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 5-Methyl-1*H*-indole-3-carbaldehyde

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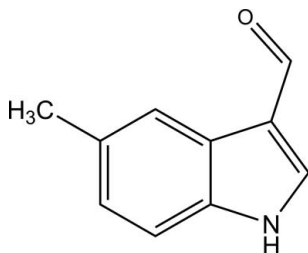
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
 R factor = 0.038; wR factor = 0.107; data-to-parameter ratio = 10.2.

The title molecule, $\text{C}_{10}\text{H}_9\text{NO}$, is almost planar with an r.m.s. deviation for all non-H atoms of 0.0115 Å. In the crystal, molecules are connected through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into chains running along [021]. The chains are further connected *via* $\text{C}-\text{H}\cdots\pi$ interactions, forming layers in the *bc* plane.

Related literature

For the structure of 1*H*-indole-3-carbaldehyde, see: Ng (2007) and for the structure of 6-bromo-1*H*-indole-3-carbaldehyde, see: Johnson *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_9\text{NO}$
 $M_r = 159.18$
 Orthorhombic, $Pca2_1$
 $a = 16.9456$ (19) Å

 $b = 5.7029$ (6) Å
 $c = 8.6333$ (9) Å
 $V = 834.31$ (15) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 296$ K
 $0.47 \times 0.15 \times 0.05$ mm

Data collection

 Bruker APEXII CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.962$, $T_{\max} = 0.996$

 5499 measured reflections
 1147 independent reflections
 717 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.107$
 $S = 0.98$
 1147 reflections
 113 parameters
 1 restraint

 H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.12$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

 C_g is the centroid of the N1/C1/C2/C3/C8 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.93 (3)	1.90 (3)	2.818 (3)	169 (3)
$\text{C9}-\text{H9}\cdots\text{C}_g^{\text{ii}}$	0.93	2.91	3.312 (3)	107

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

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: SHELXL97 and publCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2498).

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supporting information

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5-Methyl-1*H*-indole-3-carbaldehyde

Sharifah Shafiqah Ismail, Hamid Khaledi and Hapipah Mohd Ali

S1. Comment

The structure of the title compound is isomorphous with that of 1*H*-indole-3-carbaldehyde (Ng, 2007). The planar molecules are connected *via* N—H \cdots O hydrogen bonds (Table 1) into chains in the [021] direction. The chains are further linked through C—H \cdots π interactions (Table 1) to form layers in the *bc* plane. The structure of 6-bromo-1*H*-indole-3-carbaldehyde (Johnson *et al.*, 2009) exhibits similar N—H \cdots O bonded chains, however, further supramolecular aggregation by Br-involved interactions is observed.

S2. Experimental

The title crystals were obtained by slow evaporation of an ethanolic solution of the commercially available 5-methyl-indole-3-carboxaldehyde at room temperature.

S3. Refinement

The C-bound hydrogen atoms were located in calculated positions and refined in a riding mode with C—H distances of 0.93 (C_{sp^2}) and 0.96 (C_{methyl}) Å. The N-bound H atom was found in a difference Fourier map and refined freely. For all hydrogen atoms, U_{iso} were set to 1.2–1.5 U_{eq} (carrier atom). In the absence of significant anomalous scattering effects Friedel pairs were merged.

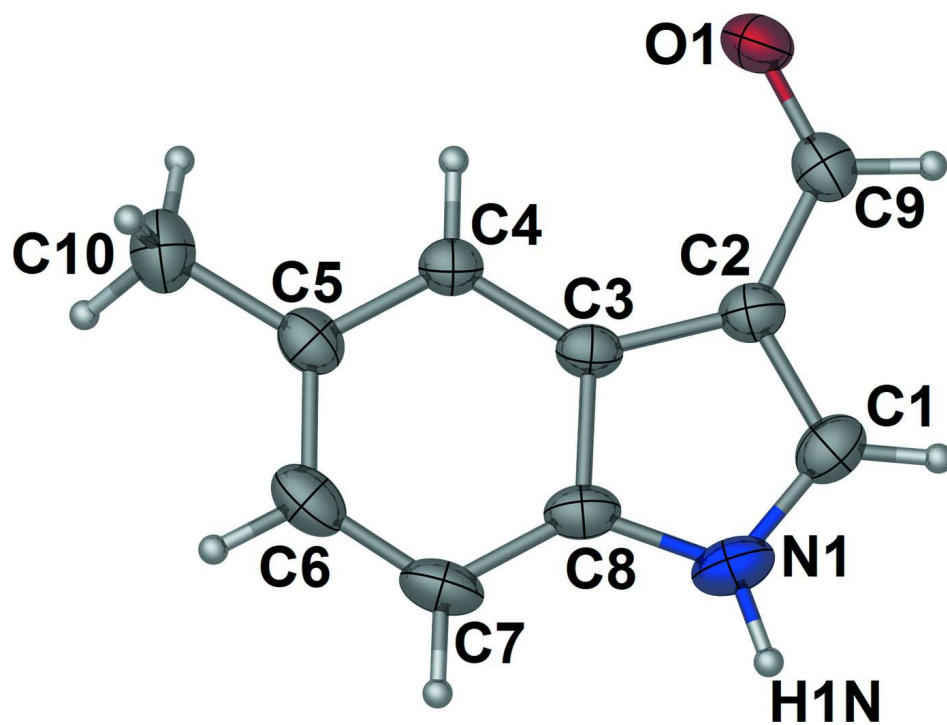


Figure 1

Molecular structure of the title compound showing thermal ellipsoids at the 30% probability level. Hydrogen spheres are drawn with an arbitrary radius.

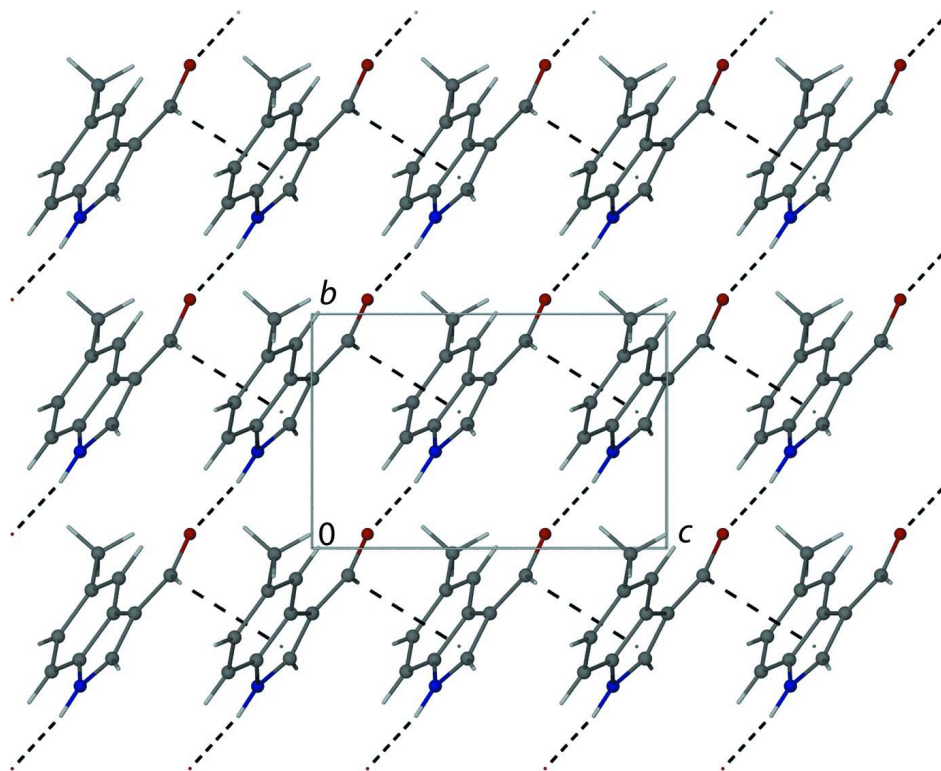


Figure 2

Crystal packing view looking down the a axis, thus showing the two-dimensional-supramolecular structure formed by N—H \cdots O and C—H \cdots π interactions (dashed lines).

5-Methyl-1H-indole-3-carbaldehyde

Crystal data

$C_{10}H_9NO$

$M_r = 159.18$

Orthorhombic, $Pca2_1$

Hall symbol: P 2c -2ac

$a = 16.9456$ (19) Å

$b = 5.7029$ (6) Å

$c = 8.6333$ (9) Å

$V = 834.31$ (15) Å³

$Z = 4$

$F(000) = 336$

$D_x = 1.267$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1172 reflections

$\theta = 2.4$ – 22.1°

$\mu = 0.08$ mm⁻¹

$T = 296$ K

Lath, yellow

$0.47 \times 0.15 \times 0.05$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.962$, $T_{\max} = 0.996$

5499 measured reflections

1147 independent reflections

717 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 28.8^\circ$, $\theta_{\min} = 2.4^\circ$

$h = -22 \rightarrow 13$

$k = -7 \rightarrow 7$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.107$
 $S = 0.98$
 1147 reflections
 113 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0596P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.12 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$

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.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.27204 (11)	1.0610 (3)	0.6573 (2)	0.0742 (6)
N1	0.29282 (16)	0.4126 (4)	0.3481 (3)	0.0760 (7)
H1N	0.2766 (15)	0.285 (5)	0.290 (5)	0.091*
C1	0.2435 (2)	0.5303 (4)	0.4384 (3)	0.0717 (8)
H1	0.1913	0.4879	0.4563	0.086*
C2	0.27969 (14)	0.7233 (4)	0.5018 (3)	0.0587 (6)
C3	0.35916 (14)	0.7227 (4)	0.4414 (3)	0.0535 (6)
C4	0.42432 (15)	0.8710 (4)	0.4561 (3)	0.0554 (6)
H4	0.4212	1.0036	0.5185	0.066*
C5	0.49316 (16)	0.8208 (4)	0.3782 (3)	0.0638 (7)
C6	0.49673 (18)	0.6225 (5)	0.2839 (4)	0.0785 (8)
H6	0.5434	0.5904	0.2311	0.094*
C7	0.4341 (2)	0.4736 (5)	0.2661 (3)	0.0766 (8)
H7	0.4377	0.3422	0.2026	0.092*
C8	0.36502 (18)	0.5241 (4)	0.3453 (3)	0.0626 (7)
C9	0.24192 (16)	0.8851 (4)	0.6026 (3)	0.0640 (6)
H9	0.1897	0.8544	0.6292	0.077*
C10	0.56347 (18)	0.9773 (6)	0.3966 (4)	0.0860 (10)
H10A	0.5654	1.0866	0.3121	0.129*
H10B	0.6107	0.8842	0.3969	0.129*
H10C	0.5595	1.0615	0.4926	0.129*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0843 (15)	0.0640 (9)	0.0743 (12)	0.0106 (9)	0.0086 (10)	-0.0101 (9)
N1	0.109 (2)	0.0565 (10)	0.0622 (13)	-0.0129 (12)	-0.0146 (15)	-0.0053 (11)
C1	0.0820 (19)	0.0660 (13)	0.0671 (17)	-0.0143 (15)	-0.0109 (17)	0.0081 (14)
C2	0.0722 (18)	0.0533 (11)	0.0508 (12)	-0.0012 (11)	-0.0030 (12)	0.0033 (10)
C3	0.0668 (17)	0.0499 (10)	0.0440 (11)	0.0056 (10)	-0.0070 (11)	-0.0013 (10)
C4	0.0639 (16)	0.0528 (10)	0.0495 (12)	0.0026 (11)	-0.0052 (12)	-0.0003 (10)
C5	0.0640 (18)	0.0699 (14)	0.0575 (14)	0.0141 (12)	-0.0022 (13)	0.0073 (13)
C6	0.081 (2)	0.0869 (18)	0.0678 (16)	0.0295 (15)	0.0074 (16)	0.0032 (15)
C7	0.105 (2)	0.0657 (14)	0.0590 (16)	0.0237 (16)	0.0008 (17)	-0.0133 (12)
C8	0.089 (2)	0.0488 (10)	0.0502 (13)	0.0051 (12)	-0.0112 (14)	-0.0021 (11)
C9	0.0662 (17)	0.0683 (13)	0.0574 (14)	0.0083 (14)	0.0013 (13)	0.0124 (13)
C10	0.068 (2)	0.102 (2)	0.088 (2)	0.0008 (17)	0.0032 (16)	0.0136 (17)

Geometric parameters (Å, °)

O1—C9	1.221 (3)	C4—H4	0.9300
N1—C1	1.326 (4)	C5—C6	1.395 (4)
N1—C8	1.379 (4)	C5—C10	1.497 (4)
N1—H1N	0.93 (3)	C6—C7	1.368 (4)
C1—C2	1.374 (3)	C6—H6	0.9300
C1—H1	0.9300	C7—C8	1.385 (4)
C2—C9	1.421 (3)	C7—H7	0.9300
C2—C3	1.444 (3)	C9—H9	0.9300
C3—C4	1.397 (3)	C10—H10A	0.9600
C3—C8	1.408 (3)	C10—H10B	0.9600
C4—C5	1.377 (3)	C10—H10C	0.9600
C1—N1—C8	109.7 (2)	C7—C6—C5	122.3 (3)
C1—N1—H1N	121.9 (18)	C7—C6—H6	118.8
C8—N1—H1N	128.1 (18)	C5—C6—H6	118.8
N1—C1—C2	111.0 (3)	C6—C7—C8	118.1 (2)
N1—C1—H1	124.5	C6—C7—H7	121.0
C2—C1—H1	124.5	C8—C7—H7	121.0
C1—C2—C9	124.3 (3)	N1—C8—C7	131.4 (2)
C1—C2—C3	105.7 (2)	N1—C8—C3	107.3 (2)
C9—C2—C3	130.0 (2)	C7—C8—C3	121.2 (3)
C4—C3—C8	119.0 (2)	O1—C9—C2	125.6 (3)
C4—C3—C2	134.7 (2)	O1—C9—H9	117.2
C8—C3—C2	106.3 (2)	C2—C9—H9	117.2
C5—C4—C3	120.0 (2)	C5—C10—H10A	109.5
C5—C4—H4	120.0	C5—C10—H10B	109.5
C3—C4—H4	120.0	H10A—C10—H10B	109.5
C4—C5—C6	119.4 (3)	C5—C10—H10C	109.5
C4—C5—C10	119.9 (2)	H10A—C10—H10C	109.5
C6—C5—C10	120.7 (3)	H10B—C10—H10C	109.5

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the N1/C1/C2/C3/C8 ring.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O1 ⁱ	0.93 (3)	1.90 (3)	2.818 (3)	169 (3)
C9—H9···Cg ⁱⁱ	0.93	2.91	3.312 (3)	107

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