

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Ethyl 5-formyl-3,4-dimethyl-1*H*-pyrrole-2-carboxylate

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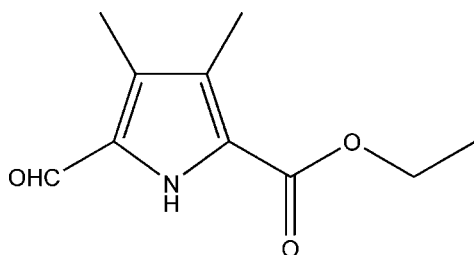
Received 14 May 2009; accepted 17 June 2009

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.060; wR factor = 0.212; data-to-parameter ratio = 18.7.

The molecule of the title compound, $\text{C}_{10}\text{H}_{13}\text{NO}_3$, is approximately planar (maximum deviation 0.1424 Å). In the crystal, molecules are linked into inversion dimers by pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, and the dimeric units are linked by non-classical $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a layered structure.

Related literature

For a related structure, see: Kang *et al.* (2008). For our studies of bis(pyrrol-2-yl-methyleneamine) ligands, see: Wang *et al.* (2008, 2009). For the synthesis, see: Wang *et al.* (2008).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{13}\text{NO}_3$
 $M_r = 195.21$

Triclinic, $P\bar{1}$
 $a = 7.2223$ (12) Å

$b = 7.4347$ (12) Å
 $c = 10.0488$ (17) Å
 $\alpha = 78.412$ (2)°
 $\beta = 84.191$ (2)°
 $\gamma = 79.051$ (2)°
 $V = 517.84$ (15) Å³

 $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 296$ K $0.30 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.980$, $T_{\max} = 0.986$

6232 measured reflections
 2416 independent reflections
 1692 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.212$
 $S = 1.07$
 2416 reflections

129 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1A}\cdots\text{O2}^{\text{i}}$ | 0.86 | 2.07 | 2.919 (2) | 169 |
| $\text{C5}-\text{H5A}\cdots\text{O1}^{\text{ii}}$ | 0.93 | 2.54 | 3.347 (3) | 145 |

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXL97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2066).

References

- Bruker (2001). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Kang, S.-S., Li, H.-L., Zeng, H.-S. & Wang, H.-B. (2008). *Acta Cryst.* **E64**, o1125.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
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 Wang, Y., Wu, W.-N. & Yang, Z.-Y. (2009). *X-ray Struct. Anal. Online*, **25**, 33–34.
 Wang, Y., Yang, Z.-Y. & Chen, Z.-N. (2008). *Bioorg. Med. Chem. Lett.*, **18**, 298–303.

supplementary materials

Acta Cryst. (2009). E65, o1661 [doi:10.1107/S160053680902323X]

Ethyl 5-formyl-3,4-dimethyl-1*H*-pyrrole-2-carboxylate

W.-N. Wu, Y. Wang and Q.-F. Wang

Comment

Schiff Base bearing pyrrole units have been extensively investigated for a long time because they could stabilize mono- or binuclear metal complexes which have various structures and special properties. As part of our ongoing studies of bis(pyrrol-2-yl-methyleneamine) ligands (Wang *et al.*, 2008), we report here the crystal structure of the title compound, (I), (Fig. 1), which is approximately planar.

The molecules are joined to dimers *via* intermolecular N—H···O hydrogen bonds (Table 1), and the dimeric units are linked with each other by nonclassical C—H···O hydrogen bonds (Table 1) to form a layered geometry (Fig. 2).

Experimental

A quantity of POCl₃ (2.30 g, 0.015 mol) was added dropwise to DMF (1.10 g, 0.015 mol) under stirring on an ice-water bath, then a CH₂Cl₂ solution (30 ml) containing 3,4-dimethyl-2-ethoxycarbonyl-pyrrole (2.51 g, 0.015 mol) was added. After stirring at room temperature for 4 h, a 10% Na₂CO₃ solution (80 ml) was added. The reaction mixture was refluxing for 0.5 h, then cooled to room temperature, extracted with CH₂Cl₂ (3×10 ml), and dried with anhydrous Na₂CO₃. The solvent was evaporated under reduced pressure. The crude product was treated with column chromatography on silica gel [petroleum ether-ethyl acetate (100:1)] to yield (I) 2.25 g (77%). Colorless prisms of (I) were obtained by slow evaporation of an ethanol solution.

Refinement

(type here to add refinement details)

Figures

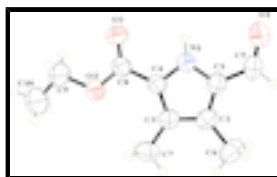


Fig. 1. The molecular structure of the title compound shown with 50% probability displacement ellipsoids.

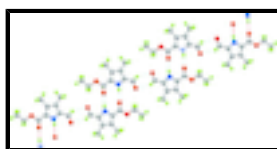


Fig. 2. Layered structure of the title compound. Hydrogen-bonding interactions are shown as dashed lines.

Ethyl 5-formyl-3,4-dimethyl-1H-pyrrole-2-carboxylate

Crystal data

| | |
|---------------------------------|---|
| $C_{10}H_{13}NO_3$ | $Z = 2$ |
| $M_r = 195.21$ | $F_{000} = 208$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.252 \text{ Mg m}^{-3}$ |
| $a = 7.2223 (12) \text{ \AA}$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $b = 7.4347 (12) \text{ \AA}$ | Cell parameters from 2123 reflections |
| $c = 10.0488 (17) \text{ \AA}$ | $\theta = 2.1\text{--}27.7^\circ$ |
| $\alpha = 78.412 (2)^\circ$ | $\mu = 0.09 \text{ mm}^{-1}$ |
| $\beta = 84.191 (2)^\circ$ | $T = 296 \text{ K}$ |
| $\gamma = 79.051 (2)^\circ$ | Prism, colorless |
| $V = 517.84 (15) \text{ \AA}^3$ | $0.30 \times 0.18 \times 0.15 \text{ mm}$ |

Data collection

| | |
|---|--|
| Bruker SMART CCD diffractometer | 2416 independent reflections |
| Radiation source: fine-focus sealed tube | 1692 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.017$ |
| $T = 296 \text{ K}$ | $\theta_{\text{max}} = 27.7^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 2.1^\circ$ |
| Absorption correction: Multi-scan (SADABS; Sheldrick, 1996) | $h = -9 \rightarrow 9$ |
| $T_{\text{min}} = 0.980$, $T_{\text{max}} = 0.986$ | $k = -9 \rightarrow 9$ |
| 6232 measured reflections | $l = -13 \rightarrow 13$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.060$ | H-atom parameters constrained |
| $wR(F^2) = 0.212$ | $w = 1/[\sigma^2(F_o^2) + (0.1226P)^2 + 0.0669P]$ |
| $S = 1.07$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2416 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 129 parameters | $\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$ |
| | Extinction correction: none |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|------------|------------|--------------|----------------------------------|
| C4 | 0.3634 (3) | 0.8003 (2) | 1.03572 (18) | 0.0519 (5) |
| C8 | 0.2197 (3) | 0.8074 (3) | 1.1488 (2) | 0.0593 (5) |
| C3 | 0.5533 (3) | 0.7196 (2) | 1.0262 (2) | 0.0556 (5) |
| C1 | 0.4613 (3) | 0.8683 (3) | 0.8185 (2) | 0.0585 (5) |
| C2 | 0.6149 (3) | 0.7620 (3) | 0.8883 (2) | 0.0605 (5) |
| C5 | 0.4495 (4) | 0.9519 (4) | 0.6766 (2) | 0.0780 (7) |
| H5A | 0.5582 | 0.9328 | 0.6195 | 0.094* |
| C7 | 0.6715 (3) | 0.6084 (3) | 1.1392 (2) | 0.0720 (6) |
| H7A | 0.7978 | 0.5703 | 1.1030 | 0.108* |
| H7B | 0.6181 | 0.5001 | 1.1808 | 0.108* |
| H7C | 0.6741 | 0.6836 | 1.2060 | 0.108* |
| C9 | 0.1496 (4) | 0.7387 (5) | 1.3863 (3) | 0.1023 (10) |
| H9A | 0.1126 | 0.8660 | 1.4008 | 0.123* |
| H9B | 0.0375 | 0.6948 | 1.3693 | 0.123* |
| C10 | 0.2342 (5) | 0.6221 (6) | 1.5051 (3) | 0.1191 (12) |
| H10A | 0.1453 | 0.6265 | 1.5826 | 0.179* |
| H10B | 0.3450 | 0.6663 | 1.5212 | 0.179* |
| H10C | 0.2687 | 0.4960 | 1.4906 | 0.179* |
| C6 | 0.8106 (3) | 0.7039 (4) | 0.8285 (3) | 0.0872 (8) |
| H6A | 0.8884 | 0.6318 | 0.8993 | 0.131* |
| H6B | 0.8631 | 0.8128 | 0.7869 | 0.131* |
| H6C | 0.8059 | 0.6301 | 0.7612 | 0.131* |
| O3 | 0.2874 (2) | 0.7315 (2) | 1.26943 (15) | 0.0771 (5) |
| O2 | 0.0572 (2) | 0.8726 (3) | 1.13418 (17) | 0.0899 (6) |
| O1 | 0.3095 (3) | 1.0448 (3) | 0.62612 (17) | 0.1053 (7) |
| N1 | 0.3105 (2) | 0.8889 (2) | 0.90931 (15) | 0.0539 (4) |
| H1A | 0.1993 | 0.9484 | 0.8903 | 0.065* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| C4 | 0.0506 (9) | 0.0554 (9) | 0.0481 (10) | -0.0087 (7) | -0.0052 (7) | -0.0051 (7) |
| C8 | 0.0550 (11) | 0.0682 (11) | 0.0493 (10) | -0.0082 (8) | -0.0053 (8) | 0.0004 (8) |
| C3 | 0.0517 (10) | 0.0526 (9) | 0.0619 (11) | -0.0072 (7) | -0.0068 (8) | -0.0095 (8) |
| C1 | 0.0601 (11) | 0.0638 (11) | 0.0518 (11) | -0.0101 (8) | 0.0033 (8) | -0.0155 (8) |
| C2 | 0.0555 (11) | 0.0608 (10) | 0.0652 (12) | -0.0072 (8) | 0.0024 (9) | -0.0180 (9) |
| C5 | 0.0779 (15) | 0.1027 (17) | 0.0473 (12) | -0.0053 (13) | 0.0084 (11) | -0.0156 (11) |

supplementary materials

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C7 | 0.0584 (12) | 0.0679 (12) | 0.0837 (15) | 0.0005 (9) | -0.0165 (10) | -0.0048 (10) |
| C9 | 0.0778 (16) | 0.148 (3) | 0.0571 (14) | -0.0005 (16) | 0.0093 (12) | 0.0119 (14) |
| C10 | 0.108 (2) | 0.161 (3) | 0.0668 (17) | -0.010 (2) | 0.0026 (16) | 0.0126 (18) |
| C6 | 0.0651 (14) | 0.0912 (16) | 0.0983 (19) | -0.0010 (12) | 0.0157 (13) | -0.0233 (14) |
| O3 | 0.0625 (9) | 0.1094 (12) | 0.0475 (8) | -0.0070 (8) | -0.0043 (7) | 0.0061 (7) |
| O2 | 0.0552 (9) | 0.1288 (15) | 0.0616 (10) | 0.0092 (9) | 0.0013 (7) | 0.0126 (9) |
| O1 | 0.0927 (14) | 0.1528 (19) | 0.0505 (10) | 0.0128 (12) | -0.0006 (9) | -0.0065 (10) |
| N1 | 0.0507 (8) | 0.0637 (9) | 0.0445 (8) | -0.0055 (7) | -0.0028 (6) | -0.0083 (6) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|---------------|--------------|
| C4—N1 | 1.364 (2) | C7—H7B | 0.9600 |
| C4—C3 | 1.390 (3) | C7—H7C | 0.9600 |
| C4—C8 | 1.461 (3) | C9—C10 | 1.443 (4) |
| C8—O2 | 1.194 (2) | C9—O3 | 1.463 (3) |
| C8—O3 | 1.330 (2) | C9—H9A | 0.9700 |
| C3—C2 | 1.405 (3) | C9—H9B | 0.9700 |
| C3—C7 | 1.500 (3) | C10—H10A | 0.9600 |
| C1—N1 | 1.354 (2) | C10—H10B | 0.9600 |
| C1—C2 | 1.396 (3) | C10—H10C | 0.9600 |
| C1—C5 | 1.440 (3) | C6—H6A | 0.9600 |
| C2—C6 | 1.498 (3) | C6—H6B | 0.9600 |
| C5—O1 | 1.206 (3) | C6—H6C | 0.9600 |
| C5—H5A | 0.9300 | N1—H1A | 0.8600 |
| C7—H7A | 0.9600 | | |
| N1—C4—C3 | 108.93 (17) | H7B—C7—H7C | 109.5 |
| N1—C4—C8 | 117.47 (17) | C10—C9—O3 | 108.7 (3) |
| C3—C4—C8 | 133.61 (18) | C10—C9—H9A | 109.9 |
| O2—C8—O3 | 123.29 (19) | O3—C9—H9A | 109.9 |
| O2—C8—C4 | 123.38 (19) | C10—C9—H9B | 109.9 |
| O3—C8—C4 | 113.32 (17) | O3—C9—H9B | 109.9 |
| C4—C3—C2 | 106.33 (17) | H9A—C9—H9B | 108.3 |
| C4—C3—C7 | 127.64 (19) | C9—C10—H10A | 109.5 |
| C2—C3—C7 | 126.03 (19) | C9—C10—H10B | 109.5 |
| N1—C1—C2 | 108.25 (17) | H10A—C10—H10B | 109.5 |
| N1—C1—C5 | 121.70 (19) | C9—C10—H10C | 109.5 |
| C2—C1—C5 | 130.0 (2) | H10A—C10—H10C | 109.5 |
| C1—C2—C3 | 107.43 (18) | H10B—C10—H10C | 109.5 |
| C1—C2—C6 | 126.8 (2) | C2—C6—H6A | 109.5 |
| C3—C2—C6 | 125.8 (2) | C2—C6—H6B | 109.5 |
| O1—C5—C1 | 125.0 (2) | H6A—C6—H6B | 109.5 |
| O1—C5—H5A | 117.5 | C2—C6—H6C | 109.5 |
| C1—C5—H5A | 117.5 | H6A—C6—H6C | 109.5 |
| C3—C7—H7A | 109.5 | H6B—C6—H6C | 109.5 |
| C3—C7—H7B | 109.5 | C8—O3—C9 | 115.39 (18) |
| H7A—C7—H7B | 109.5 | C1—N1—C4 | 109.06 (16) |
| C3—C7—H7C | 109.5 | C1—N1—H1A | 125.5 |
| H7A—C7—H7C | 109.5 | C4—N1—H1A | 125.5 |
| N1—C4—C8—O2 | 6.4 (3) | C7—C3—C2—C1 | -179.66 (18) |

| | | | |
|-------------|--------------|--------------|--------------|
| C3—C4—C8—O2 | -173.7 (2) | C4—C3—C2—C6 | -179.9 (2) |
| N1—C4—C8—O3 | -174.52 (16) | C7—C3—C2—C6 | -0.1 (3) |
| C3—C4—C8—O3 | 5.4 (3) | N1—C1—C5—O1 | -0.2 (4) |
| N1—C4—C3—C2 | -0.1 (2) | C2—C1—C5—O1 | -178.7 (2) |
| C8—C4—C3—C2 | 179.9 (2) | O2—C8—O3—C9 | -2.1 (4) |
| N1—C4—C3—C7 | -179.99 (18) | C4—C8—O3—C9 | 178.8 (2) |
| C8—C4—C3—C7 | 0.1 (3) | C10—C9—O3—C8 | 169.4 (3) |
| N1—C1—C2—C3 | -0.7 (2) | C2—C1—N1—C4 | 0.6 (2) |
| C5—C1—C2—C3 | 178.0 (2) | C5—C1—N1—C4 | -178.23 (18) |
| N1—C1—C2—C6 | 179.8 (2) | C3—C4—N1—C1 | -0.3 (2) |
| C5—C1—C2—C6 | -1.5 (4) | C8—C4—N1—C1 | 179.68 (15) |
| C4—C3—C2—C1 | 0.5 (2) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i> | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1A···O2 ⁱ | 0.86 | 2.07 | 2.919 (2) | 169 |
| C5—H5A···O1 ⁱⁱ | 0.93 | 2.54 | 3.347 (3) | 145 |

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

Fig. 1

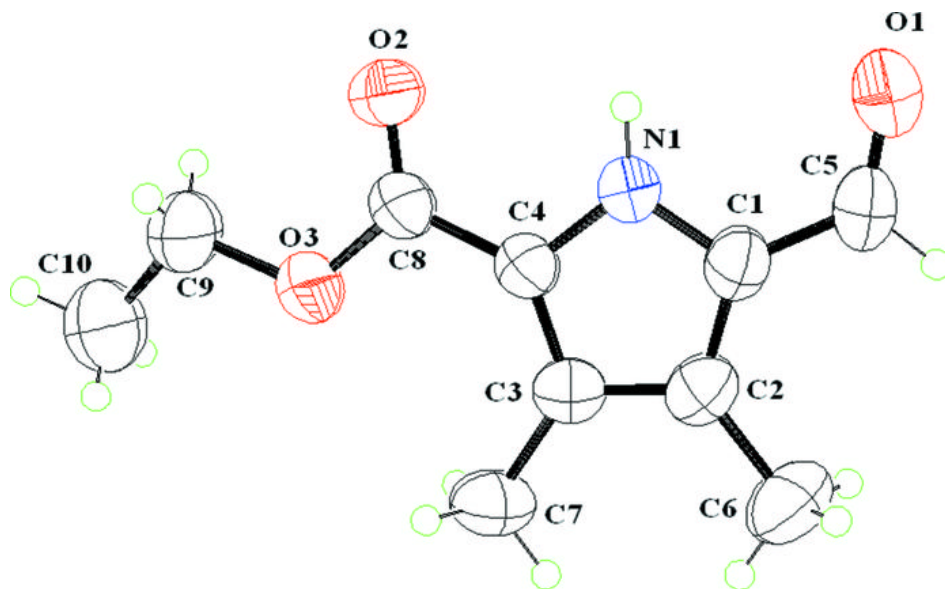


Fig. 2

