Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 3,4-dimethyl-1*H*-pyrrole-2-carboxylate

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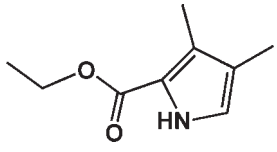
Received 16 July 2010; accepted 8 August 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.136; data-to-parameter ratio = 19.2.

The non-H atoms of the title compound, $C_9H_{13}NO_2$, are almost coplanar (r.m.s. deviation = 0.0358 Å). Weak intermolecular N—H···O hydrogen bonds link the molecules into zigzag chains along the *b* axis with graph-set motif *C*(5). The chains are further linked into a three-dimensional network by C—H···O hydrogen bonds and C—H···π interactions.

Related literature

Schiff bases containing pyrrole units have been extensively investigated due to their excellent coordination abilities, see: Wu *et al.* (2003). For our studies on bis(pyrrol-2-yl-methyleneamine) ligands, see: Wang *et al.*, (2008). For a similar structure, 5-formyl-3,4-dimethyl-1*H*-pyrrole-2-carboxylate, see Wu *et al.* (2009). For the preparation, see: Helms *et al.* (1992). For graph-set motifs, see: Etter *et al.* (1990).



Experimental

Crystal data

$C_9H_{13}NO_2$
M_r = 167.20
Monoclinic, $P2_1/c$
a = 7.7485 (2) Å
b = 7.0611 (2) Å
c = 17.2167 (5) Å
β = 95.103 (2)°
V = 938.24 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.28 × 0.26 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.977, T_{max} = 0.985
8174 measured reflections
2146 independent reflections
1579 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.136
S = 1.04
2146 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

*Cg*1 is the centroid of the N1,C1–C4 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.13	2.9264 (16)	154
C4—H4···*Cg*1^{ii}	0.93	2.92	3.7520 (17)	149
C9—H9*A*···*Cg*1^{iii}	0.96	2.86	3.650 (2)	141

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors are grateful for financial support by the Doctoral Foundation of Henan Polytechnic University (B2009–70 648364).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2205).

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supporting information

Acta Cryst. (2010). E**66**, o2309 [https://doi.org/10.1107/S160053681003179X]

Ethyl 3,4-dimethyl-1*H*-pyrrole-2-carboxylate

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S1. Comment

Schiff bases containing pyrrole units have been extensively investigated due to their excellent coordination abilities (Wu *et al.*, 2003). As a part of our studies on bis(pyrrol-2-yl-methyleneamine) ligands (Wang *et al.*, 2008), the crystal structure of the title compound is reported here.

The non-hydrogen atoms of the title molecule (Fig. 1) are situated in a fair plane (r.m.s. deviation of the non-hydrogen atoms being 0.0358 Å). In the crystal structure, the molecules are linked by weak intermolecular N—H···O hydrogen bonds, forming zig-zag chains with the graph-set motifs C(5) (Etter & MacDonald, 1990). The chains are extended along the *b* axis (Tab. 1, Fig. 2, Fig. 3). The structure is also stabilized by the C—H···O hydrogen bonds (Tab. 1) and C—H···π-electron ring interactions (Tab. 1).

S2. Experimental

The title compound was prepared according to Helms *et al.* (1992). Acetic acid (114 ml) was placed in a 1-*L* round-bottom flask and heated to 85 °C. Sodium acetate (31.09 g), 27.54 g of sodium 2-methyl-3-oxo-l-butene-1-oxide, 37.20 g of diethyl 2-(hydroxyimino)malonate, and a solution of 47 ml of acetic acid in 19.6 ml of H_2O were then added in the respective order. The reaction temperature was raised to 95 °C, and 43.26 g of Zn-dust was added over 45 min while maintaining the temperature between 95 and 110 °C. After the addition of Zn-dust had been completed, the mixture was stirred while keeping its temperature at 110 °C for further 45 min. The reaction mixture was then poured into 500 ml of ice water. The obtained solid was filtered, washed with water and subsequently dissolved in dichloromethane. The solution was washed with saturated sodium hydrogencarbonate, dried with anhydrous sodium sulfate and then the solvent was removed under reduced pressure. The crude product was purified by column chromatography on a silica gel [R_f = 0.68, petroleum ether-ethyl acetate (100:1) as an eluent] to yield 4.82 g (13%) of the title compound. Colourless block crystals [average size: 0.25× 0.25 × 0.20 mm] were obtained by slow evaporation of the ethyl acetate solution at room temperature.

S3. Refinement

All the H atoms were located in the difference electron density map. The H atoms were situated into the idealized positions with the carrier atom-H distances = 0.93 Å for aryl, 0.97 for methylene, 0.96 Å for the methyl and 0.86 Å for the secondary amine hydrogens. The U_{iso} values were constrained to be 1.5U_{eq} of the carrier atom for the methyl H atoms and 1.2U_{eq} for the remaining H atoms.

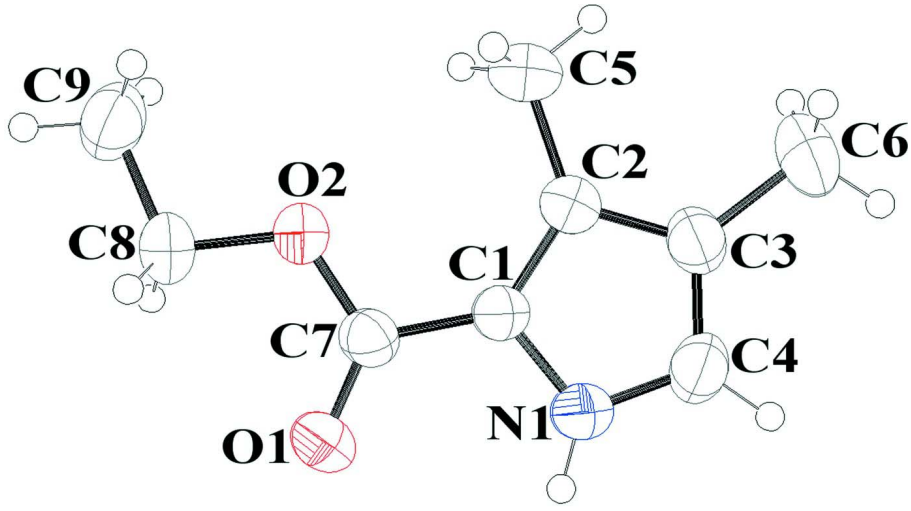

Figure 1

The title molecule with the displacement ellipsoids shown at the 50% probability level.

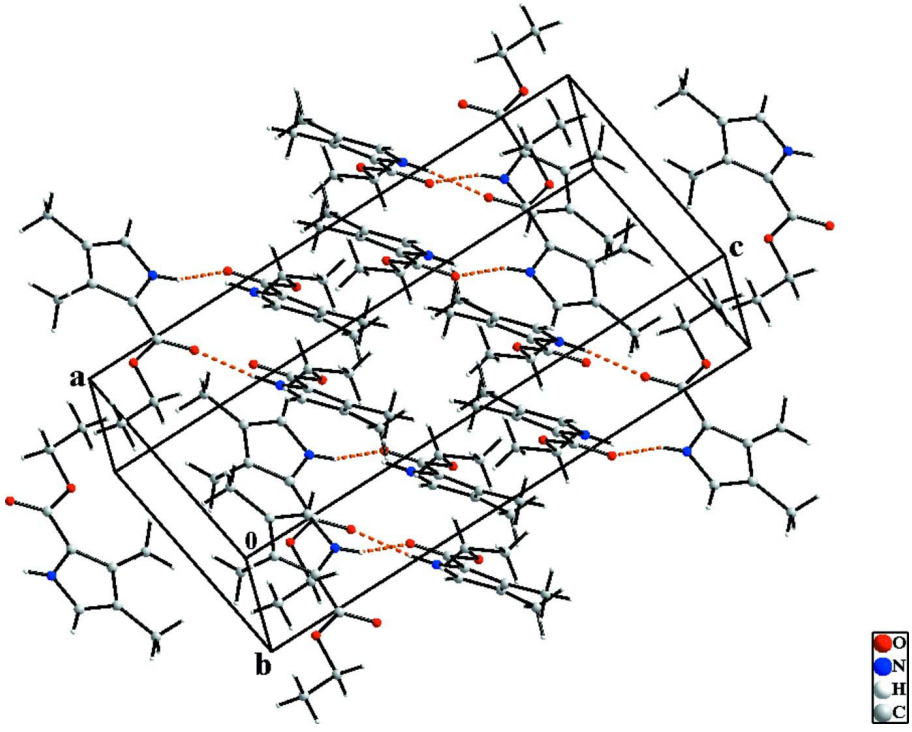

Figure 2

The crystal packing for the title compound *via* N—H···O hydrogen bonds shown as the dashed lines.

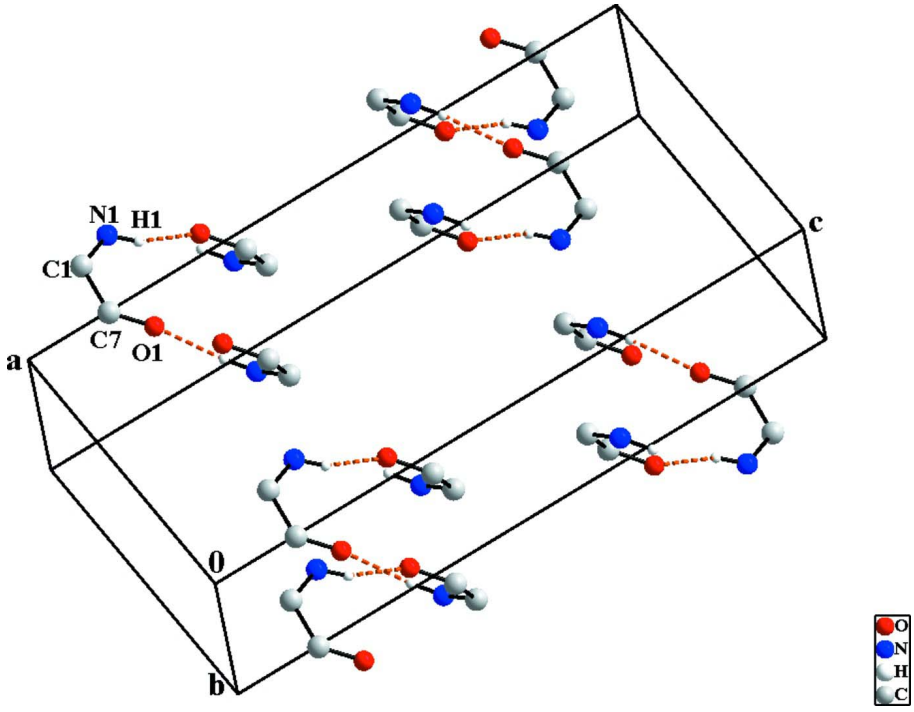

Figure 3

A view showing zig-zag chains with the graph-set motifs C(5) pertinent to the N—H···O hydrogen bonds (the dashed lines) in the title structure. The atoms not involved in this motif have been omitted for clarity.

Ethyl 3,4-dimethyl-1*H*-pyrrole-2-carboxylate

Crystal data

$C_9H_{13}NO_2$
$M_r = 167.20$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.7485\ (2)$ Å
$b = 7.0611\ (2)$ Å
$c = 17.2167\ (5)$ Å
$\beta = 95.103\ (2)°$
$V = 938.24\ (5)$ Å^3
$Z = 4$
$F(000) = 360$
$D_x = 1.184$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3131 reflections
$\theta = 2.4$–$24.8°$
$\mu = 0.08$ mm^{-1}
$T = 296$ K
Block, colourless
0.28 × 0.26 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.977$, $T_{max} = 0.985$
8174 measured reflections
2146 independent reflections
1579 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -9\rightarrow10$
$k = -9\rightarrow9$
$l = -22\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.136$
$S = 1.04$
2146 reflections
112 parameters
0 restraints
49 constraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 0.1432P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ 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}
O2	0.14704 (13)	0.48430 (14)	0.11453 (6)	0.0534 (3)
O1	−0.00527 (14)	0.33077 (15)	0.19987 (7)	0.0615 (3)
N1	0.24389 (16)	0.04205 (17)	0.20998 (7)	0.0498 (3)
H1	0.1597	0.0168	0.2377	0.060*
C2	0.41028 (18)	0.1886 (2)	0.12961 (8)	0.0463 (4)
C7	0.12075 (18)	0.34064 (19)	0.16247 (8)	0.0445 (3)
C1	0.25621 (17)	0.20054 (19)	0.16438 (8)	0.0422 (3)
C8	0.0178 (2)	0.6323 (2)	0.10833 (10)	0.0582 (4)
H8A	0.0125	0.6946	0.1583	0.070*
H8B	−0.0953	0.5796	0.0923	0.070*
C4	0.3843 (2)	−0.0672 (2)	0.20438 (10)	0.0561 (4)
H4	0.4060	−0.1820	0.2299	0.067*
C3	0.49035 (19)	0.0178 (2)	0.15508 (9)	0.0521 (4)
C9	0.0697 (3)	0.7705 (3)	0.04886 (11)	0.0686 (5)
H9A	0.1837	0.8179	0.0644	0.103*
H9B	−0.0111	0.8738	0.0447	0.103*
H9C	0.0699	0.7085	−0.0007	0.103*
C5	0.4845 (2)	0.3287 (3)	0.07667 (11)	0.0702 (5)
H5A	0.3973	0.3650	0.0363	0.105*
H5B	0.5806	0.2725	0.0536	0.105*
H5C	0.5234	0.4386	0.1060	0.105*
C6	0.6620 (2)	−0.0563 (3)	0.13353 (13)	0.0784 (6)
H6A	0.7528	0.0278	0.1533	0.118*
H6B	0.6604	−0.0640	0.0778	0.118*

H6C	0.6820	−0.1799	0.1557	0.118*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0535 (6)	0.0449 (6)	0.0639 (7)	0.0109 (4)	0.0178 (5)	0.0096 (5)
O1	0.0585 (7)	0.0564 (7)	0.0742 (8)	0.0072 (5)	0.0314 (6)	0.0035 (5)
N1	0.0523 (7)	0.0466 (7)	0.0523 (7)	0.0007 (5)	0.0151 (6)	0.0061 (5)
C2	0.0450 (7)	0.0479 (8)	0.0469 (8)	0.0013 (6)	0.0092 (6)	0.0019 (6)
C7	0.0464 (7)	0.0416 (7)	0.0467 (8)	−0.0002 (6)	0.0107 (6)	−0.0038 (6)
C1	0.0433 (7)	0.0403 (7)	0.0440 (7)	0.0001 (5)	0.0091 (6)	0.0021 (6)
C8	0.0595 (9)	0.0451 (8)	0.0711 (10)	0.0133 (7)	0.0119 (8)	0.0018 (8)
C4	0.0615 (9)	0.0468 (8)	0.0595 (9)	0.0088 (7)	0.0025 (7)	0.0079 (7)
C3	0.0462 (8)	0.0545 (9)	0.0560 (9)	0.0084 (6)	0.0059 (7)	−0.0004 (7)
C9	0.0832 (12)	0.0541 (10)	0.0685 (11)	0.0128 (9)	0.0066 (9)	0.0102 (9)
C5	0.0643 (10)	0.0732 (12)	0.0775 (12)	0.0024 (9)	0.0302 (9)	0.0191 (9)
C6	0.0571 (10)	0.0842 (13)	0.0950 (14)	0.0260 (9)	0.0134 (10)	0.0026 (11)

Geometric parameters (Å, º)

O2—C7	1.3347 (17)	C4—C3	1.371 (2)
O2—C8	1.4446 (17)	C4—H4	0.9300
O1—C7	1.2186 (17)	C3—C6	1.506 (2)
N1—C4	1.3440 (19)	C9—H9A	0.9600
N1—C1	1.3752 (17)	C9—H9B	0.9600
N1—H1	0.8600	C9—H9C	0.9600
C2—C1	1.3849 (19)	C5—H5A	0.9600
C2—C3	1.409 (2)	C5—H5B	0.9600
C2—C5	1.494 (2)	C5—H5C	0.9600
C7—C1	1.4406 (19)	C6—H6A	0.9600
C8—C9	1.495 (2)	C6—H6B	0.9600
C8—H8A	0.9700	C6—H6C	0.9600
C8—H8B	0.9700		
C7—O2—C8	116.91 (12)	C4—C3—C2	107.21 (13)
C4—N1—C1	109.16 (12)	C4—C3—C6	126.27 (15)
C4—N1—H1	125.4	C2—C3—C6	126.51 (15)
C1—N1—H1	125.4	C8—C9—H9A	109.5
C1—C2—C3	106.86 (12)	C8—C9—H9B	109.5
C1—C2—C5	128.10 (13)	H9A—C9—H9B	109.5
C3—C2—C5	125.02 (13)	C8—C9—H9C	109.5
O1—C7—O2	122.94 (13)	H9A—C9—H9C	109.5
O1—C7—C1	124.41 (14)	H9B—C9—H9C	109.5
O2—C7—C1	112.65 (12)	C2—C5—H5A	109.5
N1—C1—C2	107.69 (12)	C2—C5—H5B	109.5
N1—C1—C7	118.98 (12)	H5A—C5—H5B	109.5
C2—C1—C7	133.32 (13)	C2—C5—H5C	109.5
O2—C8—C9	107.22 (13)	H5A—C5—H5C	109.5

O2—C8—H8A	110.3	H5B—C5—H5C	109.5
C9—C8—H8A	110.3	C3—C6—H6A	109.5
O2—C8—H8B	110.3	C3—C6—H6B	109.5
C9—C8—H8B	110.3	H6A—C6—H6B	109.5
H8A—C8—H8B	108.5	C3—C6—H6C	109.5
N1—C4—C3	109.09 (14)	H6A—C6—H6C	109.5
N1—C4—H4	125.5	H6B—C6—H6C	109.5
C3—C4—H4	125.5		

C8—O2—C7—O1	0.1 (2)	O1—C7—C1—C2	−177.02 (15)
C8—O2—C7—C1	179.83 (13)	O2—C7—C1—C2	3.3 (2)
C4—N1—C1—C2	−0.19 (16)	C7—O2—C8—C9	−177.04 (13)
C4—N1—C1—C7	−178.84 (13)	C1—N1—C4—C3	−0.06 (18)
C3—C2—C1—N1	0.37 (16)	N1—C4—C3—C2	0.29 (18)
C5—C2—C1—N1	−178.16 (16)	N1—C4—C3—C6	179.24 (16)
C3—C2—C1—C7	178.74 (16)	C1—C2—C3—C4	−0.40 (17)
C5—C2—C1—C7	0.2 (3)	C5—C2—C3—C4	178.18 (16)
O1—C7—C1—N1	1.2 (2)	C1—C2—C3—C6	−179.35 (16)
O2—C7—C1—N1	−178.46 (12)	C5—C2—C3—C6	−0.8 (3)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the N1,C1–C4 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.13	2.9264 (16)	154
C5—H5*A*···O2	0.96	2.60	2.962 (2)	103
C4—H4···*Cg*1^{ii}	0.93	2.92	3.7520 (17)	149
C9—H9*A*···*Cg*1^{iii}	0.96	2.86	3.650 (2)	141

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