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2-(1-Hexyl-2-oxo-2,3-dihydro-1*H*-indol-3-ylidene)-propanedinitrile

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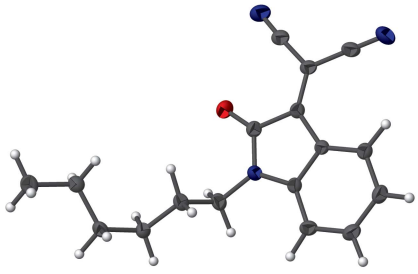
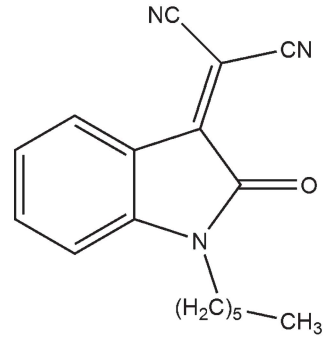
In the title compound, $C_{17}H_{17}N_3O$, the indolone ring system is almost planar, with an angle of 0.76 (14)° between the five- and six-membered rings. The dicyanomethylidene substituent lies close to the indolene plane. In the crystal, offset π-stacking interactions between five- and six-membered rings of indolene stack the molecules along the *c*-axis direction.

3D view



Chemical scheme



Structure description

Oxindole derivatives are used in a wide range of biological applications, including as NMDA antagonists (Kang *et al.*, 2002) and calcium channel blockers (Swensen *et al.*, 2012), as well as having antiangiogenic (Whatmore *et al.*, 2002) and anticancer properties (Peddibhotla, 2009). In a continuaton of our previous research on the synthesis of new heterocyclic systems containing the oxindole unit (Alsubari *et al.*, 2009; Al Mamari *et al.*, 2012), we report here the synthesis and the crystal structure of the title compound (Fig. 1).

The bicyclic portion of the molecule is almost planar with the five- and six-membered rings inclined to one another by 0.76 (14)°. The dicyanomethylidene substituent is with this ring system, as indicated by the C6—C7—C9—C10 torsion angle of −1.5 (5)°.

In the crystal, the indolone units stack along the *c*-axis direction as a result of π–π stacking interactions between the five- and six-membered rings (Fig. 2), with $Cg1\cdots Cg2^{i} = Cg1^{ii}\cdots Cg2 = 3.6985$ (18) Å. The stacking is such that the *n*-hexyl chains all point in the same direction and are arranged one on top of the other (Fig. 3).

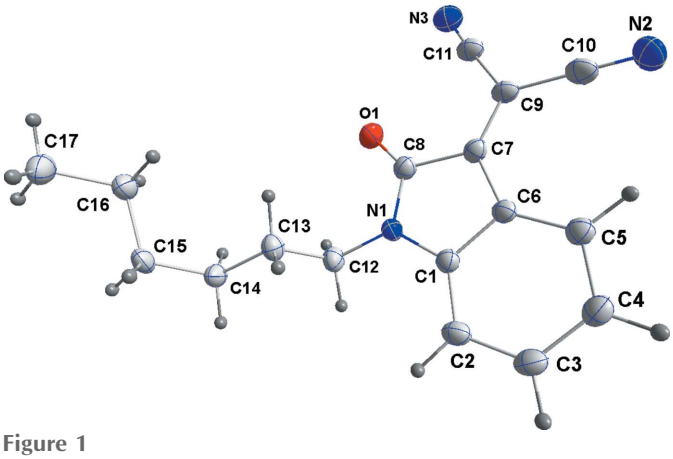


Figure 1
The title molecule, showing the atom-labelling scheme and 50% probability displacement ellipsoids.

Synthesis and crystallization

A mixture of 1-hexyl-1*H*-indol-2,3-dione (0.5 g, 2.1 mmol), malononitrile (0.14 g, 2.1 mmol) and iodine (0.05 g, 0.21 mmol) in ethanol (10 ml), was heated at 333 K for appropriately 1 h. The reaction was monitored by thin-layer chromatography. After completion, the mixture was treated with aqueous $Na_2S_2O_3$ solution, extracted with ethyl acetate (2 × 10 ml). The extract was dried over sodium sulfate, filtered and the solvent was evaporated under reduced pressure. The product obtained was recrystallized from an ethanol solution to afford the title compound as orange plate-like crystals.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{17}N_3O$
M_r	279.33
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	150
a, b, c (Å)	12.0350 (5), 24.5052 (9), 4.9909 (2)
V (Å^3)	1471.92 (10)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.64
Crystal size (mm)	0.31 × 0.12 × 0.03
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.86, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10951, 2456, 2293
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.619
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.114, 1.08
No. of reflections	2456
No. of parameters	247
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.19

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal and refinement details appear in Table 1. Independent refinement of the H atoms attached to C17 did not result in a satisfactory geometry for the methyl group so these H atoms were included as riding contributions in idealized positions. The absolute structure could not be determined with certainty.

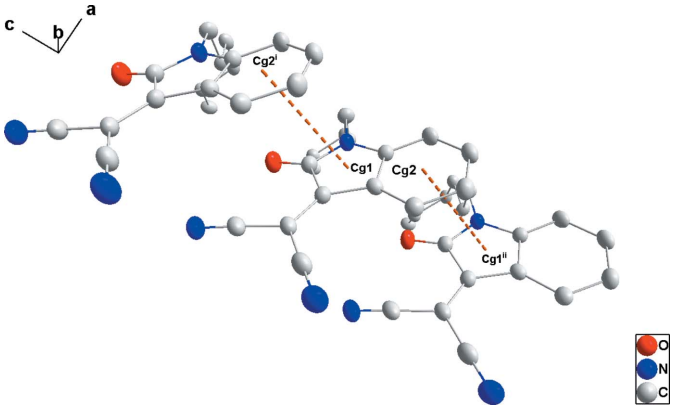


Figure 2
Detail of the π-stacking interactions. *Cg*1 and *Cg*2 are, respectively, the centroids of the five- and six-membered rings. [Symmetry codes: (i) x, y, $z + 1$; (ii) x, y, $z - 1$.]

Acknowledgements

The support of NSF-MRI Grant #1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

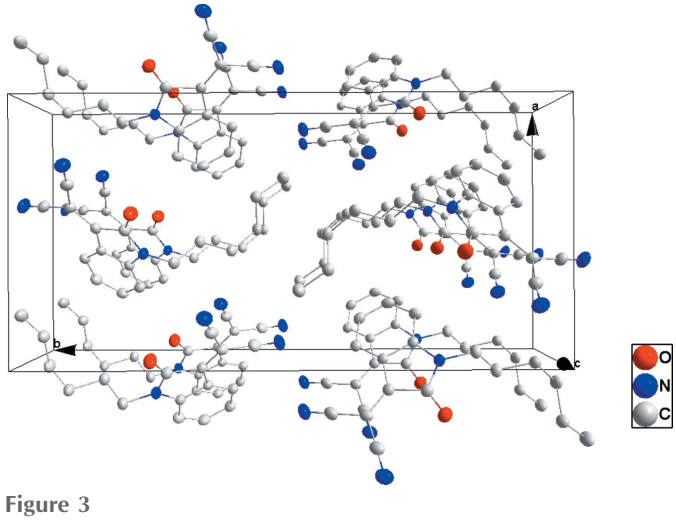


Figure 3
The packing viewed along the *c*-axis direction.

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full crystallographic data

IUCrData (2017). **2**, x170706 [https://doi.org/10.1107/S2414314617007064]

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Crystal data

$C_{17}H_{17}N_3O$
M_r = 279.33
Orthorhombic, $Pna2_1$
a = 12.0350 (5) Å
b = 24.5052 (9) Å
c = 4.9909 (2) Å
V = 1471.92 (10) Å^3
Z = 4
F(000) = 592
D_x = 1.261 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9234 reflections
θ = 3.6–72.6°
μ = 0.64 mm^{-1}
T = 150 K
Plate, orange
0.31 × 0.12 × 0.03 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.86, T_{max} = 0.98
10951 measured reflections
2456 independent reflections
2293 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 72.7°, θ_{min} = 3.6°
h = −14→14
k = −30→26
l = −6→5

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.114
S = 1.08
2456 reflections
247 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.2296P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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. Independent refinement of the hydrogen atoms attached to C17 did not result in a satisfactory geometry for the methyl group so these hydroges were inclused as riding contributions in idealized positions. The absolute structure could not be determined with certainty.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.43707 (17)	0.23427 (8)	0.8224 (5)	0.0360 (5)
N1	0.57751 (17)	0.23919 (8)	0.5072 (5)	0.0253 (5)
N2	0.4030 (2)	0.02200 (11)	0.4344 (7)	0.0404 (7)
N3	0.2816 (2)	0.13700 (11)	1.0310 (6)	0.0380 (6)
C1	0.6233 (2)	0.20390 (10)	0.3149 (6)	0.0226 (6)
C2	0.7104 (2)	0.21380 (11)	0.1406 (6)	0.0273 (6)
H2	0.754 (3)	0.2494 (13)	0.128 (8)	0.034 (9)*
C3	0.7402 (2)	0.17153 (12)	−0.0332 (6)	0.0296 (6)
H3	0.800 (3)	0.1766 (14)	−0.155 (9)	0.036 (9)*
C4	0.6858 (2)	0.12108 (12)	−0.0306 (7)	0.0297 (6)
H4	0.707 (3)	0.0939 (15)	−0.151 (9)	0.037 (9)*
C5	0.5998 (2)	0.11140 (11)	0.1507 (6)	0.0253 (6)
H5	0.562 (3)	0.0746 (13)	0.145 (8)	0.027 (8)*
C6	0.56841 (19)	0.15300 (10)	0.3244 (6)	0.0217 (5)
C7	0.48545 (19)	0.15684 (10)	0.5328 (6)	0.0230 (5)
C8	0.4937 (2)	0.21416 (11)	0.6448 (6)	0.0263 (6)
C9	0.41289 (19)	0.11947 (11)	0.6307 (6)	0.0247 (6)
C10	0.4070 (2)	0.06503 (12)	0.5225 (7)	0.0308 (6)
C11	0.3402 (2)	0.13069 (11)	0.8536 (6)	0.0286 (6)
C12	0.6099 (2)	0.29548 (11)	0.5559 (7)	0.0283 (6)
H12A	0.611 (3)	0.3029 (16)	0.756 (9)	0.045 (11)*
H12B	0.690 (3)	0.3001 (15)	0.493 (9)	0.047 (10)*
C13	0.5359 (2)	0.33637 (11)	0.4133 (6)	0.0262 (6)
H13A	0.455 (3)	0.3282 (13)	0.473 (8)	0.032 (8)*
H13B	0.543 (3)	0.3296 (15)	0.234 (9)	0.038 (10)*
C14	0.5682 (2)	0.39492 (11)	0.4838 (7)	0.0281 (6)
H14A	0.564 (3)	0.3969 (14)	0.707 (8)	0.034 (9)*
H14B	0.644 (3)	0.4013 (17)	0.415 (9)	0.054 (12)*
C15	0.4910 (2)	0.43796 (10)	0.3688 (6)	0.0276 (6)
H15A	0.480 (3)	0.4318 (16)	0.158 (10)	0.054 (12)*
H15B	0.529 (3)	0.4761 (15)	0.403 (9)	0.042 (10)*
C16	0.3748 (2)	0.43823 (11)	0.4915 (7)	0.0292 (6)
H16A	0.386 (4)	0.4422 (16)	0.692 (10)	0.050 (11)*
H16B	0.338 (3)	0.4034 (14)	0.448 (7)	0.033 (9)*
C17	0.3042 (2)	0.48580 (12)	0.3981 (8)	0.0369 (7)
H17A	0.2310	0.4839	0.4838	0.055*
H17B	0.2953	0.4841	0.2031	0.055*

H17C	0.3406	0.5202	0.4469	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0386 (11)	0.0338 (11)	0.0357 (13)	0.0015 (9)	0.0127 (10)	−0.0065 (9)
N1	0.0279 (10)	0.0208 (11)	0.0272 (13)	−0.0012 (8)	0.0058 (10)	−0.0009 (9)
N2	0.0466 (14)	0.0266 (12)	0.0480 (18)	−0.0084 (11)	0.0080 (13)	0.0004 (12)
N3	0.0358 (12)	0.0448 (15)	0.0335 (15)	−0.0027 (11)	0.0075 (13)	0.0050 (12)
C1	0.0231 (11)	0.0216 (12)	0.0231 (14)	0.0015 (10)	0.0004 (10)	0.0002 (10)
C2	0.0264 (12)	0.0294 (13)	0.0262 (15)	−0.0021 (10)	0.0041 (12)	0.0047 (12)
C3	0.0245 (11)	0.0352 (14)	0.0291 (16)	0.0025 (11)	0.0061 (12)	0.0033 (13)
C4	0.0302 (13)	0.0298 (13)	0.0290 (16)	0.0055 (11)	0.0050 (12)	−0.0030 (12)
C5	0.0245 (11)	0.0248 (13)	0.0265 (15)	0.0020 (10)	0.0005 (11)	−0.0001 (12)
C6	0.0193 (11)	0.0214 (12)	0.0245 (14)	0.0013 (9)	0.0002 (10)	0.0027 (10)
C7	0.0238 (11)	0.0232 (12)	0.0221 (13)	0.0020 (9)	0.0006 (10)	0.0008 (11)
C8	0.0270 (11)	0.0260 (12)	0.0258 (14)	0.0015 (10)	0.0016 (12)	−0.0005 (12)
C9	0.0221 (11)	0.0282 (13)	0.0238 (14)	−0.0009 (9)	0.0020 (11)	0.0041 (11)
C10	0.0292 (12)	0.0307 (14)	0.0324 (16)	−0.0047 (10)	0.0047 (12)	0.0063 (13)
C11	0.0246 (12)	0.0320 (14)	0.0292 (16)	−0.0008 (10)	0.0006 (12)	0.0059 (12)
C12	0.0305 (12)	0.0224 (13)	0.0320 (17)	−0.0010 (10)	−0.0017 (12)	−0.0044 (11)
C13	0.0312 (13)	0.0224 (12)	0.0249 (15)	−0.0002 (10)	−0.0010 (11)	−0.0016 (11)
C14	0.0269 (12)	0.0226 (12)	0.0347 (17)	−0.0007 (10)	0.0053 (12)	−0.0032 (12)
C15	0.0331 (14)	0.0205 (12)	0.0291 (16)	−0.0015 (10)	0.0049 (12)	0.0050 (12)
C16	0.0288 (12)	0.0255 (13)	0.0333 (17)	−0.0007 (11)	0.0017 (12)	0.0006 (12)
C17	0.0323 (14)	0.0287 (14)	0.050 (2)	0.0023 (12)	−0.0044 (14)	0.0024 (14)

Geometric parameters (Å, º)

O1—C8	1.222 (4)	C9—C11	1.442 (4)
N1—C8	1.365 (4)	C12—C13	1.518 (4)
N1—C1	1.405 (4)	C12—H12A	1.01 (4)
N1—C12	1.454 (3)	C12—H12B	1.02 (4)
N2—C10	1.144 (4)	C13—C14	1.527 (4)
N3—C11	1.142 (4)	C13—H13A	1.03 (3)
C1—C2	1.383 (4)	C13—H13B	0.91 (4)
C1—C6	1.412 (4)	C14—C15	1.518 (4)
C2—C3	1.398 (4)	C14—H14A	1.12 (4)
C2—H2	1.02 (3)	C14—H14B	0.99 (4)
C3—C4	1.399 (4)	C15—C16	1.526 (4)
C3—H3	0.95 (4)	C15—H15A	1.07 (5)
C4—C5	1.395 (4)	C15—H15B	1.06 (4)
C4—H4	0.93 (4)	C16—C17	1.516 (4)
C5—C6	1.390 (4)	C16—H16A	1.02 (5)
C5—H5	1.01 (3)	C16—H16B	0.99 (3)
C6—C7	1.445 (4)	C17—H17A	0.9800
C7—C9	1.357 (4)	C17—H17B	0.9800
C7—C8	1.515 (4)	C17—H17C	0.9800

C9—C10	1.441 (4)		
C8—N1—C1	110.9 (2)	C13—C12—H12A	111 (2)
C8—N1—C12	122.7 (2)	N1—C12—H12B	108 (2)
C1—N1—C12	126.4 (2)	C13—C12—H12B	110 (2)
C2—C1—N1	128.2 (2)	H12A—C12—H12B	106 (3)
C2—C1—C6	122.0 (3)	C12—C13—C14	111.3 (2)
N1—C1—C6	109.7 (2)	C12—C13—H13A	107 (2)
C1—C2—C3	117.0 (2)	C14—C13—H13A	110.7 (19)
C1—C2—H2	125 (2)	C12—C13—H13B	107 (2)
C3—C2—H2	118 (2)	C14—C13—H13B	112 (2)
C2—C3—C4	121.9 (3)	H13A—C13—H13B	110 (3)
C2—C3—H3	120 (2)	C15—C14—C13	114.2 (2)
C4—C3—H3	118 (2)	C15—C14—H14A	108.5 (19)
C5—C4—C3	120.2 (3)	C13—C14—H14A	105.0 (18)
C5—C4—H4	120 (2)	C15—C14—H14B	109 (2)
C3—C4—H4	120 (2)	C13—C14—H14B	108 (2)
C6—C5—C4	118.8 (2)	H14A—C14—H14B	113 (3)
C6—C5—H5	124 (2)	C14—C15—C16	114.3 (2)
C4—C5—H5	118 (2)	C14—C15—H15A	110 (2)
C5—C6—C1	120.0 (2)	C16—C15—H15A	107 (2)
C5—C6—C7	133.2 (2)	C14—C15—H15B	107 (2)
C1—C6—C7	106.9 (2)	C16—C15—H15B	109 (2)
C9—C7—C6	131.3 (3)	H15A—C15—H15B	110 (3)
C9—C7—C8	122.4 (2)	C17—C16—C15	113.2 (2)
C6—C7—C8	106.3 (2)	C17—C16—H16A	107 (2)
O1—C8—N1	126.6 (3)	C15—C16—H16A	106 (3)
O1—C8—C7	127.2 (3)	C17—C16—H16B	110.2 (19)
N1—C8—C7	106.2 (2)	C15—C16—H16B	108.7 (19)
C7—C9—C10	121.4 (3)	H16A—C16—H16B	111 (3)
C7—C9—C11	122.7 (3)	C16—C17—H17A	109.5
C10—C9—C11	115.8 (2)	C16—C17—H17B	109.5
N2—C10—C9	179.3 (3)	H17A—C17—H17B	109.5
N3—C11—C9	176.8 (3)	C16—C17—H17C	109.5
N1—C12—C13	113.0 (2)	H17A—C17—H17C	109.5
N1—C12—H12A	110 (2)	H17B—C17—H17C	109.5
C8—N1—C1—C2	−178.7 (3)	C1—N1—C8—O1	−179.2 (3)
C12—N1—C1—C2	2.8 (5)	C12—N1—C8—O1	−0.7 (5)
C8—N1—C1—C6	−0.3 (3)	C1—N1—C8—C7	0.7 (3)
C12—N1—C1—C6	−178.7 (3)	C12—N1—C8—C7	179.2 (2)
N1—C1—C2—C3	−179.9 (3)	C9—C7—C8—O1	−2.6 (5)
C6—C1—C2—C3	1.8 (4)	C6—C7—C8—O1	179.0 (3)
C1—C2—C3—C4	−0.7 (4)	C9—C7—C8—N1	177.5 (3)
C2—C3—C4—C5	−0.7 (4)	C6—C7—C8—N1	−0.9 (3)
C3—C4—C5—C6	1.0 (4)	C6—C7—C9—C10	−1.5 (5)
C4—C5—C6—C1	0.0 (4)	C8—C7—C9—C10	−179.5 (3)
C4—C5—C6—C7	−179.6 (3)	C6—C7—C9—C11	176.2 (3)

C2—C1—C6—C5	−1.5 (4)	C8—C7—C9—C11	−1.8 (4)
N1—C1—C6—C5	179.9 (2)	C8—N1—C12—C13	−81.7 (3)
C2—C1—C6—C7	178.2 (3)	C1—N1—C12—C13	96.6 (3)
N1—C1—C6—C7	−0.4 (3)	N1—C12—C13—C14	176.4 (2)
C5—C6—C7—C9	2.2 (5)	C12—C13—C14—C15	−174.6 (3)
C1—C6—C7—C9	−177.5 (3)	C13—C14—C15—C16	68.5 (3)
C5—C6—C7—C8	−179.5 (3)	C14—C15—C16—C17	173.0 (3)
C1—C6—C7—C8	0.8 (3)		