Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[(2-Chloro-5-ethyl-3-pyridinyl)(hydroxy)-methyl]acrylonitrile

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.127
Data-to-parameter ratio = 19.6

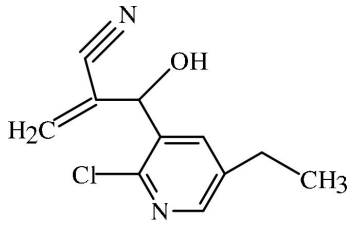
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{11}H_{11}ClN_2O$, molecules are linked *via* O—H···N hydrogen bonds, forming infinite chains running along the *c* axis with a graph-set motif of *C*(6). The structure is further stabilized by weak π–π and C—H···π interactions.

Received 31 January 2005
Accepted 9 February 2005
Online 19 February 2005

Comment

Baylis–Hillman adducts are well known in organic synthesis because of their biological relevance (Kabat *et al.*, 1996; Kim *et al.*, 2002; Shi *et al.*, 2002). We report here the crystal structure of the title compound, (I), which is a Baylis–Hillman product.



(I)

The molecular configuration of (I) is shown in Fig. 1. The molecule contains a chiral atom (C9), although it belongs to a centrosymmetric space group and the compound is thus a racemic mixture. The least-squares plane containing the ethyl group is nearly perpendicular [84.6 (2)°] to the least-squares plane of the pyridine ring. The hydroxy O atom is tilted away from the acrylonitrile group as the C10—C9—O–1 bond angle is distinctly larger than that of C1—C9—O1 (Table 1).

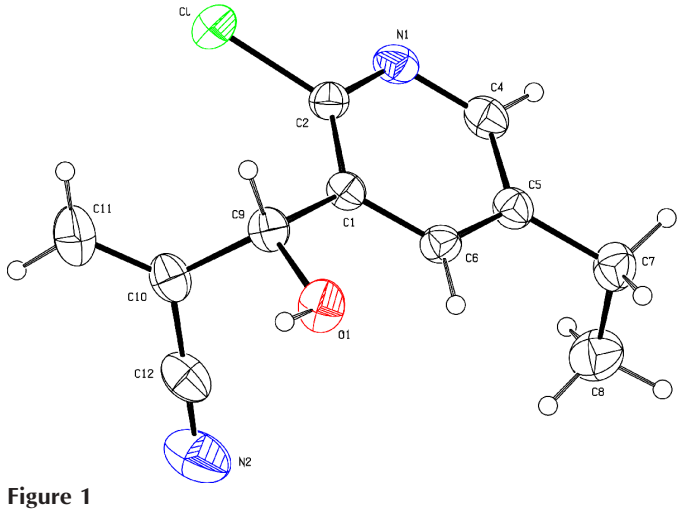


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

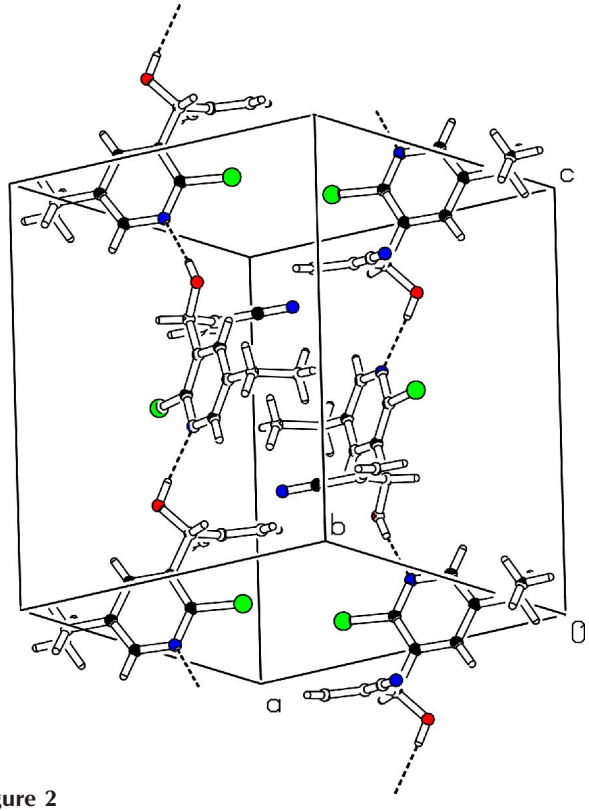


Figure 2
A partial packing diagram of (I), showing the O—H···N hydrogen-bonded (dashed lines) molecules forming chains along the *c* axis.

The crystal packing of (I) is dictated by intermolecular O—H···N hydrogen bonds (Table 2), forming chains [*C*(6) type; Bernstein *et al.*, 1995] along the *c* axis. An intramolecular C—H···O interaction is also observed. This interaction closes the five-membered pseudo-ring O1—C9—C1—C6—H6 according to an *S*(5) pattern. Similar interactions have been reported in the literature (Pálinkó, 1999). Additionally, a weak C—H···π contact exists, involving the π-system of the pyridyl ring (Spek, 2003). The crystal structure is further stabilized by π–π interactions (Steed & Atwood, 2000), with a centre-to-centre distance of 3.654 Å.

Experimental

Compound (I) was prepared by the coupling of 2-chloro-5-ethylpyridine-3-carbaldehyde (5 mmol) and acrylonitrile (5 mmol) in methanol, the reaction mixture being stirred at room temperature in the presence of 1,4-diazabicyclo[2.2.2]octane (5 mmol) for 15 min. This mixture was washed with water, extracted with chloroform and recrystallized from acetonitrile (yield 96%).

Crystal data

$C_{11}H_{11}ClN_2O$
M_r = 222.67
Orthorhombic, *Pccn*
a = 12.5354 (7) Å
b = 12.6028 (7) Å
c = 14.3120 (8) Å
V = 2261.0 (2) Å^3
Z = 8
D_x = 1.308 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 9355 reflections
θ = 4.6–55.8°
μ = 0.31 mm^{-1}
T = 273 (2) K
Block, colourless
0.22 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
18128 measured reflections
2699 independent reflections
2305 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.0°
$h = -16 \rightarrow 16$
$k = -16 \rightarrow 16$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.127
S = 1.05
2699 reflections
138 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N1	1.3203 (17)	C9—O1	1.4057 (17)
C2—Cl	1.7349 (14)	C12—N2	1.142 (3)
C4—N1	1.337 (2)		
O1—C9—C1	107.51 (11)	O1—C9—C10	109.90 (12)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.82	2.00	2.8123 (15)	174
C6—H6···O1	0.93	2.31	2.6646 (16)	102
C8—H8*C*···*Cg*^{ii}	0.96	2.95	3.581 (4)	124

Symmetry codes: (i) $\frac{1}{2} - x, y, z - \frac{1}{2}$; (ii) $-x, -y, -z$.
Note: *Cg* is the centroid of the pyridyl ring.

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms [C—H = 0.93–0.98 Å and O—H = 0.82 Å, and U_{iso}(H) = 1.5U_{eq}(C,O) for methyl and hydroxy H atoms and 1.2U_{eq}(C) for all other H atoms].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; structure solution: *SHELXS97* (Sheldrick, 1997); structure refinement: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

The authors thank Dr J. S. Yadav, Director, IICT, Hyderabad, for his kind encouragement.

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