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

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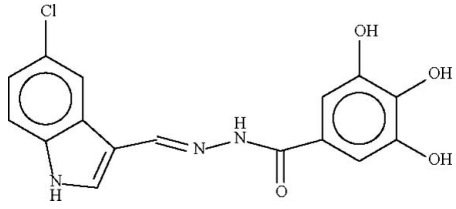
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.093; data-to-parameter ratio = 15.4.

The two aromatic parts of the title compound, $C_{16}H_{13}ClN_3O_4$, are connected through a conjugated –CH=N–NH–C(O)– fragment, giving an almost planar molecule (r.m.s. deviation 0.08 Å). In the crystal structure, adjacent molecules are linked by N—H···O and O—H···O hydrogen bonds into a three-dimensional network.

Related literature

For the isostructural $C_{16}H_{13}BrN_3O_4$ analog, see: Khaledi *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{12}ClN_3O_4$
M_r = 345.74
Monoclinic, $P2_1/n$
a = 9.6481 (2) Å
b = 15.1408 (3) Å
c = 10.2206 (2) Å
β = 98.232 (1)°
V = 1477.64 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 100 (2) K
0.32 × 0.22 × 0.12 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.914, T_{max} = 0.966
10185 measured reflections
3389 independent reflections
2907 reflections with $I > 2\sigma(I)$
R_{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
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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	2.20	2.6726 (14)	116
O3—H3o···O1^{i}	0.84	1.74	2.5818 (14)	175
O4—H4o···N2^{i}	0.84	2.01	2.7668 (15)	149
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N3—H3n···O4^{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: *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, 2009).

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

References

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supporting information

Acta Cryst. (2009). E**65**, o169 [doi:10.1107/S160053680804258X]

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

Hamid Khaledi, Hapipah Mohd Ali and Seik Weng Ng

S1. 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.

S2. 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.

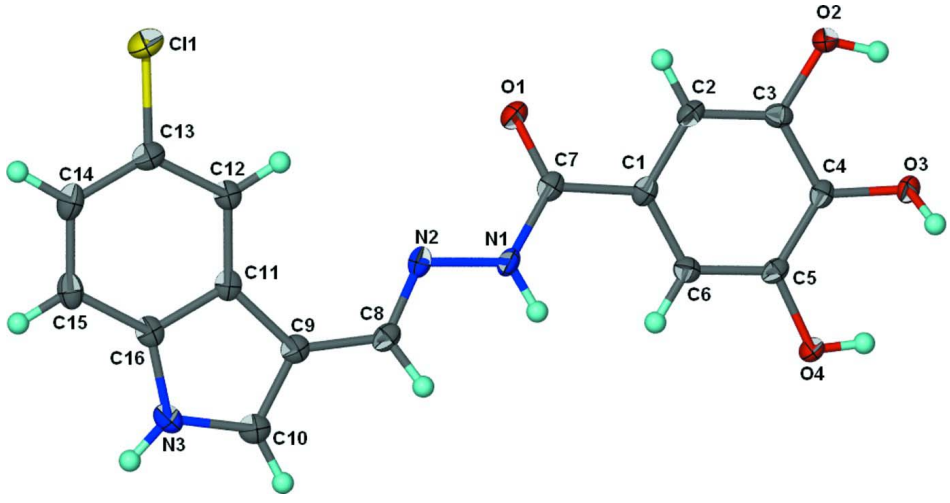

Figure 1

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.

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Crystal data

$C_{16}H_{12}ClN_3O_4$
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Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.6481 (2) Å
b = 15.1408 (3) Å
c = 10.2206 (2) Å
β = 98.232 (1)°
V = 1477.64 (5) Å^3
Z = 4
F(000) = 712
D_x = 1.554 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4113 reflections

θ = 2.4–28.2°
μ = 0.29 mm^{-1}
T = 100 K

Prism, orange
0.32 × 0.22 × 0.12 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.914, T_{max} = 0.966

10185 measured reflections
3389 independent reflections
2907 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −11→12
k = −19→19
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.06
3389 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
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.6635P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	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 (Å²)

	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)

Geometric parameters (Å, º)

Cl1—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—Cl1	118.55 (11)
O3—C4—C5	123.80 (13)	C14—C13—Cl1	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—Cl1	178.50 (10)
O4—C5—C6—C1	−179.44 (12)	C12—C13—C14—C15	0.8 (2)
C4—C5—C6—C1	0.2 (2)	Cl1—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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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O3—H3o···O1^{i}	0.84	1.74	2.5818 (14)	175
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Symmetry codes: (i) $x-1/2, -y+3/2, z+1/2$; (ii) $x-1/2, -y+3/2, z-1/2$; (iii) $-x+1/2, y+1/2, -z+1/2$.