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Ethyl 3-(2-chloro-5-methyl-3-pyridyl)-3-hydroxy-2-methylenepropanoate

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Key indicators

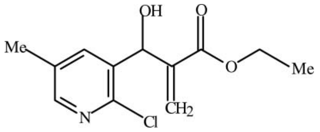
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.111
Data-to-parameter ratio = 18.9

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

In the crystal structure of the title compound, $C_{12}H_{14}ClNO_3$, molecules are linked into chains *via* O—H⋯N hydrogen bonds. In addition, the structure is further stabilized by C—H⋯O and intramolecular C—H⋯Cl interactions.

Comment

Baylis–Hillman (BH) adducts (Baylis & Hillman, 1972) are well known in organic synthesis because of their biological relevance (Kabat *et al.*, 1996; Shi *et al.*, 2002). It has been observed that the reaction processes are very slow (Basavaiah *et al.*, 2003). We reported earlier (Narender *et al.*, 2006) that substituted 2-chloropyridine-3-carbaldehydes undergo extremely fast BH reactions under normal conditions. These pyridine carbaldehyde derivatives serve as synthons for the present BH adduct. In a continuation of our studies on these BH adducts (Swamy *et al.*, 2005, 2007), we report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The propanoate group has an extended conformation (Table 1). The dihedral angle between the mean planes through the pyridine ring (atoms N1/C1–C5) and the propanoate unit (atoms C6/C7/C9/O2/O3) is 88.8 (1)°.

In the crystal packing, molecules are linked into chains *via* O—H⋯N hydrogen bonds. In addition, the structure is further stabilized by C—H⋯O and intramolecular C—H⋯Cl interactions (Table 2).

Experimental

Compound (I) was prepared by the coupling of 2-chloro-5-methylpyridine-3-carbaldehyde (5 mmol) and ethyl acrylate (5 mmol) in methanol (10 ml), the reaction mixture being stirred at room temperature in the presence of 1,4-diazabicyclo[2.2.2]octane (5 mmol) for 15 min. The mixture was washed with water, extracted with chloroform and recrystallized from chloroform (yield 96%).

Crystal data

$C_{12}H_{14}ClNO_3$
M_r = 255.69
Monoclinic, $P2_1/c$
a = 10.8209 (5) Å
b = 10.4195 (5) Å
c = 11.2937 (5) Å
β = 97.845 (1)°
V = 1261.43 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 (2) K
0.22 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
14023 measured reflections
2968 independent reflections
2669 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.111$
$S = 1.03$
2968 reflections
157 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.29$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.29$ e Å^{-3}

Table 1
Selected torsion angles (°).

C6—C7—C9—O3	−171.92 (11)	C11—C10—O3—C9	176.65 (14)
C7—C9—O3—C10	−179.93 (12)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1*A*⋯N1$^{\rm i}$	0.82	2.05	2.8098 (15)	154
C1—H1⋯O2$^{\rm ii}$	0.93	2.49	3.3878 (17)	163
C6—H6⋯Cl1	0.98	2.66	3.0710 (13)	105

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x, -y+2, -z+2$.

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(methyl C), 1.5$U_{\rm eq}$(O) or 1.2$U_{\rm eq}$(C). The methyl and hydroxyl groups were allowed to rotate but not to tip.

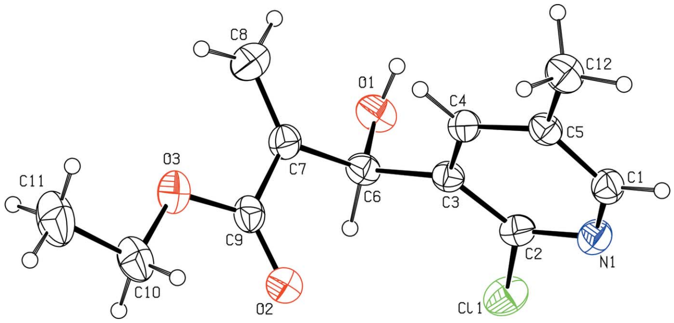


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *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).

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