

Methyl (*E*)-2-cyano-3-(1*H*-indol-3-yl)acrylateVijayakumar N. Sonar,^a Sean Parkin^b and Peter A. Crooks^{a*}^aDepartment of Pharmaceutical Sciences, College of Pharmacy, University of Kentucky, Lexington KY 40536, USA, and ^bDepartment of Chemistry, University of Kentucky, Lexington, KY 40506, USA

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Key indicators

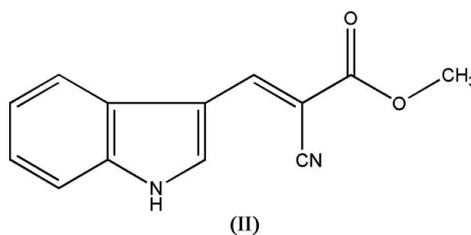
Single-crystal X-ray study
T = 90 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.047
wR factor = 0.134
Data-to-parameter ratio = 16.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{13}\text{H}_{10}\text{N}_2\text{O}_2$, 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.

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Comment

The Knoevenagel 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.



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. ¹H 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). ¹³C NMR (DMSO): δ 52.6, 91.9,

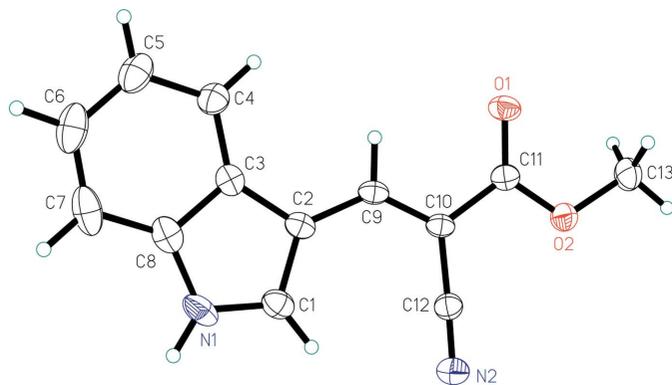


Figure 1

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

$C_{13}H_{10}N_2O_2$
 $M_r = 226.23$
 Monoclinic, $P2_1/c$
 $a = 11.3655$ (5) Å
 $b = 6.2177$ (3) Å
 $c = 15.9239$ (8) Å
 $\beta = 94.552$ (2)°
 $V = 1121.75$ (9) Å³
 $Z = 4$

$D_x = 1.340$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2793 reflections
 $\theta = 1.0$ – 27.5 °
 $\mu = 0.09$ mm⁻¹
 $T = 90.0$ (2) K
 Cut needle, yellow
 $0.32 \times 0.20 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer
 ω scans
 Absorption correction: none
 4603 measured reflections
 2573 independent reflections
 1703 reflections with $I > 2\sigma(I)$

$R_{int} = 0.039$
 $\theta_{max} = 27.5$ °
 $h = -14 \rightarrow 14$
 $k = -7 \rightarrow 8$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.134$
 $S = 1.03$
 2573 reflections
 155 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0778P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.22$ e Å⁻³
 $\Delta\rho_{min} = -0.24$ e Å⁻³

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)		

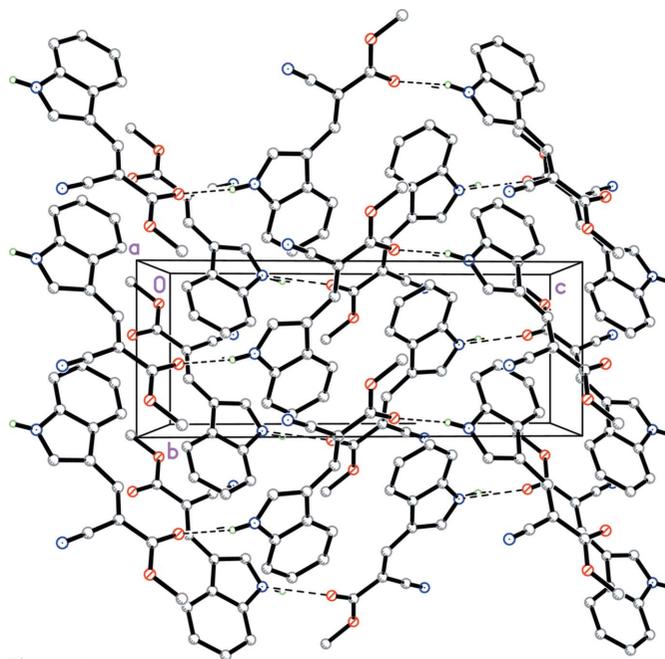


Figure 2

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1N \cdots 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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