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3,4,5-Trihydroxy-*N*′-[(1-methyl-1*H*-indol-2-yl)methylidene]benzohydrazide

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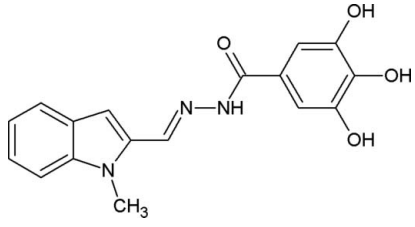
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.127; data-to-parameter ratio = 18.4.

The structure of the title compound, $C_{17}H_{15}N_3O_4$, displays intermolecular O—H···N and O—H···O hydrogen bonding between adjacent molecules. Intramolecular O—H···O hydrogen bonds also occur. The molecule is essentially planar with a deviation of 0.090 (1) Å from the best plane running through the connected ring systems.

Related literature

For related compounds see: Khaledi *et al.* (2008*a*,*b*, 2009).



Experimental

Crystal data

$C_{17}H_{15}N_3O_4$
M_r = 325.32
Monoclinic, $P2_1/n$
a = 9.0839 (2) Å
b = 13.1684 (3) Å
c = 12.4414 (3) Å
β = 104.2740 (10)°
V = 1442.30 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.49 × 0.16 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.991
10177 measured reflections
4070 independent reflections
3153 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.127
S = 0.99
4070 reflections
221 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*O*···O4^{i}	0.84	1.80	2.6119 (14)	164
O1—H1*O*···N2^{i}	0.84	2.06	2.7759 (15)	142
O3—H3*O*···O2^{ii}	0.84	2.12	2.8469 (14)	144
O1—H1*O*···O2	0.84	2.51	2.8570 (14)	106
O3—H3*O*···O2	0.84	2.31	2.7560 (14)	113

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); 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: HG2532).

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supplementary materials

Acta Cryst. (2009). E**65**, o1920 [**doi:10.1107/S1600536809027032**]

3,4,5-Trihydroxy-*N*'-[(1-methyl-1*H*-indol-2-yl)methylidene]benzohydrazide

H. Khaledi, S. M. Saharin, H. Mohd Ali, W. T. Robinson and M. A. Abdulla

Experimental

A mixture of 1-Methylindole-2-carboxaldehyde (0.80 g, 5 mmol) and 3,4,5-trihydroxybenzoylhydrazine(0.92 g, 5 mmol) in the presence of acetic acid (1 ml) was heated in ethanol (70 ml) for 6 h. The solution was then cooled and filtered to remove the unreacted hydrazine. The filtrate was poured to water (400 ml), the solid product formed were filtered off, washed with diethyl ether,and dried in an oven. Suitable crystals for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

All Hydrogen atoms were placed at calculated positions (C—H 0.95–0.98, N—H 0.88 and O—H 0.84 Å), with U(H) set to 1.2–1.5 times $U_{eq}(C,N,O)$.

Figures

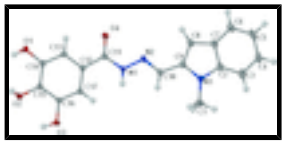


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of the title compound at 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

3,4,5-Trihydroxy-*N*'-[(1-methyl-1*H*-indol-2- yl)methylidene]benzohydrazide

Crystal data

$C_{17}H_{15}N_3O_4$	F_{000} = 680
M_r = 325.32	D_x = 1.498 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3739 reflections
a = 9.0839 (2) Å	θ = 2.3–30.4º
b = 13.1684 (3) Å	μ = 0.11 mm^{-1}
c = 12.4414 (3) Å	T = 100 K
β = 104.2740 (10)º	Block, green
V = 1442.30 (6) Å^3	0.49 × 0.16 × 0.09 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	4070 independent reflections

Radiation source: fine-focus sealed tube	3153 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 100$ K	$\theta_{max} = 30.5°$
ω scans	$\theta_{min} = 2.3°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 12$
$T_{min} = 0.948$, $T_{max} = 0.991$	$k = -18 \rightarrow 18$
10177 measured reflections	$l = -17 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.127$	$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.9156P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} < 0.001$
4070 reflections	$\Delta\rho_{max} = 0.63$ e Å^{-3}
221 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.47487 (13)	0.36671 (9)	0.05923 (10)	0.0132 (2)
H1N	0.4655	0.3333	−0.0034	0.016*
N2	0.38051 (13)	0.44784 (9)	0.06479 (10)	0.0134 (2)
N3	0.06173 (13)	0.56577 (9)	−0.13005 (10)	0.0151 (2)
O1	0.99391 (12)	0.12533 (8)	0.33141 (8)	0.0165 (2)
H1O	1.0219	0.0645	0.3313	0.025*
O2	0.98618 (11)	0.01716 (7)	0.13104 (8)	0.0149 (2)
H2O	0.9766	−0.0305	0.1737	0.022*
O3	0.77044 (12)	0.07206 (8)	−0.05744 (8)	0.0189 (2)
H3O	0.8473	0.0349	−0.0506	0.028*

O4	0.59664 (13)	0.38379 (8)	0.23996 (8)	0.0199 (2)
C1	0.04811 (18)	0.51469 (13)	−0.23489 (13)	0.0226 (3)
H1A	−0.0426	0.5393	−0.2887	0.034*
H1B	0.0393	0.4413	−0.2247	0.034*
H1C	0.1383	0.5288	−0.2623	0.034*
C2	−0.02892 (15)	0.64421 (11)	−0.11137 (12)	0.0155 (3)
C3	−0.15129 (16)	0.69271 (11)	−0.18421 (13)	0.0192 (3)
H3	−0.1834	0.6736	−0.2600	0.023*
C4	−0.22304 (17)	0.76936 (12)	−0.14091 (14)	0.0213 (3)
H4	−0.3059	0.8039	−0.1883	0.026*
C5	−0.17667 (17)	0.79760 (11)	−0.02853 (14)	0.0219 (3)
H5	−0.2291	0.8505	−0.0016	0.026*
C6	−0.05636 (17)	0.74988 (11)	0.04323 (13)	0.0195 (3)
H6	−0.0259	0.7692	0.1191	0.023*
C7	0.02010 (16)	0.67213 (11)	0.00177 (12)	0.0163 (3)
C8	0.14341 (16)	0.60759 (11)	0.05098 (12)	0.0162 (3)
H8	0.1997	0.6085	0.1263	0.019*
C9	0.16628 (15)	0.54349 (10)	−0.03077 (11)	0.0137 (3)
C10	0.27384 (15)	0.46201 (10)	−0.02389 (11)	0.0134 (3)
H10	0.2662	0.4180	−0.0856	0.016*
C11	0.58127 (15)	0.33924 (10)	0.15038 (11)	0.0129 (3)
C12	0.68091 (14)	0.25165 (10)	0.13972 (11)	0.0123 (3)
C13	0.78798 (15)	0.22437 (10)	0.23633 (11)	0.0132 (3)
H13	0.7917	0.2597	0.3035	0.016*
C14	0.88907 (15)	0.14590 (10)	0.23472 (11)	0.0128 (3)
C15	0.88441 (15)	0.09305 (10)	0.13689 (11)	0.0122 (2)
C16	0.77684 (15)	0.12083 (10)	0.04008 (11)	0.0135 (3)
C17	0.67475 (15)	0.19925 (10)	0.04105 (11)	0.0143 (3)
H17	0.6014	0.2171	−0.0249	0.017*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0140 (5)	0.0112 (5)	0.0140 (5)	0.0024 (4)	0.0029 (4)	−0.0013 (4)
N2	0.0120 (5)	0.0110 (5)	0.0173 (5)	0.0015 (4)	0.0040 (4)	0.0006 (4)
N3	0.0132 (5)	0.0141 (5)	0.0174 (6)	0.0019 (4)	0.0028 (4)	0.0011 (4)
O1	0.0180 (5)	0.0172 (5)	0.0121 (4)	0.0047 (4)	−0.0006 (4)	0.0011 (4)
O2	0.0183 (5)	0.0122 (5)	0.0157 (5)	0.0049 (4)	0.0069 (4)	0.0039 (4)
O3	0.0196 (5)	0.0214 (5)	0.0139 (5)	0.0075 (4)	0.0010 (4)	−0.0046 (4)
O4	0.0232 (5)	0.0203 (5)	0.0151 (5)	0.0068 (4)	0.0027 (4)	−0.0035 (4)
C1	0.0219 (7)	0.0248 (8)	0.0192 (7)	0.0035 (6)	0.0014 (6)	−0.0027 (6)
C2	0.0135 (6)	0.0124 (6)	0.0219 (7)	0.0001 (5)	0.0066 (5)	0.0025 (5)
C3	0.0148 (6)	0.0182 (7)	0.0247 (7)	0.0009 (5)	0.0050 (5)	0.0049 (6)
C4	0.0151 (6)	0.0168 (7)	0.0325 (8)	0.0031 (5)	0.0070 (6)	0.0071 (6)
C5	0.0185 (7)	0.0134 (6)	0.0370 (9)	0.0020 (5)	0.0128 (6)	0.0013 (6)
C6	0.0193 (7)	0.0154 (7)	0.0263 (8)	−0.0008 (5)	0.0101 (6)	−0.0018 (5)
C7	0.0149 (6)	0.0129 (6)	0.0220 (7)	−0.0004 (5)	0.0068 (5)	0.0009 (5)
C8	0.0155 (6)	0.0142 (6)	0.0197 (7)	0.0003 (5)	0.0061 (5)	0.0004 (5)

C9	0.0122 (6)	0.0123 (6)	0.0165 (6)	−0.0002 (5)	0.0034 (5)	0.0022 (5)
C10	0.0125 (6)	0.0126 (6)	0.0155 (6)	−0.0008 (5)	0.0042 (5)	−0.0007 (5)
C11	0.0134 (6)	0.0121 (6)	0.0135 (6)	0.0001 (5)	0.0042 (5)	0.0011 (5)
C12	0.0122 (6)	0.0116 (6)	0.0131 (6)	0.0007 (4)	0.0031 (5)	0.0013 (5)
C13	0.0148 (6)	0.0131 (6)	0.0120 (6)	0.0008 (5)	0.0034 (5)	−0.0004 (5)
C14	0.0131 (6)	0.0132 (6)	0.0117 (6)	0.0001 (5)	0.0022 (4)	0.0029 (5)
C15	0.0120 (6)	0.0111 (6)	0.0142 (6)	0.0012 (4)	0.0042 (5)	0.0020 (5)
C16	0.0147 (6)	0.0142 (6)	0.0120 (6)	0.0006 (5)	0.0038 (5)	−0.0016 (5)
C17	0.0142 (6)	0.0147 (6)	0.0129 (6)	0.0025 (5)	0.0013 (5)	0.0007 (5)

Geometric parameters (Å, °)

N1—C11	1.3455 (17)	C4—C5	1.407 (2)
N1—N2	1.3821 (15)	C4—H4	0.9500
N1—H1N	0.8800	C5—C6	1.380 (2)
N2—C10	1.2900 (17)	C5—H5	0.9500
N3—C2	1.3760 (18)	C6—C7	1.405 (2)
N3—C9	1.3912 (17)	C6—H6	0.9500
N3—C1	1.4455 (19)	C7—C8	1.419 (2)
O1—C14	1.3647 (16)	C8—C9	1.376 (2)
O1—H1O	0.8400	C8—H8	0.9500
O2—C15	1.3755 (16)	C9—C10	1.4394 (19)
O2—H2O	0.8400	C10—H10	0.9500
O3—C16	1.3613 (16)	C11—C12	1.4921 (18)
O3—H3O	0.8400	C12—C13	1.3941 (18)
O4—C11	1.2365 (17)	C12—C17	1.3972 (18)
C1—H1A	0.9800	C13—C14	1.3859 (18)
C1—H1B	0.9800	C13—H13	0.9500
C1—H1C	0.9800	C14—C15	1.3936 (18)
C2—C3	1.403 (2)	C15—C16	1.3995 (18)
C2—C7	1.416 (2)	C16—C17	1.3900 (19)
C3—C4	1.381 (2)	C17—H17	0.9500
C3—H3	0.9500		
C11—N1—N2	119.41 (11)	C6—C7—C8	133.41 (14)
C11—N1—H1N	120.3	C2—C7—C8	107.14 (12)
N2—N1—H1N	120.3	C9—C8—C7	107.18 (13)
C10—N2—N1	114.41 (11)	C9—C8—H8	126.4
C2—N3—C9	108.32 (12)	C7—C8—H8	126.4
C2—N3—C1	125.46 (12)	C8—C9—N3	109.41 (12)
C9—N3—C1	126.22 (12)	C8—C9—C10	129.58 (13)
C14—O1—H1O	109.5	N3—C9—C10	120.98 (12)
C15—O2—H2O	109.5	N2—C10—C9	120.95 (13)
C16—O3—H3O	109.5	N2—C10—H10	119.5
N3—C1—H1A	109.5	C9—C10—H10	119.5
N3—C1—H1B	109.5	O4—C11—N1	122.01 (12)
H1A—C1—H1B	109.5	O4—C11—C12	120.71 (12)
N3—C1—H1C	109.5	N1—C11—C12	117.28 (12)
H1A—C1—H1C	109.5	C13—C12—C17	119.91 (12)
H1B—C1—H1C	109.5	C13—C12—C11	115.60 (12)

N3—C2—C3	130.13 (14)	C17—C12—C11	124.48 (12)
N3—C2—C7	107.95 (12)	C14—C13—C12	120.23 (12)
C3—C2—C7	121.92 (13)	C14—C13—H13	119.9
C4—C3—C2	117.12 (14)	C12—C13—H13	119.9
C4—C3—H3	121.4	O1—C14—C13	117.13 (12)
C2—C3—H3	121.4	O1—C14—C15	122.35 (12)
C3—C4—C5	121.76 (14)	C13—C14—C15	120.49 (12)
C3—C4—H4	119.1	O2—C15—C14	122.20 (12)
C5—C4—H4	119.1	O2—C15—C16	118.70 (12)
C6—C5—C4	121.17 (14)	C14—C15—C16	119.04 (12)
C6—C5—H5	119.4	O3—C16—C17	118.44 (12)
C4—C5—H5	119.4	O3—C16—C15	120.73 (12)
C5—C6—C7	118.60 (15)	C17—C16—C15	120.83 (12)
C5—C6—H6	120.7	C16—C17—C12	119.49 (12)
C7—C6—H6	120.7	C16—C17—H17	120.3
C6—C7—C2	119.43 (13)	C12—C17—H17	120.3
C11—N1—N2—C10	−173.94 (12)	C8—C9—C10—N2	−9.1 (2)
C9—N3—C2—C3	−179.04 (14)	N3—C9—C10—N2	172.91 (13)
C1—N3—C2—C3	0.1 (2)	N2—N1—C11—O4	0.8 (2)
C9—N3—C2—C7	0.29 (15)	N2—N1—C11—C12	−179.57 (11)
C1—N3—C2—C7	179.39 (13)	O4—C11—C12—C13	0.73 (19)
N3—C2—C3—C4	179.14 (14)	N1—C11—C12—C13	−178.95 (12)
C7—C2—C3—C4	−0.1 (2)	O4—C11—C12—C17	−177.78 (13)
C2—C3—C4—C5	−0.4 (2)	N1—C11—C12—C17	2.5 (2)
C3—C4—C5—C6	0.3 (2)	C17—C12—C13—C14	0.5 (2)
C4—C5—C6—C7	0.2 (2)	C11—C12—C13—C14	−178.13 (12)
C5—C6—C7—C2	−0.7 (2)	C12—C13—C14—O1	177.99 (12)
C5—C6—C7—C8	−178.83 (15)	C12—C13—C14—C15	−0.4 (2)
N3—C2—C7—C6	−178.74 (13)	O1—C14—C15—O2	−0.6 (2)
C3—C2—C7—C6	0.7 (2)	C13—C14—C15—O2	177.66 (12)
N3—C2—C7—C8	−0.15 (16)	O1—C14—C15—C16	−177.82 (12)
C3—C2—C7—C8	179.24 (13)	C13—C14—C15—C16	0.4 (2)
C6—C7—C8—C9	178.27 (16)	O2—C15—C16—O3	1.6 (2)
C2—C7—C8—C9	−0.04 (16)	C14—C15—C16—O3	178.93 (12)
C7—C8—C9—N3	0.22 (16)	O2—C15—C16—C17	−177.94 (12)
C7—C8—C9—C10	−177.98 (14)	C14—C15—C16—C17	−0.6 (2)
C2—N3—C9—C8	−0.32 (16)	O3—C16—C17—C12	−178.85 (12)
C1—N3—C9—C8	−179.42 (13)	C15—C16—C17—C12	0.7 (2)
C2—N3—C9—C10	178.06 (12)	C13—C12—C17—C16	−0.6 (2)
C1—N3—C9—C10	−1.0 (2)	C11—C12—C17—C16	177.82 (13)
N1—N2—C10—C9	179.93 (12)		

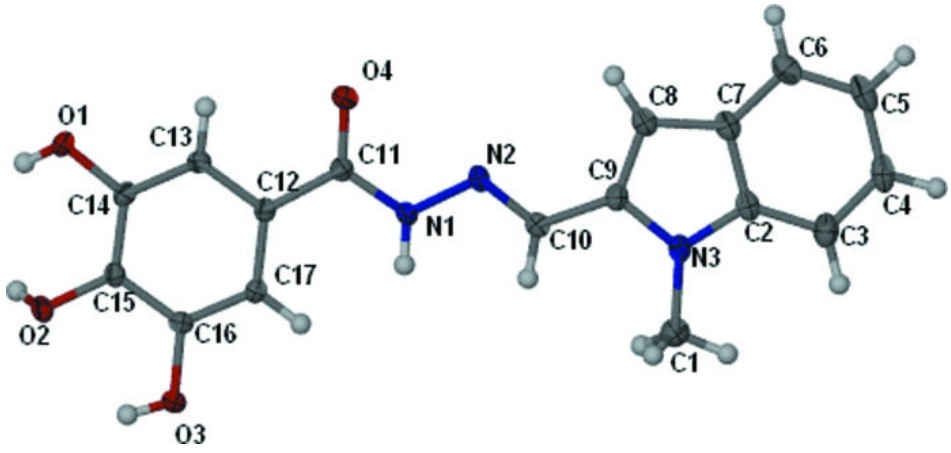
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···O4^{i}	0.84	1.80	2.6119 (14)	164
O1—H1O···N2^{i}	0.84	2.06	2.7759 (15)	142
O3—H3O···O2^{ii}	0.84	2.12	2.8469 (14)	144

O1—H1O···O2	0.84	2.51	2.8570 (14)	106
O3—H3O···O2	0.84	2.31	2.7560 (14)	113

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

Fig. 1


O4
O1
C13
C11
N2
C8
C7
C6
C5
C12
C9
C14
N1
C10
C2
C3
C4
N3
C17
C15
O2
C16
C1
O3