

Ethyl 4-methyl-1*H*-pyrrole-3-carboxylate

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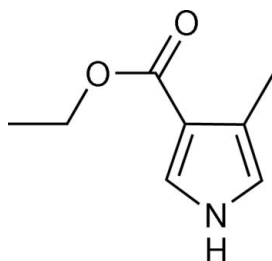
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Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}—\text{C}) = 0.002$ Å; R factor = 0.045; wR factor = 0.124; data-to-parameter ratio = 18.0.

The molecule of the title compound, $\text{C}_8\text{H}_{11}\text{NO}_2$, is nearly planar, possibly due to aromatic conjugation of the carboxylate group with the aromatic pyrrole ring. The molecules form infinite supramolecular chains along the [101] direction *via* intermolecular $\text{N}—\text{H} \cdots \text{O}$ hydrogen bonding.

Related literature

The title compound was prepared according to a synthesis described by Büchel *et al.* (1994). For the structure of a related compound, see: Baures *et al.* (2002). For related literature, see: Cheng *et al.* (1976).



Experimental

Crystal data

 $\text{C}_8\text{H}_{11}\text{NO}_2$ $M_r = 153.18$ Monoclinic, $C2/c$ $a = 11.3757$ (5) Å $b = 10.9074$ (4) Å $c = 14.1470$ (4) Å $\beta = 110.644$ (2)° $V = 1642.64$ (11) Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 0.09$ mm^{−1} $T = 200$ (2) K $0.23 \times 0.12 \times 0.08$ mm

Data collection

Nonius KappaCCD diffractometer

Absorption correction: none

3569 measured reflections

1859 independent reflections

1468 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.020$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$ $wR(F^2) = 0.124$ $S = 1.06$

1859 reflections

103 parameters

Only H-atom displacement parameters refined

 $\Delta\rho_{\text{max}} = 0.21$ e Å^{−3} $\Delta\rho_{\text{min}} = -0.19$ e Å^{−3}

Table 1

Hydrogen-bond geometry (Å, °).

$D—H \cdots A$	$D—H$	$H \cdots A$	$D \cdots A$	$D—H \cdots A$
$\text{N1}—\text{H101} \cdots \text{O2}^i$	0.88	1.96	2.809 (2)	163

Symmetry code: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK* program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank Anna Zangl for experimental support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2348).

References

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supplementary materials

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Ethyl 4-methyl-1*H*-pyrrole-3-carboxylate

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Comment

The title compound, C₈H₁₁NO₂, was prepared as a tentative ligand for transition metal complexes.

The molecular structure is shown in Fig. 1. The molecule is nearly planar which might be due to aromatic conjugation of the carboxyl group with the aromatic pyrrol ring, and packing effects for the ethyl group (Fig. 2).

In the crystal structure, the molecules are connected to form supramolecular chains *via* N—H···O hydrogen bonds (Table 1).

For the structures of a series of imidazole-4,5-dicarboxylic-acid derivatives see Baures *et al.* (2002).

Experimental

The title compound was prepared according to a synthesis described in Büchel *et al.* (1994). The title compound was obtained upon reaction of 4-methylphenylsulfonylmethylisocyanide with 2-butenic acid ethyl ester in a mixture of dry ether and dimethylsulfoxide (2:1) under reductive conditions (NaH) at room temperature. After 30 minutes the excess sodium hydride was quenched by the addition of water. After another 30 minutes the reaction mixture was extracted with diethylether three times. The combined organic phases were combined yielding a yellow oil. After column chromatography on aluminium oxide with dichloromethane as solvent and removal of the solvent of the first fraction, light yellow crystals were obtained.

Refinement

All H atoms were placed in calculated positions with C—H = 0.95 (aromatic), 0.98(methyl), 0.99 Å (methylene) an N—H = 0.88 Å, and refined as riding on their parent atoms with one common isotropic displacement parameter.

Figures

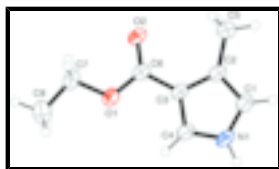


Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

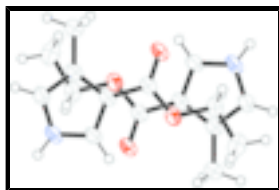


Fig. 2. Packing of two closest non-hydrogen-bonded neighbours of (I) in a normal view.

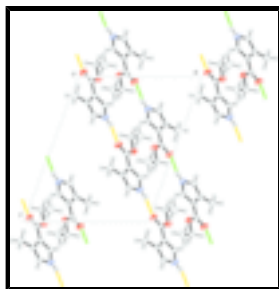


Fig. 3. Hydrogen-bonded chains, view along $[0 - 1 0]$, $0.0 < y < 0.5$ only.

Ethyl 4-methyl-1*H*-pyrrole-3-carboxylate

Crystal data

$C_8H_{11}NO_2$

$M_r = 153.18$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 11.3757 (5) \text{ \AA}$

$b = 10.9074 (4) \text{ \AA}$

$c = 14.1470 (4) \text{ \AA}$

$\beta = 110.644 (2)^\circ$

$V = 1642.64 (11) \text{ \AA}^3$

$Z = 8$

$F_{000} = 656$

$D_x = 1.239 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8547 reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 200 (2) \text{ K}$

Block, light yellow

$0.23 \times 0.12 \times 0.08 \text{ mm}$

Data collection

Enraf-Nonius KappaCCD
diffractometer

Radiation source: rotating anode

Monochromator: MONTEL, graded multilayered X-ray optics

$T = 200(2) \text{ K}$

ω scan

Absorption correction: none

3569 measured reflections

1859 independent reflections

1468 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 3.4^\circ$

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.124$

$S = 1.06$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Only H-atom displacement parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 1.2343P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

1859 reflections $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$
103 parameters $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.17019 (10)	0.42295 (10)	0.01285 (8)	0.0377 (3)
O2	0.02995 (10)	0.26964 (12)	−0.03239 (8)	0.0436 (3)
C3	0.18984 (13)	0.26544 (14)	0.12870 (10)	0.0307 (3)
N1	0.33745 (12)	0.23332 (12)	0.27728 (9)	0.0376 (3)
H101	0.4069	0.2405	0.3302	0.0626 (18)*
C2	0.15334 (14)	0.16238 (14)	0.17421 (11)	0.0332 (3)
C6	0.12186 (13)	0.31647 (14)	0.02949 (11)	0.0316 (3)
C4	0.30409 (14)	0.30630 (15)	0.19499 (11)	0.0350 (4)
H41	0.3509	0.3738	0.1845	0.0626 (18)*
C7	0.10894 (16)	0.47856 (16)	−0.08579 (12)	0.0399 (4)
H71	0.0194	0.4948	−0.0970	0.0626 (18)*
H72	0.1136	0.4230	−0.1397	0.0626 (18)*
C5	0.03638 (16)	0.08732 (17)	0.13147 (13)	0.0460 (4)
H51	0.0417	0.0157	0.1746	0.0626 (18)*
H52	0.0274	0.0600	0.0633	0.0626 (18)*
H53	−0.0366	0.1371	0.1285	0.0626 (18)*
C1	0.24662 (15)	0.14637 (15)	0.26568 (11)	0.0371 (4)
H11	0.2483	0.0848	0.3136	0.0626 (18)*
C8	0.1760 (2)	0.59605 (18)	−0.08746 (16)	0.0552 (5)
H81	0.1696	0.6507	−0.0345	0.0626 (18)*
H82	0.1377	0.6355	−0.1535	0.0626 (18)*
H83	0.2647	0.5789	−0.0755	0.0626 (18)*

Atomic displacement parameters ($\text{\AA}^2$)

	<i>U</i> ¹¹	<i>U</i> ²²	<i>U</i> ³³	<i>U</i> ¹²	<i>U</i> ¹³	<i>U</i> ²³
O1	0.0391 (6)	0.0381 (6)	0.0306 (6)	0.0009 (5)	0.0055 (5)	0.0037 (4)
O2	0.0361 (6)	0.0550 (7)	0.0280 (6)	−0.0057 (5)	−0.0031 (4)	0.0044 (5)
C3	0.0289 (7)	0.0349 (8)	0.0246 (7)	0.0040 (6)	0.0049 (6)	−0.0026 (6)
N1	0.0339 (7)	0.0438 (8)	0.0257 (6)	0.0035 (6)	−0.0013 (5)	−0.0025 (5)
C2	0.0315 (7)	0.0366 (8)	0.0274 (7)	0.0023 (6)	0.0055 (6)	−0.0025 (6)
C6	0.0288 (7)	0.0367 (8)	0.0274 (7)	0.0039 (6)	0.0076 (6)	−0.0016 (6)
C4	0.0335 (8)	0.0359 (8)	0.0292 (7)	0.0021 (6)	0.0032 (6)	−0.0033 (6)
C7	0.0442 (9)	0.0423 (9)	0.0312 (8)	0.0086 (7)	0.0110 (7)	0.0070 (6)
C5	0.0418 (9)	0.0492 (10)	0.0404 (9)	−0.0076 (8)	0.0063 (7)	0.0031 (7)
C1	0.0395 (8)	0.0392 (9)	0.0286 (8)	0.0035 (7)	0.0069 (6)	0.0011 (6)
C8	0.0637 (13)	0.0478 (11)	0.0561 (12)	0.0024 (9)	0.0236 (10)	0.0128 (9)

Geometric parameters ($\text{\AA}$, $^\circ$)

O1—C6	1.3408 (18)	C4—H41	0.9500
O1—C7	1.4544 (18)	C7—C8	1.496 (3)
O2—C6	1.2145 (18)	C7—H71	0.9900
C3—C4	1.381 (2)	C7—H72	0.9900
C3—C2	1.428 (2)	C5—H51	0.9800
C3—C6	1.453 (2)	C5—H52	0.9800
N1—C4	1.350 (2)	C5—H53	0.9800
N1—C1	1.369 (2)	C1—H11	0.9500
N1—H101	0.8800	C8—H81	0.9800
C2—C1	1.365 (2)	C8—H82	0.9800
C2—C5	1.496 (2)	C8—H83	0.9800
C6—O1—C7	116.25 (12)	O1—C7—H72	110.2
C4—C3—C2	107.54 (13)	C8—C7—H72	110.2
C4—C3—C6	125.81 (14)	H71—C7—H72	108.5
C2—C3—C6	126.62 (13)	C2—C5—H51	109.5
C4—N1—C1	109.53 (13)	C2—C5—H52	109.5
C4—N1—H101	125.2	H51—C5—H52	109.5
C1—N1—H101	125.2	C2—C5—H53	109.5
C1—C2—C3	106.00 (13)	H51—C5—H53	109.5
C1—C2—C5	126.62 (15)	H52—C5—H53	109.5
C3—C2—C5	127.37 (13)	C2—C1—N1	109.07 (14)
O2—C6—O1	122.68 (14)	C2—C1—H11	125.5
O2—C6—C3	124.38 (15)	N1—C1—H11	125.5
O1—C6—C3	112.94 (12)	C7—C8—H81	109.5
N1—C4—C3	107.85 (14)	C7—C8—H82	109.5
N1—C4—H41	126.1	H81—C8—H82	109.5
C3—C4—H41	126.1	C7—C8—H83	109.5
O1—C7—C8	107.33 (14)	H81—C8—H83	109.5
O1—C7—H71	110.2	H82—C8—H83	109.5
C8—C7—H71	110.2		
C4—C3—C2—C1	0.30 (17)	C2—C3—C6—O1	171.47 (13)
C6—C3—C2—C1	178.57 (14)	C1—N1—C4—C3	−0.30 (17)
C4—C3—C2—C5	179.78 (15)	C2—C3—C4—N1	0.00 (17)
C6—C3—C2—C5	−2.0 (3)	C6—C3—C4—N1	−178.29 (14)
C7—O1—C6—O2	−2.1 (2)	C6—O1—C7—C8	179.17 (13)
C7—O1—C6—C3	178.68 (12)	C3—C2—C1—N1	−0.48 (17)
C4—C3—C6—O2	170.21 (15)	C5—C2—C1—N1	−179.97 (15)
C2—C3—C6—O2	−7.7 (2)	C4—N1—C1—C2	0.50 (17)
C4—C3—C6—O1	−10.6 (2)		

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H101 $\cdots$ O2 ⁱ	0.88	1.96	2.809 (2)	163

Symmetry codes: (i) $x+1/2, -y+1/2, z+1/2$.

Fig. 1

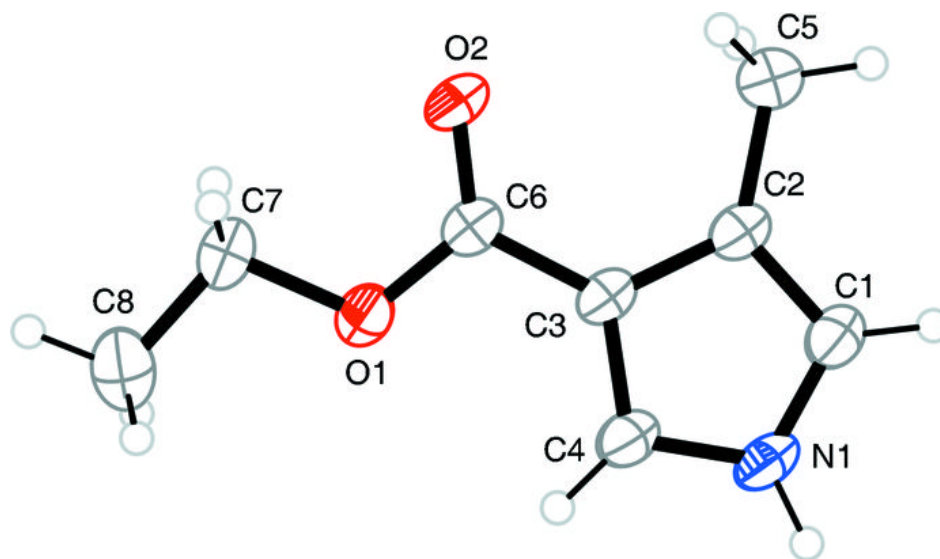


Fig. 2

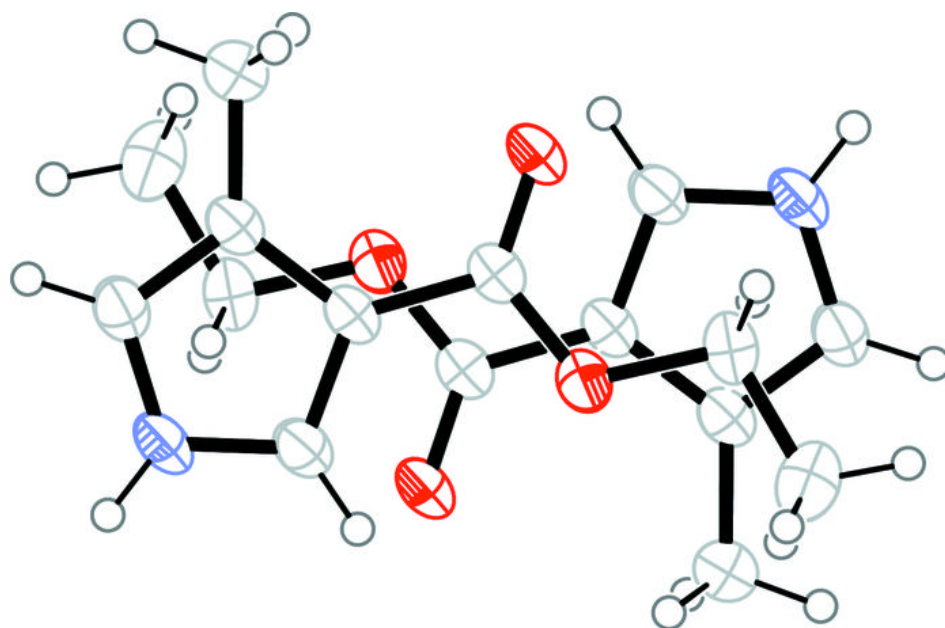


Fig. 3

