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Low-temperature redetermination of 1*H*-indole-3-carbaldehyde

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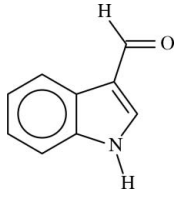
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.096; data-to-parameter ratio = 8.5.

This high precision study has confirmed the previous structure [Golubev & Kondrashev (1984). *Zh. Strukt. Khim.* **25**, 145–149] of the title compound, C_9H_7NO, and the H atoms have been located. In the crystal structure, molecules are linked by an N—H···O hydrogen bond [N···O = 2.826 (2) Å] into a helical chain running along the polar c axis of the orthorhombic unit cell.

Related literature

For the previous structure determination, see: Golubev & Kondrashev (1984).



Experimental

Crystal data

C_9H_7NO
M_r = 145.16
Orthorhombic, $Pca2_1$
a = 13.9016 (1) Å
b = 5.8655 (1) Å
c = 8.5928 (1) Å
V = 700.66 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.40 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: none
11794 measured reflections
1086 independent reflections
966 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.10
1086 reflections
128 parameters
1 restraint
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.95 (3)	1.91 (3)	2.826 (2)	161 (3)

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

Data collection: *APEXII* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2388).

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supplementary materials

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Comment

The crystal structure of the title compound, (I), indole-3-carboxaldehyde was refined to R(F) = 0.053 from room-temperature diffraction measurements (Golubev & Kondrashev, 1984). This re-refinement represents an improvement; all H atoms were located and refined. In the crystal structure, the molecules are linked by an N—H···O hydrogen bond (Table 1) to form a helical C(6) chain that propagates along the c-axis.

Experimental

Commercially-available indole-3-carboxaldehyde was recrystallized from ethanol to yield crystals of (I).

Refinement

Anomalous dispersion was negligible and Friedel pairs were merged before refinement. All the H atoms were located in difference maps and their positions and U_{iso} values were freely refined.

Figures

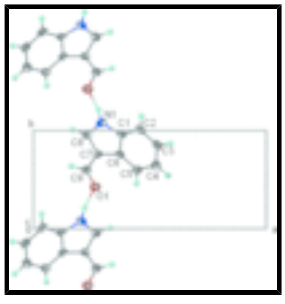

Fig. 1. View of a fragment of a chain of molecules of (I) connected by hydrogen bonds (dashed lines). Displacement ellipsoids are drawn at the 70% probability level, and H atoms are shown as spheres of arbitrary radius.

1*H*-indole-3-carbaldehyde

Crystal data

C_9H_7NO	F_{000} = 304
M_r = 145.16	D_x = 1.376 Mg m^{-3}
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 4178 reflections
a = 13.9016 (1) Å	θ = 2.9–29.8º
b = 5.8655 (1) Å	μ = 0.09 mm^{-1}
c = 8.5928 (1) Å	*T* = 173 (2) K

V = 700.66 (2) Å^3	Needle, colorless
Z = 4	0.40 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer	966 reflections with $I > 2\sigma(I)$
Radiation source: medium-focus sealed tube	R_{int} = 0.041
Monochromator: graphite	θ_{max} = 30.0º
T = 173(2) K	θ_{min} = 2.9º
φ and ω scans	h = −19→19
Absorption correction: none	k = −8→8
11794 measured reflections	l = −12→12
1086 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.036	All H-atom parameters refined
$wR(F^2)$ = 0.096	$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.0666P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max}$ = 0.001
1086 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
128 parameters	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.26655 (11)	0.4245 (2)	0.49999 (18)	0.0281 (3)
N1	0.29363 (13)	1.0779 (3)	0.2067 (2)	0.0246 (4)
C1	0.37908 (13)	0.9581 (3)	0.1919 (2)	0.0203 (4)
C2	0.45965 (15)	1.0078 (3)	0.1013 (2)	0.0244 (4)
C3	0.53265 (15)	0.8489 (4)	0.0993 (2)	0.0257 (4)
C4	0.52637 (14)	0.6458 (3)	0.1862 (2)	0.0258 (4)
C5	0.44695 (14)	0.5987 (3)	0.2777 (2)	0.0216 (4)
C6	0.37122 (13)	0.7567 (3)	0.28066 (19)	0.0190 (3)
C7	0.27630 (14)	0.7600 (3)	0.3492 (2)	0.0201 (4)
C8	0.23266 (15)	0.9593 (3)	0.2984 (2)	0.0244 (4)
C9	0.22880 (14)	0.5972 (3)	0.4472 (2)	0.0219 (4)
H1	0.281 (2)	1.216 (5)	0.153 (4)	0.047 (8)*
H2	0.4644 (19)	1.148 (4)	0.042 (3)	0.028 (7)*
H3	0.590 (2)	0.870 (5)	0.027 (4)	0.044 (9)*
H4	0.5805 (19)	0.540 (5)	0.183 (4)	0.037 (7)*

H5	0.4432 (17)	0.461 (4)	0.334 (3)	0.020 (6)*
H8	0.169 (2)	1.007 (5)	0.316 (4)	0.036 (7)*
H9	0.1626 (17)	0.638 (4)	0.466 (3)	0.022 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0332 (8)	0.0218 (6)	0.0292 (7)	0.0000 (6)	0.0022 (6)	0.0071 (6)
N1	0.0322 (9)	0.0182 (7)	0.0233 (8)	0.0048 (6)	0.0001 (7)	0.0052 (6)
C1	0.0276 (9)	0.0155 (7)	0.0179 (7)	−0.0005 (6)	−0.0032 (7)	0.0002 (7)
C2	0.0330 (10)	0.0197 (9)	0.0204 (8)	−0.0069 (7)	−0.0017 (8)	0.0016 (7)
C3	0.0234 (9)	0.0307 (10)	0.0231 (8)	−0.0064 (7)	0.0010 (7)	−0.0013 (8)
C4	0.0249 (9)	0.0262 (9)	0.0262 (9)	0.0017 (7)	−0.0009 (8)	0.0003 (8)
C5	0.0258 (9)	0.0187 (8)	0.0203 (7)	0.0005 (7)	−0.0028 (7)	0.0016 (7)
C6	0.0253 (8)	0.0170 (7)	0.0147 (7)	−0.0022 (6)	−0.0026 (7)	0.0002 (6)
C7	0.0254 (9)	0.0179 (7)	0.0169 (7)	0.0009 (6)	−0.0006 (6)	0.0016 (6)
C8	0.0290 (10)	0.0226 (8)	0.0217 (8)	0.0050 (7)	0.0007 (7)	0.0023 (7)
C9	0.0257 (10)	0.0220 (8)	0.0182 (8)	−0.0001 (7)	0.0028 (7)	−0.0020 (7)

Geometric parameters (Å, °)

O1—C9	1.228 (2)	C4—C5	1.383 (3)
N1—C8	1.350 (3)	C4—H4	0.98 (3)
N1—C1	1.386 (2)	C5—C6	1.403 (3)
N1—H1	0.95 (3)	C5—H5	0.94 (2)
C1—C2	1.395 (3)	C6—C7	1.445 (3)
C1—C6	1.410 (2)	C7—C8	1.388 (3)
C2—C3	1.378 (3)	C7—C9	1.434 (3)
C2—H2	0.97 (3)	C8—H8	0.94 (3)
C3—C4	1.409 (3)	C9—H9	0.96 (2)
C3—H3	1.02 (3)		
C8—N1—C1	109.28 (16)	C4—C5—C6	118.50 (17)
C8—N1—H1	127.1 (19)	C4—C5—H5	120.5 (15)
C1—N1—H1	123.5 (19)	C6—C5—H5	121.0 (15)
N1—C1—C2	129.28 (17)	C5—C6—C1	119.06 (17)
N1—C1—C6	108.00 (16)	C5—C6—C7	134.52 (16)
C2—C1—C6	122.63 (17)	C1—C6—C7	106.27 (15)
C3—C2—C1	117.20 (17)	C8—C7—C9	122.99 (18)
C3—C2—H2	121.1 (16)	C8—C7—C6	106.37 (16)
C1—C2—H2	121.7 (16)	C9—C7—C6	130.62 (17)
C2—C3—C4	121.29 (19)	N1—C8—C7	110.08 (18)
C2—C3—H3	120.1 (17)	N1—C8—H8	122.1 (18)
C4—C3—H3	118.4 (17)	C7—C8—H8	127.6 (18)
C5—C4—C3	121.32 (19)	O1—C9—C7	124.71 (18)
C5—C4—H4	120.4 (17)	O1—C9—H9	123.4 (15)
C3—C4—H4	118.3 (17)	C7—C9—H9	111.9 (15)
C8—N1—C1—C2	175.59 (18)	N1—C1—C6—C7	0.35 (18)
C8—N1—C1—C6	−0.8 (2)	C2—C1—C6—C7	−176.38 (17)

N1—C1—C2—C3	−175.09 (19)	C5—C6—C7—C8	−174.9 (2)
C6—C1—C2—C3	0.9 (3)	C1—C6—C7—C8	0.25 (19)
C1—C2—C3—C4	−0.5 (3)	C5—C6—C7—C9	3.4 (3)
C2—C3—C4—C5	−0.4 (3)	C1—C6—C7—C9	178.53 (19)
C3—C4—C5—C6	1.0 (3)	C1—N1—C8—C7	1.0 (2)
C4—C5—C6—C1	−0.6 (3)	C9—C7—C8—N1	−179.22 (18)
C4—C5—C6—C7	174.08 (19)	C6—C7—C8—N1	−0.8 (2)
N1—C1—C6—C5	176.42 (17)	C8—C7—C9—O1	−176.6 (2)
C2—C1—C6—C5	−0.3 (2)	C6—C7—C9—O1	5.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.95 (3)	1.91 (3)	2.826 (2)	161 (3)

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

Fig. 1