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1-Methyl-3-(1-methyl-2,3-dihydro-1*H*-indol-2-yl)-1*H*-indole

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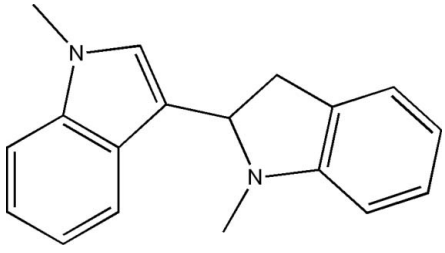
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.063; wR factor = 0.215; data-to-parameter ratio = 18.0.

The title compound, $C_{18}H_{18}N_2$, was synthesized by the dimerization of *N*-methylindole in the presence of iodine as a catalyst. In the crystal structure, the dihedral angle between the indole and indoline ring systems is 86.30 (3)°.

Related literature

For related literature on indole derivatives, see: Farhanullah *et al.* (2004); Noland & Hammer (1960); Sundberg (1996); Wu *et al.* (1972).



Experimental

Crystal data

$C_{18}H_{18}N_2$
M_r = 262.34
Triclinic, $P\overline{1}$
a = 7.3843 (15) Å
b = 10.282 (2) Å
c = 10.757 (2) Å
α = 65.54 (3)°
β = 75.14 (3)°
γ = 84.22 (3)°
V = 718.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.51 × 0.49 × 0.23 mm

Data collection

Bruker SMART CCD APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.984
7121 measured reflections
3267 independent reflections
2203 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.215
S = 1.09
3267 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2083).

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supplementary materials

Acta Cryst. (2007). E**63**, o3229 [**doi:10.1107/S1600536807028243**]

1-Methyl-3-(1-methyl-2,3-dihydro-1*H*-indol-2-yl)-1*H*-indole

A. Chen, J.-W. Zou and W.-N. Zhao

Comment

Indole has potentially reactive sites for a variety of chemical reactions to generate molecular diversity (Farhanullah *et al.*, 2004). Indole derivatives, either natural or synthetic products, have been widely studied because of their therapeutic importance (Sundberg, 1996). Indoles are known to dimerize and trimerize in acidic media (Noland *et al.*, 1960), and the derivatives of the indole dimer, *i.e.* a series of 1-imidoyl-2-(2- and 3-indolyl)indolines, are found to present potential diuretic activity (Wu *et al.*, 1972). The title compound (**I**) was synthesized by dimerization of *N*-methylindole in the presence of iodine as a catalyst. Herein we report the crystal structure of (**I**).

The molecular structure of (**I**) is shown in Fig.1. A l l bond distances and angles are normal. The length of the bond linking the indole and the indoline rings (C2—C10) is 1.486 (3) Å. The indoline ring adopts an envelope conformation with atom C10 deviating from the N2/C11/C12/C17 plane by 0.501 (3) Å. The indole ring and the indoline ring are approximately perpendicular, and the dihedral angle between them is 86.30 (3)°.

Experimental

A mixture of *N*-methylindole (10 mmol) and I_2 (1 mmol) was stirred in acetonitrile (30 ml) at room temperature for a few s. After completion of the reaction (TLC, < 1 min), the mixture was treated with aqueous $Na_2S_2O_3$ solution (5%, 30 ml). The product was extracted with ethyl acetate (3 × 30 ml). The combined organic layer was dried with anhydrous sodium sulfate, and purified by column chromatography (ethyl acetate:petroleum ether = 1:20) to afford the title compound (85% yield). Recrystallization by slow evaporation of a methanol solution was carried out to obtain good, diffraction quality crystals.

Refinement

All H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with C—H = 0.93, 0.96, 0.97 or 0.98 Å and U_{iso}(H) = 1.2 or 1.5 U_{eq}(C).

Figures

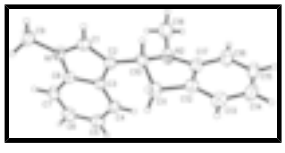

Fig. 1. A perspective view of (**I**) with atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

1-Methyl-3-(1-methyl-2,3-dihydro-1*H*-indol-2-yl)-1*H*-indole

Crystal data

$C_{18}H_{18}N_2$	$Z = 2$
$M_r = 262.34$	$F_{000} = 280$
Triclinic, $P\overline{1}$	$D_x = 1.212$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.3843$ (15) Å	Cell parameters from 3267 reflections
$b = 10.282$ (2) Å	$\theta = 3.1$–27.5º
$c = 10.757$ (2) Å	$\mu = 0.07$ mm^{-1}
$\alpha = 65.54$ (3)º	$T = 298$ (2) K
$\beta = 75.14$ (3)º	Block, colorless
$\gamma = 84.22$ (3)º	0.51 × 0.49 × 0.23 mm
$V = 718.6$ (3) Å^3	

Data collection

Bruker SMART CCD APEXII diffractometer	3267 independent reflections
Radiation source: fine-focus sealed tube	2203 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.017$
Detector resolution: 8.40 pixels mm^{-1}	$\theta_{max} = 27.5$º
$T = 298(2)$ K	$\theta_{min} = 3.1$º
ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -13 \rightarrow 13$
$T_{min} = 0.964$, $T_{max} = 0.984$	$l = -13 \rightarrow 13$
7121 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.063$	H-atom parameters constrained
$wR(F^2) = 0.215$	$w = 1/[\sigma^2(F_o^2) + (0.1209P)^2 + 0.0915P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
3267 reflections	$\Delta\rho_{max} = 0.58$ e Å^{-3}
181 parameters	$\Delta\rho_{min} = -0.29$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.0594 (2)	0.54541 (17)	0.62999 (18)	0.0532 (5)
N2	−0.2976 (3)	0.2855 (2)	1.07571 (19)	0.0608 (5)
C1	−0.1060 (3)	0.5440 (2)	0.7237 (2)	0.0528 (5)
H1A	−0.1762	0.6248	0.7220	0.063*
C2	−0.1544 (3)	0.4082 (2)	0.8201 (2)	0.0478 (5)
C3	−0.0081 (3)	0.3184 (2)	0.78479 (19)	0.0441 (5)
C4	0.0237 (3)	0.1719 (2)	0.8403 (2)	0.0527 (5)
H4A	−0.0607	0.1108	0.9186	0.063*
C5	0.1830 (3)	0.1188 (2)	0.7771 (3)	0.0644 (6)
H5A	0.2053	0.0210	0.8135	0.077*
C6	0.3100 (3)	0.2088 (3)	0.6604 (3)	0.0678 (7)
H6A	0.4167	0.1700	0.6209	0.081*
C7	0.2826 (3)	0.3533 (3)	0.6016 (2)	0.0586 (6)
H7A	0.3671	0.4129	0.5221	0.070*
C8	0.1232 (3)	0.4075 (2)	0.66545 (19)	0.0457 (5)
C9	0.1532 (4)	0.6710 (2)	0.5147 (2)	0.0681 (7)
H9A	0.0801	0.7542	0.5128	0.102*
H9B	0.1671	0.6616	0.4276	0.102*
H9C	0.2746	0.6805	0.5273	0.102*
C10	−0.3284 (3)	0.3675 (2)	0.9342 (2)	0.0514 (5)
H10A	−0.3935	0.4557	0.9334	0.062*
C11	−0.4692 (4)	0.2706 (3)	0.9273 (3)	0.0717 (7)
H11A	−0.5534	0.3262	0.8688	0.086*
H11B	−0.4053	0.2011	0.8921	0.086*
C12	−0.5719 (3)	0.1998 (2)	1.0790 (2)	0.0596 (6)
C13	−0.7391 (4)	0.1275 (3)	1.1416 (3)	0.0749 (7)
H13A	−0.8123	0.1184	1.0870	0.090*
C14	−0.7986 (4)	0.0678 (3)	1.2871 (3)	0.0731 (7)
H14A	−0.9110	0.0172	1.3301	0.088*
C15	−0.6936 (3)	0.0830 (2)	1.3672 (3)	0.0687 (7)
H15A	−0.7364	0.0435	1.4645	0.082*
C16	−0.5242 (3)	0.1559 (2)	1.3072 (2)	0.0622 (6)
H16A	−0.4531	0.1665	1.3624	0.075*

C17	−0.4639 (3)	0.2130 (2)	1.1609 (2)	0.0554 (6)
C18	−0.1963 (4)	0.3514 (3)	1.1257 (3)	0.0840 (8)
H18A	−0.1844	0.2871	1.2180	0.126*
H18B	−0.2603	0.4360	1.1298	0.126*
H18C	−0.0740	0.3767	1.0638	0.126*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0550 (10)	0.0429 (9)	0.0495 (9)	−0.0051 (7)	−0.0071 (8)	−0.0087 (7)
N2	0.0512 (10)	0.0688 (12)	0.0529 (10)	0.0022 (9)	−0.0069 (8)	−0.0189 (9)
C1	0.0551 (12)	0.0434 (10)	0.0558 (11)	0.0070 (9)	−0.0126 (9)	−0.0178 (9)
C2	0.0487 (11)	0.0434 (10)	0.0451 (10)	0.0047 (8)	−0.0064 (8)	−0.0156 (8)
C3	0.0453 (10)	0.0429 (10)	0.0402 (9)	0.0012 (8)	−0.0059 (8)	−0.0156 (8)
C4	0.0558 (12)	0.0440 (10)	0.0503 (11)	0.0023 (9)	−0.0062 (9)	−0.0155 (9)
C5	0.0671 (14)	0.0476 (12)	0.0725 (15)	0.0120 (10)	−0.0087 (12)	−0.0255 (11)
C6	0.0541 (13)	0.0698 (15)	0.0773 (15)	0.0100 (11)	−0.0001 (11)	−0.0388 (13)
C7	0.0468 (11)	0.0680 (14)	0.0539 (12)	−0.0071 (10)	0.0031 (9)	−0.0247 (10)
C8	0.0456 (10)	0.0462 (10)	0.0426 (9)	−0.0014 (8)	−0.0082 (8)	−0.0163 (8)
C9	0.0785 (16)	0.0528 (13)	0.0565 (13)	−0.0198 (11)	−0.0123 (12)	−0.0036 (10)
C10	0.0492 (11)	0.0502 (11)	0.0475 (10)	0.0091 (9)	−0.0044 (9)	−0.0187 (9)
C11	0.0630 (14)	0.0657 (15)	0.0731 (15)	−0.0067 (12)	0.0096 (12)	−0.0286 (12)
C12	0.0640 (13)	0.0521 (12)	0.0594 (12)	0.0009 (10)	−0.0056 (11)	−0.0247 (10)
C13	0.0754 (16)	0.0698 (16)	0.0752 (16)	−0.0182 (13)	−0.0058 (14)	−0.0278 (13)
C14	0.0671 (15)	0.0553 (13)	0.0749 (16)	−0.0123 (11)	0.0024 (13)	−0.0132 (12)
C15	0.0663 (15)	0.0488 (12)	0.0572 (13)	0.0060 (11)	0.0047 (11)	−0.0011 (10)
C16	0.0575 (13)	0.0578 (12)	0.0536 (12)	0.0109 (10)	−0.0093 (10)	−0.0099 (10)
C17	0.0459 (11)	0.0416 (10)	0.0581 (12)	0.0104 (8)	0.0020 (9)	−0.0108 (9)
C18	0.0743 (17)	0.102 (2)	0.0733 (17)	−0.0125 (16)	−0.0119 (14)	−0.0337 (16)

Geometric parameters (Å, °)

N1—C1	1.367 (3)	C9—H9B	0.9600
N1—C8	1.374 (3)	C9—H9C	0.9600
N1—C9	1.449 (3)	C10—C11	1.546 (3)
N2—C18	1.384 (3)	C10—H10A	0.9800
N2—C17	1.395 (3)	C11—C12	1.504 (3)
N2—C10	1.468 (3)	C11—H11A	0.9700
C1—C2	1.365 (3)	C11—H11B	0.9700
C1—H1A	0.9300	C12—C13	1.369 (3)
C2—C3	1.430 (3)	C12—C17	1.383 (3)
C2—C10	1.486 (3)	C13—C14	1.388 (4)
C3—C4	1.391 (3)	C13—H13A	0.9300
C3—C8	1.412 (3)	C14—C15	1.359 (4)
C4—C5	1.383 (3)	C14—H14A	0.9300
C4—H4A	0.9300	C15—C16	1.385 (3)
C5—C6	1.386 (3)	C15—H15A	0.9300
C5—H5A	0.9300	C16—C17	1.396 (3)
C6—C7	1.370 (3)	C16—H16A	0.9300

C6—H6A	0.9300	C18—H18A	0.9600
C7—C8	1.392 (3)	C18—H18B	0.9600
C7—H7A	0.9300	C18—H18C	0.9600
C9—H9A	0.9600		
C1—N1—C8	108.25 (16)	N2—C10—C2	114.55 (17)
C1—N1—C9	125.9 (2)	N2—C10—C11	101.59 (18)
C8—N1—C9	125.8 (2)	C2—C10—C11	116.85 (18)
C18—N2—C17	120.3 (2)	N2—C10—H10A	107.8
C18—N2—C10	117.3 (2)	C2—C10—H10A	107.8
C17—N2—C10	107.50 (18)	C11—C10—H10A	107.8
C2—C1—N1	111.00 (19)	C12—C11—C10	101.8 (2)
C2—C1—H1A	124.5	C12—C11—H11A	111.4
N1—C1—H1A	124.5	C10—C11—H11A	111.4
C1—C2—C3	105.94 (17)	C12—C11—H11B	111.4
C1—C2—C10	125.18 (19)	C10—C11—H11B	111.4
C3—C2—C10	128.86 (18)	H11A—C11—H11B	109.3
C4—C3—C8	118.78 (18)	C13—C12—C17	119.9 (2)
C4—C3—C2	134.08 (18)	C13—C12—C11	131.8 (3)
C8—C3—C2	107.13 (17)	C17—C12—C11	108.3 (2)
C5—C4—C3	118.9 (2)	C12—C13—C14	119.6 (3)
C5—C4—H4A	120.5	C12—C13—H13A	120.2
C3—C4—H4A	120.5	C14—C13—H13A	120.2
C4—C5—C6	121.1 (2)	C15—C14—C13	120.4 (2)
C4—C5—H5A	119.4	C15—C14—H14A	119.8
C6—C5—H5A	119.4	C13—C14—H14A	119.8
C7—C6—C5	121.7 (2)	C14—C15—C16	121.5 (2)
C7—C6—H6A	119.1	C14—C15—H15A	119.2
C5—C6—H6A	119.1	C16—C15—H15A	119.2
C6—C7—C8	117.4 (2)	C15—C16—C17	117.6 (2)
C6—C7—H7A	121.3	C15—C16—H16A	121.2
C8—C7—H7A	121.3	C17—C16—H16A	121.2
N1—C8—C7	130.23 (19)	C12—C17—N2	110.0 (2)
N1—C8—C3	107.68 (17)	C12—C17—C16	121.0 (2)
C7—C8—C3	122.08 (19)	N2—C17—C16	128.9 (2)
N1—C9—H9A	109.5	N2—C18—H18A	109.5
N1—C9—H9B	109.5	N2—C18—H18B	109.5
H9A—C9—H9B	109.5	H18A—C18—H18B	109.5
N1—C9—H9C	109.5	N2—C18—H18C	109.5
H9A—C9—H9C	109.5	H18A—C18—H18C	109.5
H9B—C9—H9C	109.5	H18B—C18—H18C	109.5
C8—N1—C1—C2	−0.5 (2)	C18—N2—C10—C11	−171.3 (2)
C9—N1—C1—C2	−179.3 (2)	C17—N2—C10—C11	−32.1 (2)
N1—C1—C2—C3	0.5 (2)	C1—C2—C10—N2	−125.8 (2)
N1—C1—C2—C10	−178.07 (19)	C3—C2—C10—N2	56.0 (3)
C1—C2—C3—C4	−179.1 (2)	C1—C2—C10—C11	115.6 (3)
C10—C2—C3—C4	−0.6 (4)	C3—C2—C10—C11	−62.6 (3)
C1—C2—C3—C8	−0.3 (2)	N2—C10—C11—C12	30.0 (2)
C10—C2—C3—C8	178.2 (2)	C2—C10—C11—C12	155.37 (19)

C8—C3—C4—C5	0.2 (3)	C10—C11—C12—C13	163.0 (3)
C2—C3—C4—C5	178.9 (2)	C10—C11—C12—C17	−18.9 (2)
C3—C4—C5—C6	0.0 (4)	C17—C12—C13—C14	−0.1 (4)
C4—C5—C6—C7	−0.8 (4)	C11—C12—C13—C14	177.8 (2)
C5—C6—C7—C8	1.3 (4)	C12—C13—C14—C15	1.2 (4)
C1—N1—C8—C7	178.9 (2)	C13—C14—C15—C16	−0.9 (4)
C9—N1—C8—C7	−2.2 (4)	C14—C15—C16—C17	−0.4 (3)
C1—N1—C8—C3	0.2 (2)	C13—C12—C17—N2	177.8 (2)
C9—N1—C8—C3	179.1 (2)	C11—C12—C17—N2	−0.6 (2)
C6—C7—C8—N1	−179.5 (2)	C13—C12—C17—C16	−1.3 (3)
C6—C7—C8—C3	−1.0 (3)	C11—C12—C17—C16	−179.6 (2)
C4—C3—C8—N1	179.05 (18)	C18—N2—C17—C12	159.2 (2)
C2—C3—C8—N1	0.0 (2)	C10—N2—C17—C12	21.5 (2)
C4—C3—C8—C7	0.3 (3)	C18—N2—C17—C16	−21.8 (3)
C2—C3—C8—C7	−178.73 (19)	C10—N2—C17—C16	−159.6 (2)
C18—N2—C10—C2	61.8 (3)	C15—C16—C17—C12	1.5 (3)
C17—N2—C10—C2	−158.96 (18)	C15—C16—C17—N2	−177.4 (2)

Fig. 1