Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-Ethyl 2-cyano-3-(1*H*-imidazol-2-yl)-acrylate

Rajesh G. Kalkhambkar,[a] Mahesh Kumar,[a] D. Gayathri,[b] Jeongsu Oh[c] and Yeon Tae Jeong[c]*

[a]Department of Chemistry, Karnatak University's Karnatak Science College, Dharwad 580 001, Karnatak, India, [b]Department of Biotechnology, Dr. M.G.R Educational and Research Institute University, Periyar E.V.R. High Road, Maduravoyal, Chennai 600 095, India, and [c]Department of Image Science and Engineering, Pukyong National University, Busan 608 739, Republic of Korea
Correspondence e-mail: ytjeong@pknu.ac.kr

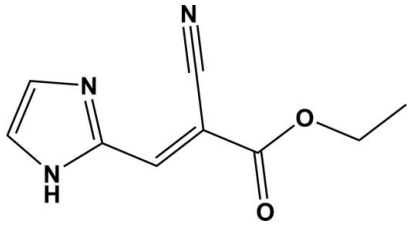
Received 8 June 2013; accepted 2 July 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.049; wR factor = 0.145; data-to-parameter ratio = 11.2.

The crystal structure of the title compound, $C_9H_9N_3O_2$, features N—H···N and C—H···O interactions. The N—H···N interaction generates a chain running along the *a* axis and the C—H···O interaction generates a chain along the *c* axis. An intramolecular C—H···O interaction is also observed.

Related literature

For background references and the biological importance of related compounds, see: Bigi *et al.* (1999); Yu *et al.* (2000). For the synthesis, see: Knoevenagel (1898); Yadav *et al.* (2004).



Experimental

Crystal data

$C_9H_9N_3O_2$ M_r = 191.19
Orthorhombic, $Pca2_1$
a = 10.0768 (7) Å
b = 12.0387 (8) Å
c = 7.7047 (6) Å
V = 934.67 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.4 × 0.23 × 0.2 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.980, T_{max} = 0.984
7118 measured reflections
1436 independent reflections
1189 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.145
S = 0.83
1436 reflections
128 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1	0.93	2.38	2.771 (3)	105
N1—H1A···N2^{i}	0.86	2.11	2.951 (3)	167
C1—H1···O1^{ii}	0.93	2.45	3.328 (4)	158

Symmetry codes: (i) $x-\frac{1}{2}, -y, z$; (ii) $-x, -y, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

The authors thank the Director, USIC Karnatak University Dharwad, India, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2635).

References

Bigi, F., Chesini, L., Maggi, R. & Sartori, G. (1999). *J. Org. Chem.* **64**, 1033–1035.
Bruker (2001). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Knoevenagel, E. (1898). *Berichte*, **31**, 2585–2596.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Yadav, J. S., Subba Reddy, B. V., Basak, A. K., Visali, B., Narsaiah, A. V. & Nagaiah, K. (2004). *Eur. J. Org. Chem.* pp. 546–551.
Yu, N., Aramini, J. M. M., Germann, W. & Huang, Z. (2000). *Tetrahedron Lett.* **41**, 6993–6996.

supporting information

Acta Cryst. (2013). E**69**, o1223 [doi:10.1107/S1600536813018278]

(*Z*)-Ethyl 2-cyano-3-(1*H*-imidazol-2-yl)acrylate

Rajesh G. Kalkhambkar, Mahesh Kumar, D. Gayathri, Jeongsu Oh and Yeon Tae Jeong

S1. Comment

The Knoevenagel condensation is an important carbon-carbon bond-forming reaction in organic synthesis (Knoevenagel, 1898; Yadav *et al.*, 2004). This reaction has been widely used in organic synthesis to prepare many biologically active derivatives, and in the synthesis of cosmetics, perfumes and pharmaceuticals (Bigi *et al.*, 1999; Yu *et al.*, 2000). With the view of biological importance, the series of compounds are synthesized and here we report the crystal structure of the title compound.

The bond lengths and bond angles are within the normal ranges. The imidazole ring is planar and the acrylate moiety is nearly planar with torsion angles C4—C5—C6—O1 and C4—C5—C6—O2 being -6.1 (5) and 174.2 (3)°, respectively. Atoms C6, C9, N3 and O1 deviate from the planarity of the title compound with maximum deviation of 0.31 (3) Å by O1 atom. The crystal structure is stabilized by C(4)—H(4)···O1 intramolecuar interaction generating S(5) motif. The crystal packing is stabilized by N—H···N and C—H···O intermolecular interaction network. Atom N1 acts as a donor to N2 generating chain of C(4) along *a* axis and atom C1 acts as a donor to O1 generating C(8) chain running along *c* axis.

S2. Experimental

A solution of 1*H*-Imidazole-2-aldehyde (1 mol), ethyl cyanoacetate (1.2 mol) and piperidine (0.1 ml) in ethanol (20 ml) was stirred at room temperature for 4 h. After removal of the volatiles *in vacuo*, yellow solid was obtained in quantitative yield. A sample for analysis was obtained by recrystallization from EtOAc as pale yellow needles.

S3. Refinement

Friedel pairs were merged in the absence of any anamalous scatterers in the molecule. The absolute structure in the present model has been chosen arbitrarily. All H-atoms were refined using a riding model with d(C—H) = 0.93 Å, U_{iso} = 1.2U_{eq} (C) for aromatic, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2 and 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms

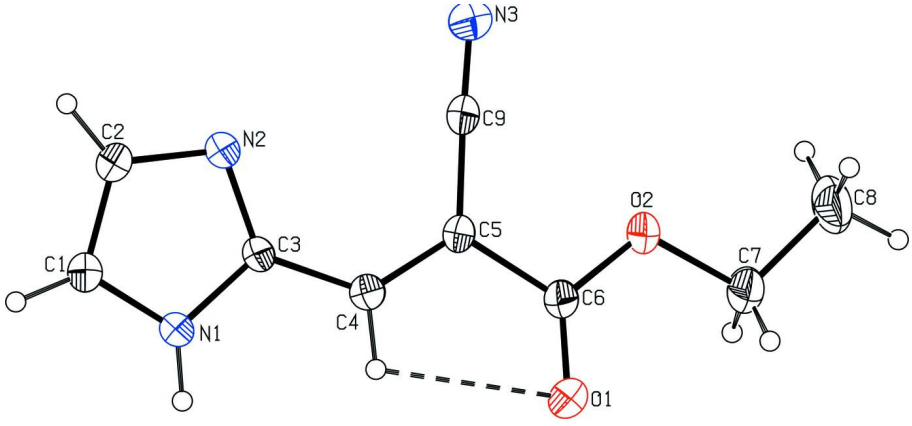


Figure 1

The molecular structure of title compound, showing 30% probability displacement ellipsoids.

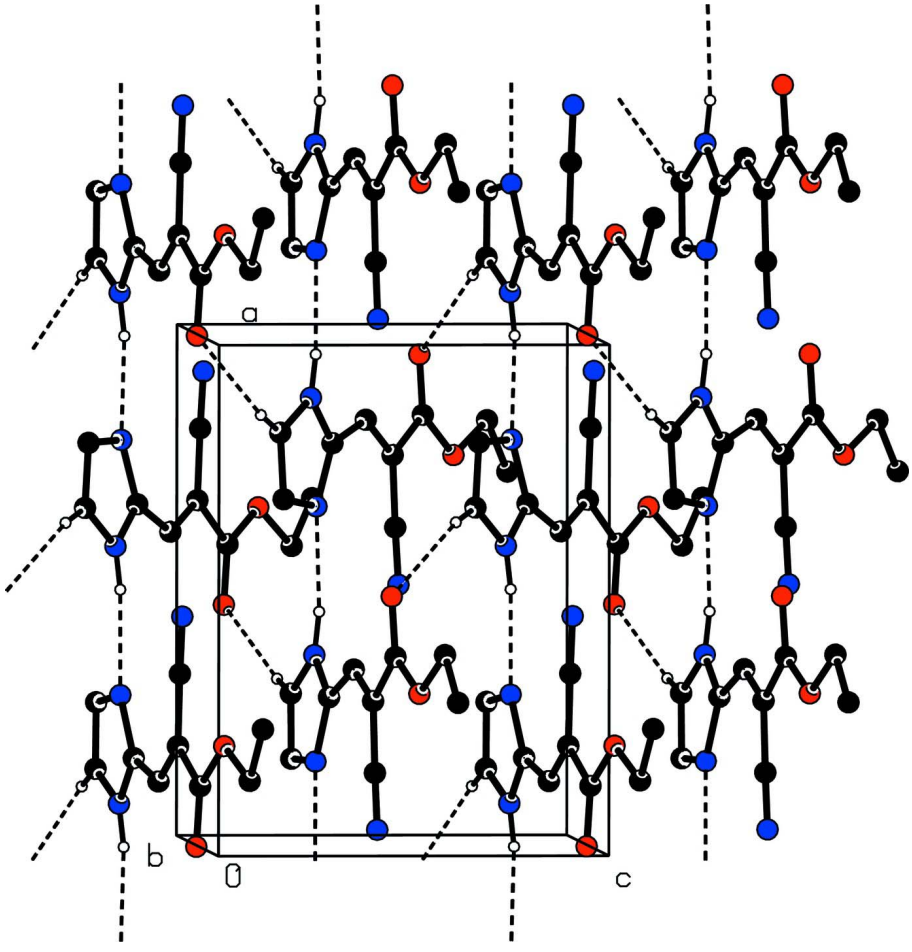


Figure 2

The molecular packing of (I). For clarity, hydrogen atoms which are not involved in hydrogen bonding are omitted.

(*Z*)-Ethyl 2-cyano-3-(1*H*-imidazol-2-yl)acrylate

Crystal data

$C_9H_9N_3O_2$
$M_r = 191.19$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 10.0768$ (7) Å
$b = 12.0387$ (8) Å
$c = 7.7047$ (6) Å
$V = 934.67$ (12) Å^3
$Z = 4$
$F(000) = 400$
$D_x = 1.359$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 2.6$–$25.0°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Needle, pale yellow
0.4 × 0.23 × 0.2 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.980$, $T_{max} = 0.984$
7118 measured reflections
1436 independent reflections
1189 reflections with ($> 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 30.0°$, $\theta_{min} = 1.7°$
$h = -13 \rightarrow 14$
$k = -16 \rightarrow 12$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.145$
$S = 0.83$
1436 reflections
128 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1215P)^2 + 0.160P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
C1	0.1765 (3)	−0.1284 (2)	−0.3293 (4)	0.0457 (6)
H1	0.1447	−0.1956	−0.3719	0.055*
C2	0.3054 (2)	−0.0942 (2)	−0.3241 (4)	0.0447 (6)
H2	0.3770	−0.1355	−0.3647	0.054*
C3	0.1905 (2)	0.03689 (18)	−0.2134 (3)	0.0364 (5)

C4	0.1407 (2)	0.13759 (19)	−0.1392 (4)	0.0394 (5)
H4	0.0487	0.1416	−0.1308	0.047*
C5	0.20598 (19)	0.22724 (19)	−0.0798 (4)	0.0366 (4)
C6	0.1238 (2)	0.32086 (19)	−0.0127 (4)	0.0416 (5)
C7	0.1271 (3)	0.4938 (2)	0.1330 (5)	0.0623 (9)
H7A	0.0624	0.5220	0.0509	0.075*
H7B	0.0806	0.4701	0.2368	0.075*
C8	0.2242 (4)	0.5808 (3)	0.1760 (6)	0.0696 (10)
H8A	0.2917	0.5503	0.2499	0.104*
H8B	0.1802	0.6406	0.2350	0.104*
H8C	0.2641	0.6082	0.0713	0.104*
C9	0.3471 (2)	0.23680 (19)	−0.0740 (4)	0.0421 (5)
N1	0.10482 (18)	−0.04511 (16)	−0.2601 (3)	0.0420 (5)
H1A	0.0200	−0.0440	−0.2477	0.050*
N2	0.31525 (19)	0.00888 (17)	−0.2512 (3)	0.0396 (5)
N3	0.4589 (2)	0.2480 (2)	−0.0668 (5)	0.0642 (8)
O1	0.00530 (18)	0.32337 (16)	−0.0221 (4)	0.0626 (7)
O2	0.19908 (17)	0.40053 (15)	0.0574 (3)	0.0515 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0384 (11)	0.0307 (12)	0.0680 (15)	0.0007 (9)	−0.0019 (11)	−0.0053 (12)
C2	0.0371 (11)	0.0332 (12)	0.0637 (15)	0.0045 (9)	0.0035 (10)	−0.0040 (12)
C3	0.0309 (9)	0.0250 (10)	0.0533 (13)	−0.0030 (8)	0.0008 (8)	0.0027 (9)
C4	0.0323 (9)	0.0308 (11)	0.0551 (12)	−0.0001 (8)	0.0024 (10)	0.0029 (9)
C5	0.0348 (9)	0.0251 (9)	0.0500 (12)	0.0025 (7)	−0.0003 (9)	0.0015 (9)
C6	0.0371 (10)	0.0286 (11)	0.0591 (14)	−0.0003 (8)	0.0065 (10)	0.0011 (10)
C7	0.0554 (16)	0.0366 (14)	0.095 (2)	0.0014 (11)	0.0195 (16)	−0.0130 (15)
C8	0.086 (3)	0.0436 (15)	0.079 (2)	−0.0112 (15)	0.017 (2)	−0.0204 (16)
C9	0.0397 (11)	0.0264 (10)	0.0602 (14)	0.0028 (8)	−0.0018 (11)	−0.0017 (10)
N1	0.0291 (8)	0.0292 (9)	0.0677 (13)	−0.0027 (7)	0.0001 (9)	−0.0027 (10)
N2	0.0323 (9)	0.0297 (10)	0.0566 (11)	−0.0022 (7)	0.0014 (9)	0.0001 (8)
N3	0.0388 (10)	0.0502 (14)	0.103 (2)	0.0028 (9)	−0.0041 (14)	−0.0151 (15)
O1	0.0395 (9)	0.0418 (10)	0.1065 (19)	0.0051 (8)	0.0059 (10)	−0.0154 (11)
O2	0.0434 (9)	0.0291 (8)	0.0819 (15)	−0.0001 (6)	0.0067 (9)	−0.0126 (9)

Geometric parameters (Å, º)

C1—N1	1.346 (3)	C6—O1	1.197 (3)
C1—C2	1.363 (3)	C6—O2	1.337 (3)
C1—H1	0.9300	C7—C8	1.472 (5)
C2—N2	1.366 (3)	C7—O2	1.458 (3)
C2—H2	0.9300	C7—H7A	0.9700
C3—N2	1.334 (3)	C7—H7B	0.9700
C3—N1	1.360 (3)	C8—H8A	0.9600
C3—C4	1.431 (3)	C8—H8B	0.9600
C4—C5	1.344 (3)	C8—H8C	0.9600

C4—H4	0.9300	C9—N3	1.135 (3)
C5—C9	1.428 (3)	N1—H1A	0.8600
C5—C6	1.491 (3)		
N1—C1—C2	105.9 (2)	C8—C7—O2	107.9 (3)
N1—C1—H1	127.0	C8—C7—H7A	110.1
C2—C1—H1	127.0	O2—C7—H7A	110.1
N2—C2—C1	110.8 (2)	C8—C7—H7B	110.1
N2—C2—H2	124.6	O2—C7—H7B	110.1
C1—C2—H2	124.6	H7A—C7—H7B	108.4
N2—C3—N1	110.9 (2)	C7—C8—H8A	109.5
N2—C3—C4	129.2 (2)	C7—C8—H8B	109.5
N1—C3—C4	119.9 (2)	H8A—C8—H8B	109.5
C5—C4—C3	130.1 (2)	C7—C8—H8C	109.5
C5—C4—H4	114.9	H8A—C8—H8C	109.5
C3—C4—H4	114.9	H8B—C8—H8C	109.5
C4—C5—C9	124.3 (2)	N3—C9—C5	177.6 (3)
C4—C5—C6	116.95 (19)	C1—N1—C3	107.74 (18)
C9—C5—C6	118.8 (2)	C1—N1—H1A	126.1
O1—C6—O2	124.9 (2)	C3—N1—H1A	126.1
O1—C6—C5	123.5 (2)	C3—N2—C2	104.54 (19)
O2—C6—C5	111.56 (19)	C6—O2—C7	115.6 (2)
N1—C1—C2—N2	0.6 (4)	C2—C1—N1—C3	−0.5 (3)
N2—C3—C4—C5	−4.1 (5)	N2—C3—N1—C1	0.2 (3)
N1—C3—C4—C5	177.9 (3)	C4—C3—N1—C1	178.6 (3)
C3—C4—C5—C9	−2.6 (5)	N1—C3—N2—C2	0.1 (3)
C3—C4—C5—C6	178.8 (3)	C4—C3—N2—C2	−178.1 (3)
C4—C5—C6—O1	−6.1 (5)	C1—C2—N2—C3	−0.4 (3)
C9—C5—C6—O1	175.1 (3)	O1—C6—O2—C7	2.7 (5)
C4—C5—C6—O2	174.2 (3)	C5—C6—O2—C7	−177.6 (3)
C9—C5—C6—O2	−4.5 (4)	C8—C7—O2—C6	−170.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1	0.93	2.38	2.771 (3)	105
N1—H1*A*···N2^{i}	0.86	2.11	2.951 (3)	167
C1—H1···O1^{ii}	0.93	2.45	3.328 (4)	158

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