48 (13)

C1—C6—H6	120.0	N3—C16—C11	107.98 (12)
O1—C7—N1	122.58 (13)	C15—C16—C11	122.54 (14)
C7—N1—N2—C8	-175.96 (12)	N2—C8—C9—C10	166.52 (13)
C6—C1—C2—C3	-1.2 (2)	N2—C8—C9—C11	-10.3 (2)
C7—C1—C2—C3	178.31 (12)	C16—N3—C10—C9	-0.12 (16)
C1—C2—C3—O2	-179.47 (12)	C8—C9—C10—N3	-177.25 (12)
C1—C2—C3—C4	-0.5 (2)	C11—C9—C10—N3	0.18 (15)
O2—C3—C4—O3	-1.36 (18)	C10—C9—C11—C12	177.59 (15)
C2—C3—C4—O3	179.69 (12)	C8—C9—C11—C12	-5.2 (3)
O2—C3—C4—C5	-178.98 (12)	C10—C9—C11—C16	-0.17 (15)
C2—C3—C4—C5	2.1 (2)	C8—C9—C11—C16	177.04 (14)
O3—C4—C5—O4	0.2 (2)	C16—C11—C12—C13	-1.65 (19)
C3—C4—C5—O4	177.70 (12)	C9—C11—C12—C13	-179.19 (14)
O3—C4—C5—C6	-179.32 (12)	C11—C12—C13—C14	0.7 (2)
C3—C4—C5—C6	-1.9 (2)	C11—C12—C13—C11	178.50 (10)
O4—C5—C6—C1	-179.44 (12)	C12—C13—C14—C15	0.8 (2)
C4—C5—C6—C1	0.2 (2)	C11—C13—C14—C15	-176.97 (11)
C2—C1—C6—C5	1.4 (2)	C13—C14—C15—C16	-1.3 (2)
C7—C1—C6—C5	-178.09 (12)	C10—N3—C16—C15	-179.12 (14)
N2—N1—C7—O1	2.73 (19)	C10—N3—C16—C11	0.00 (15)
N2—N1—C7—C1	-176.29 (11)	C14—C15—C16—N3	179.40 (14)
C6—C1—C7—O1	-173.65 (13)	C14—C15—C16—C11	0.4 (2)
C2—C1—C7—O1	6.83 (19)	C12—C11—C16—N3	-178.05 (12)
C6—C1—C7—N1	5.39 (19)	C9—C11—C16—N3	0.11 (15)
C2—C1—C7—N1	-174.13 (12)	C12—C11—C16—C15	1.1 (2)
N1—N2—C8—C9	-178.92 (12)	C9—C11—C16—C15	179.30 (13)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O2—H2 _o \cdots O3	0.84	2.20	2.6726 (14)	116
O3—H3 _o \cdots O1 ⁱ	0.84	1.74	2.5818 (14)	175
O4—H4 _o \cdots N2 ⁱ	0.84	2.01	2.7668 (15)	149
N1—H1 _n \cdots O2 ⁱⁱ	0.88	2.23	3.0875 (16)	163
N3—H3 _n \cdots O4 ⁱⁱⁱ	0.88	2.15	2.9518 (15)	152

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

Fig. 1

