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Methyl (*E*)-2-cyano-3-(1*H*-indol-3-yl)acrylate

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.047
wR factor = 0.134
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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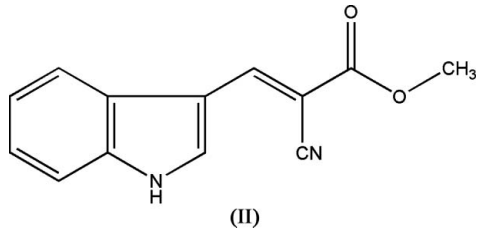
In the title compound, $C_{13}H_{10}N_2O_2$, the indole ring system is planar and the acrylate double bond adopts the *E* stereochemistry. The molecules are linked by intermolecular N—H···O hydrogen bonds.

Comment

The Knovenagal condensation is an important carbon–carbon-bond-forming reaction in organic synthesis. Utilizing this reaction, we attempted to prepare ethyl (*E*)-2-cyano-3-(1*H*-indol-3-yl)acrylate, (I), by reacting indole-3-carbaldehyde with ethyl cyanoacetate in methanol, using a catalytic amount of piperidine under reflux. However, the resultant product was not the expected compound (I), but the trans-esterified product, *viz*. methyl (*E*)-2-cyano-3-(1*H*-indol-3-yl)acrylate, (II), which was obtained as a single geometrical isomer. In order to confirm the double-bond geometry of this compound, its X-ray crystal structure determination has been carried out.



(II)

The molecular structure and atom-numbering scheme of (II) are shown in Fig. 1. Selected geometric parameters are presented in Table 1. The C9═C10 double bond is coplanar with the plane of the indole ring system, evident from the C1—C2—C9—C10 torsion angle [3.1 (3)°], facilitating extended conjugation between the π-electrons of the indole ring system and the acrylate group.

The packing of compound (II), viewed down the *a* axis, is illustrated in Fig. 2. The molecules are linked by an intermolecular N—H···O hydrogen bond, details of which are given in Table 2.

Experimental

Indole-3-carbaldehyde (0.725 g, 5 mmol) and ethyl cyanoacetate (0.566 g, 5 mmol) were mixed in 10 ml methanol. To the mixture 3–4 drops of piperidine were added and the mixture refluxed for 2 h. Crystals separated out after cooling and were collected by filtration and washed with methanol. Recrystallization from methanol afforded bright-yellow crystals of (II), which were suitable for X-ray analysis. ^{1}H NMR (DMSO): δ 3.81 (*s*, 3H), 7.20–7.29 (*m*, 2H), 7.55 (*d*, 1H), 7.94 (*d*, 1H), 8.54 (*t*, 2H), 12.58 (*s*, 1H). ^{13}C NMR (DMSO): δ 52.6, 91.9,

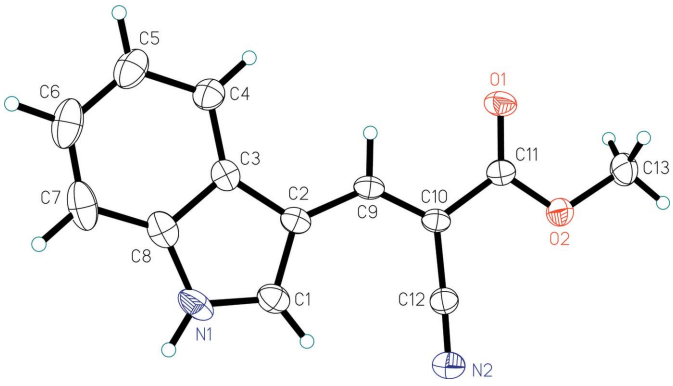


Figure 1
A view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

109.8, 112.8, 117.9, 118.4, 122.0, 123.5, 126.7, 132.6, 136.1, 146.5, 163.5.

Crystal data

$C_{13}H_{10}N_2O_2$	D_x = 1.340 Mg m^{-3}
M_r = 226.23	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2793 reflections
a = 11.3655 (5) Å	θ = 1.0–27.5°
b = 6.2177 (3) Å	μ = 0.09 mm^{-1}
c = 15.9239 (8) Å	T = 90.0 (2) K
β = 94.552 (2)°	Cut needle, yellow
V = 1121.75 (9) Å^3	0.32 × 0.20 × 0.10 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.039
ω scans	θ_{max} = 27.5°
Absorption correction: none	$h = -14 \rightarrow 14$
4603 measured reflections	$k = -7 \rightarrow 8$
2573 independent reflections	$l = -20 \rightarrow 20$
1703 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.134	$(\Delta/\sigma)_{max} < 0.001$
S = 1.03	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
2573 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
155 parameters	

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.342 (2)	O2—C11	1.3376 (18)
N2—C12	1.1522 (18)	C1—C2	1.393 (2)
O1—C11	1.2165 (16)	C9—C10	1.355 (2)
C1—C2—C9	129.94 (15)	O1—C11—O2	123.76 (13)
C10—C9—C2	131.12 (14)	O1—C11—C10	123.85 (14)
C9—C10—C12	122.12 (14)	O2—C11—C10	112.39 (12)
C9—C10—C11	119.11 (13)		
C9—C10—C11—O1	0.7 (2)		

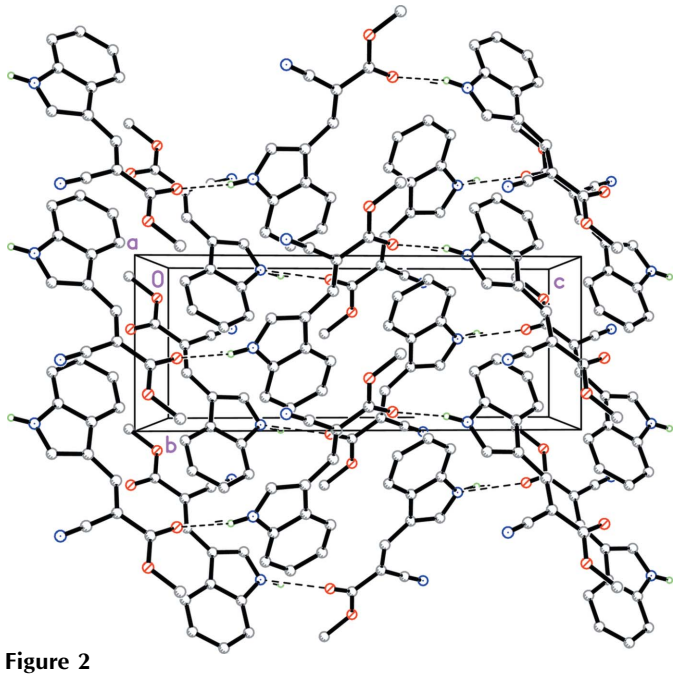


Figure 2
Packing diagram of the title compound, viewed down the *a* axis. H atoms have been omitted for clarity.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.88	2.01	2.8214 (16)	153

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

All H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.95–0.98 Å and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(C) for the methyl C atom.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

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