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Ethyl 1-methylimidazole-2-carboxylate

Brian C. Frye, Mark S. Mashuta, Christopher S. Mullins, Craig A. Grapperhaus and Robert M. Buchanan*

Department of Chemistry, University of Louisville, KY 40292, USA

Correspondence e-mail: msmashuta.xray@louisville.edu

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.030
wR factor = 0.068
Data-to-parameter ratio = 12.3

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

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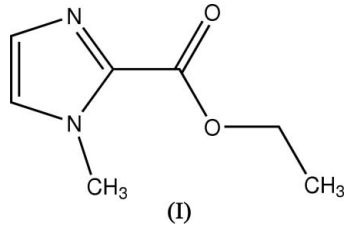
The title compound, $C_7H_{10}N_2O_2$, crystallizes as a nearly planar molecule. The carboxyethyl group is twisted by 4.43 (14)° relative to the plane that includes the imidazole group. The carbonyl unit of the carboxyethyl group is oriented *anti* to the *N*-methyl group of the imidazole.

Comment

The title compound, (I), has been used to synthesize polyamide anticancer agents that contain *N*-methylimidazole and *N*-methylpyrrole groups (Baird & Dervan, 1996; Baraldi *et al.*, 2003; Krowicki & Lown, 1987; Zaffaroni *et al.*, 2002), which bind to the minor groove of DNA and display G–C and C–G base pair recognition (Moser & Dervan, 1987; Marques *et al.*, 2002). Compound (I) is a potential synthon for synthesizing amide-functionalized imidazole chelates (Cheruzel *et al.*, 2002).



(I)

The structure of the related ethyl 1-methyl-4-nitroimidazole-2-carboxylate (Wu *et al.*, 2004), has been reported recently and contains disordered carboxyethyl and *N*-methyl groups, unlike the structure of (I). The carbonyl unit is oriented *anti* to the *N*-methyl group in (I), whereas in ethyl 1-methyl-4-nitroimidazole-2-carboxylate, the carbonyl group adopts a *syn* configuration.

Molecules of (I) are stacked along the crystallographic *a* axis (Fig. 2), the closest contacts between molecules being 3.4391 (13) Å, between N1 at (x, y, z) and C4′ at ($-x$, $1-y$, $-z$), and 3.4921 (12) Å, between C5 at (x, y, z) and O5″ at ($1-x$, $1-y$, $1-z$). The carboxyethyl groups in alternating layers of the stack are oriented in opposite directions, minimizing steric interactions between symmetry-related molecules in adjacent stacks. The imidazole ring in (I) is planar and the torsion angle (N2—C1—C5—O2) associated with the carboxyethyl group is 4.43 (14)°, compared with 15.0 (1)° reported for ethyl 1-methyl-4-nitroimidazole-2-carboxylate (Wu *et al.*, 2004).

Experimental

Compound (I) was synthesized following a previously reported procedure (Krowicki & Lown, 1987). Ethyl chloroformate (0.28 mol)

was added to an acetonitrile solution (60 ml) of *N*-methylimidazole (0.12 mol) and triethylamine (0.22 mol) at 253 K. After 24 h the solution was filtered and the solvent was removed. The resulting residue was dissolved in water and extracted with chloroform. A white solid obtained by column chromatography (silica gel, ethyl acetate) was recrystallized by slow evaporation of the solution in ethyl acetate.

Crystal data

$C_7H_{10}N_2O_2$	D_x = 1.378 Mg m^{-3}
M_r = 154.17	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5362 reflections
a = 7.1040 (8) Å	θ = 2.7–28.0°
b = 15.2244 (18) Å	μ = 0.10 mm^{-1}
c = 7.4511 (9) Å	T = 100 (2) K
β = 112.797 (2)°	Plate, colorless
V = 742.92 (15) Å^3	0.37 × 0.29 × 0.09 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	1728 independent reflections
ω scans	1636 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)	R_{int} = 0.013
T_{min} = 0.960, T_{max} = 0.988	θ_{max} = 28.0°
6360 measured reflections	$h = -9 \rightarrow 9$
	$k = -19 \rightarrow 19$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2 + 0.3603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.030	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.068	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
1728 reflections	
140 parameters	
All H-atom parameters refined	

All H atoms were located in difference electron-density maps and refined isotropically [C—H = 0.948 (13)–0.991 (15) Å].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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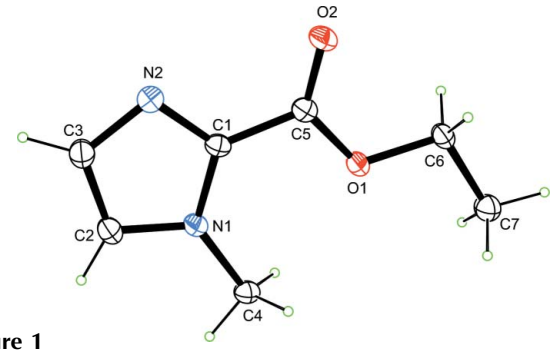

Figure 1
ORTEP-3 drawing (Farrugia, 1997) showing 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

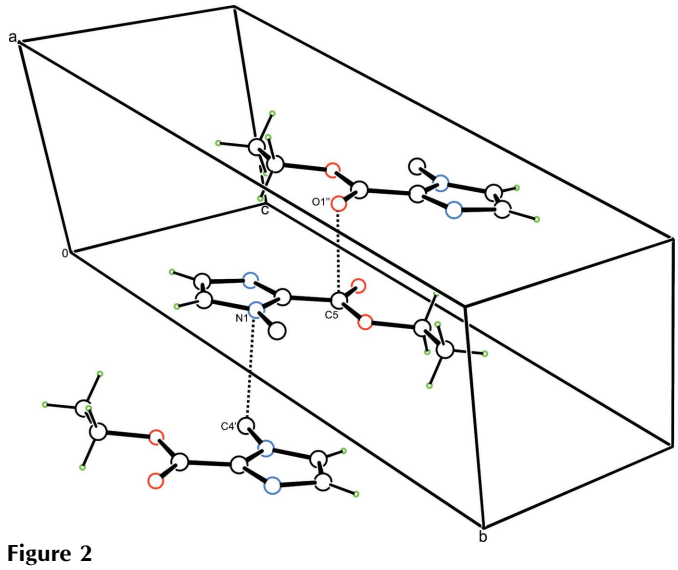

Figure 2
A partial packing diagram, showing molecules of (I) stacked along the crystallographic *a* axis. H atoms of the methyl C atoms have been omitted for clarity. Dashed lines indicate short contacts. [Symmetry codes: (′) $-x$, $1-y$, $-z$; (″) $1-x$, $1-y$, $1-z$.]

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