

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Ethyl (*E*)-2-(2-furylidene)hydrazine-carboxylateLu-Ping Lv,^a Wei-Wei Li,^a Tie-Ming Yu,^a Wen-Bo Yu^a and Xian-Chao Hu^{b*}^aDepartment of Chemical Engineering, Hangzhou Vocational and Technical College, Hangzhou 310018, People's Republic of China, and ^bResearch Center of Analysis and Measurement, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: zgdxhc@126.com

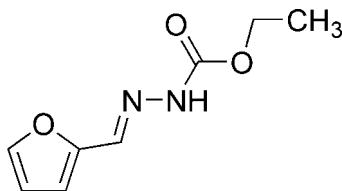
Received 14 June 2009; accepted 19 June 2009

Key indicators: single-crystal X-ray study; $T = 223$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.028; wR factor = 0.071; data-to-parameter ratio = 6.9.

In the title compound, $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_3$, the hydrazinecarboxylate group is twisted from the furan ring by 6.98 (17)°. In the crystal, the molecules are linked into one-dimensional chains running along the c axis by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987); Borg *et al.* (1999); Kahwa *et al.* (1986); Santos *et al.* (2001). For a related structure, see: Shang *et al.* (2007).



Experimental

Crystal data

$\text{C}_8\text{H}_{10}\text{N}_2\text{O}_3$
 $M_r = 182.18$
 Monoclinic, Cc
 $a = 14.150$ (6) Å
 $b = 9.285$ (5) Å

$c = 8.108$ (4) Å
 $\beta = 118.540$ (16)°
 $V = 935.8$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 223$ K

0.24 × 0.22 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.975$, $T_{\max} = 0.985$

2344 measured reflections
 816 independent reflections
 733 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.071$
 $S = 1.07$
 816 reflections

118 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.10$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------------|-------|-------------|-------------|---------------|
| $\text{N2}-\text{H2}\cdots\text{O2}^i$ | 0.86 | 2.08 | 2.916 (3) | 164 |

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); 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 from the Zhejiang University of Technology Foundation (grant No. 20080169) and the Analysis and Measurement Foundation of Zhejiang Province (grant No. 2008 F70003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2272).

References

- Borg, S., Vollinga, R. C., Labarre, M., Payza, K., Terenius, L. & Luthman, K. (1999). *J. Med. Chem.* **42**, 4331–4342.
 Bruker (2002). *SADABS, SMART and SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Hadjoudis, E., Vittorakis, M. & Moustakali-Mavridis, J. (1987). *Tetrahedron*, **43**, 1345–1360.
 Kahwa, I. A., Selbin, J., Hsieh, T. Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **151**, 201–208.
 Parashar, R. K., Sharma, R. C., Kumar, A. & Mohanm, G. (1988). *Inorg. Chim. Acta*, **151**, 201–208.
 Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
 Shang, Z.-H., Zhang, H.-L. & Ding, Y. (2007). *Acta Cryst.* **E63**, o3394.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2009). E65, o1678 [doi:10.1107/S1600536809023654]

Ethyl (*E*)-2-(2-furylidene)hydrazinecarboxylate

Lu-Ping Lv, Wei-Wei Li, Tie-Ming Yu, Wen-Bo Yu and Xian-Chao Hu

S1. Comment

Benzaldehydhydrazone derivatives have attracted much attention due to their pharmacological activity (Parashar *et al.*, 1988) and their photochromic properties (Hadjoudis *et al.*, 1987). They are important intermediates of 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many interesting properties (Borg *et al.*, 1999). Metal complexes based on Schiff bases have received considerable attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). We report here the crystal structure of the title compound (Fig. 1).

In the title compound, C₈H₁₀N₂O₃ (I), the N1/N2/O2/O3/C6/C7 planes form dihedral angles of 6.98 (17)° with the O1/C1—C4 planes. The bond lengths and angles are comparable to those observed for methyl*N*-[(*E*)-4-methoxybenzylidene]hydrazinecarboxylate (Shang *et al.*, 2007).

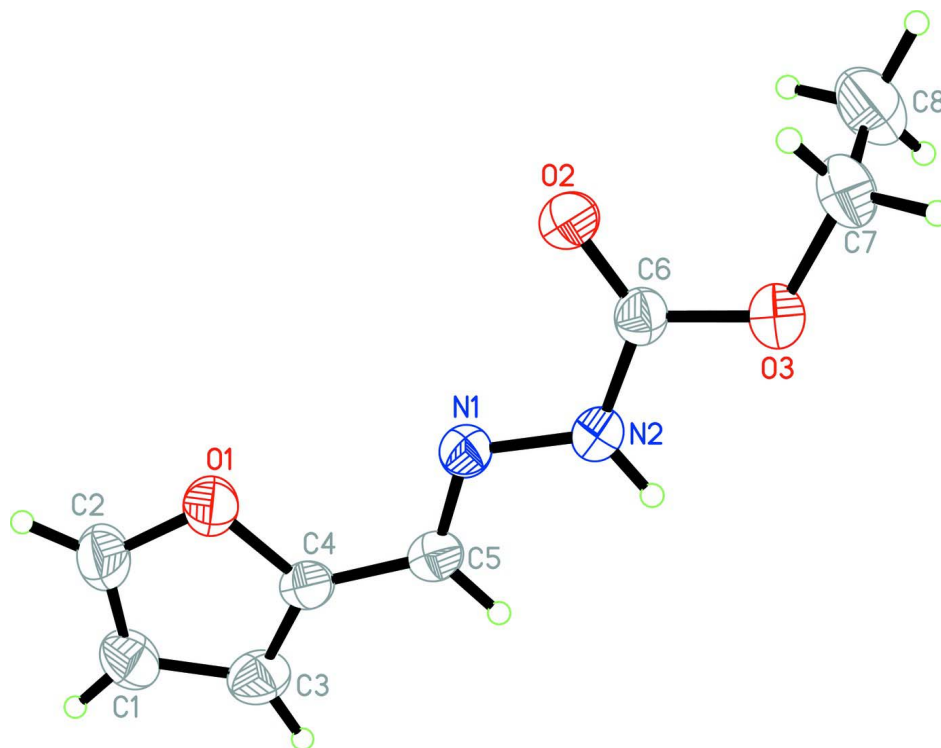
In the crystal structure, the molecules are linked into one-dimensional chains running along the *c* axis by N—H···O hydrogen bonds (Table 1, Fig. 1).

S2. Experimental

Furfuraldehyde (0.96 g, 0.01 mol) and Ethyl hydrazinecarboxylate (1.04 g, 0.01 mol) were dissolved in stirred methanol (20 ml) and left for 3 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 95% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 410–412 K).

S3. Refinement

H atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93 or 0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

**Figure 1**

The asymmetric unit of (I). Displacement ellipsoids are drawn at the 40% probability level.

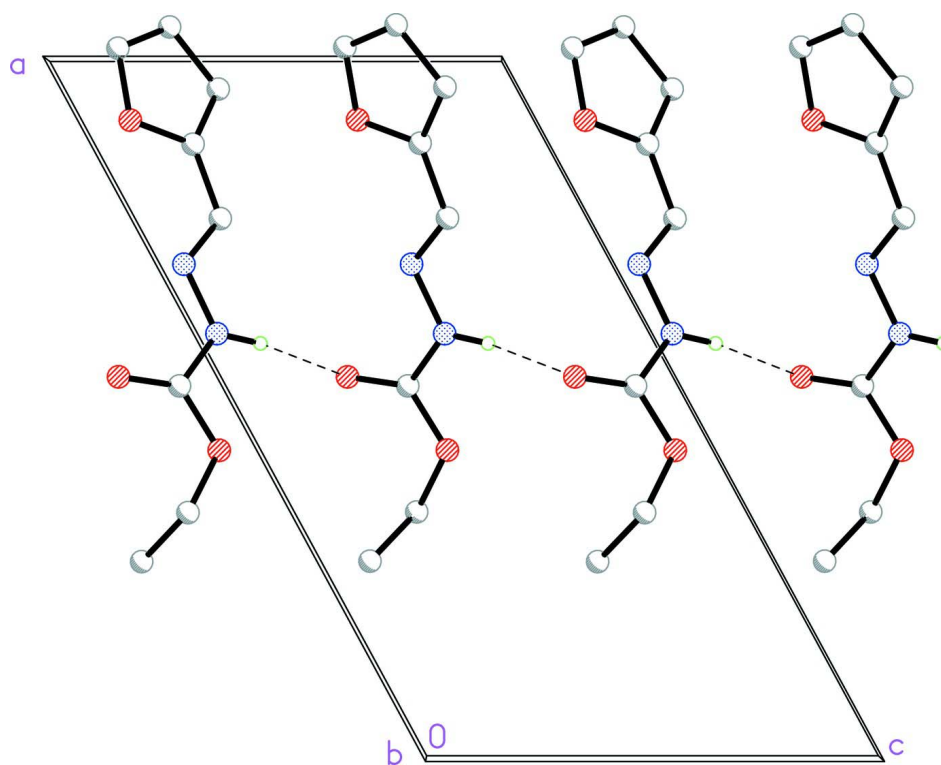


Figure 2

Crystal packing of (I), showing the formation of chains along *c*. Hydrogen bonds are shown as dashed lines.

Ethyl (*E*)-2-(2-furylidene)hydrazinecarboxylate

Crystal data

$C_8H_{10}N_2O_3$

$M_r = 182.18$

Monoclinic, *Cc*

Hall symbol: *C* -2_{yc}

$a = 14.150$ (6) Å

$b = 9.285$ (5) Å

$c = 8.108$ (4) Å

$\beta = 118.540$ (16)°

$V = 935.8$ (8) Å³

$Z = 4$

$F(000) = 384$

$D_x = 1.293$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1451 reflections

$\theta = 2.7$ – 25.0°

$\mu = 0.10$ mm⁻¹

$T = 223$ K

Block, colourless

$0.24 \times 0.22 \times 0.17$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2002)

$T_{\min} = 0.975$, $T_{\max} = 0.985$

2344 measured reflections

816 independent reflections

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

$R_{\text{int}} = 0.024$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.7^\circ$

$h = -16 \rightarrow 16$

$k = -10 \rightarrow 10$

$l = -8 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.071$

$S = 1.07$

816 reflections

118 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.0727P]$

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

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

$\Delta\rho_{\max} = 0.10$ e Å⁻³

$\Delta\rho_{\min} = -0.16$ 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 (Å²)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|---------------|-------------|------------|----------------------------------|
| O3 | -0.05924 (14) | -0.0975 (2) | 0.4141 (2) | 0.0686 (5) |

| | | | | |
|-----|--------------|-------------|------------|-------------|
| N2 | 0.10734 (15) | -0.0174 (2) | 0.5472 (3) | 0.0561 (5) |
| H2 | 0.0934 | 0.0188 | 0.6309 | 0.067* |
| O1 | 0.41276 (15) | 0.0404 (2) | 0.6109 (3) | 0.0754 (6) |
| O2 | 0.04565 (14) | -0.1545 (2) | 0.2815 (2) | 0.0654 (5) |
| N1 | 0.20660 (15) | 0.0030 (2) | 0.5573 (3) | 0.0527 (5) |
| C3 | 0.4583 (2) | 0.1980 (3) | 0.8408 (4) | 0.0702 (8) |
| H3 | 0.4555 | 0.2580 | 0.9302 | 0.084* |
| C5 | 0.27221 (19) | 0.0837 (3) | 0.6911 (3) | 0.0541 (6) |
| H5 | 0.2503 | 0.1248 | 0.7719 | 0.065* |
| C6 | 0.03274 (18) | -0.0952 (3) | 0.4038 (3) | 0.0531 (6) |
| C4 | 0.37893 (19) | 0.1127 (2) | 0.7200 (3) | 0.0533 (6) |
| C7 | -0.1479 (2) | -0.1819 (4) | 0.2726 (4) | 0.0785 (8) |
| H7A | -0.1895 | -0.2234 | 0.3273 | 0.094* |
| H7B | -0.1196 | -0.2601 | 0.2299 | 0.094* |
| C1 | 0.5472 (2) | 0.1804 (3) | 0.8079 (4) | 0.0745 (8) |
| H1 | 0.6138 | 0.2259 | 0.8705 | 0.089* |
| C2 | 0.5161 (2) | 0.0859 (4) | 0.6694 (5) | 0.0822 (9) |
| H2A | 0.5589 | 0.0544 | 0.6183 | 0.099* |
| C8 | -0.2181 (3) | -0.0897 (5) | 0.1115 (5) | 0.1058 (12) |
| H8A | -0.2758 | -0.1465 | 0.0190 | 0.159* |
| H8B | -0.1769 | -0.0492 | 0.0571 | 0.159* |
| H8C | -0.2473 | -0.0135 | 0.1537 | 0.159* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| O3 | 0.0582 (10) | 0.0793 (13) | 0.0731 (12) | -0.0042 (9) | 0.0354 (9) | -0.0022 (10) |
| N2 | 0.0546 (12) | 0.0655 (13) | 0.0533 (11) | 0.0001 (10) | 0.0299 (9) | -0.0041 (10) |
| O1 | 0.0585 (10) | 0.0904 (12) | 0.0779 (12) | -0.0088 (10) | 0.0329 (10) | -0.0277 (11) |
| O2 | 0.0669 (10) | 0.0755 (11) | 0.0593 (10) | -0.0093 (9) | 0.0347 (9) | -0.0106 (9) |
| N1 | 0.0517 (11) | 0.0574 (11) | 0.0510 (11) | 0.0013 (9) | 0.0261 (9) | 0.0025 (9) |
| C3 | 0.081 (2) | 0.0659 (17) | 0.0638 (16) | -0.0113 (15) | 0.0347 (15) | -0.0160 (13) |
| C5 | 0.0610 (14) | 0.0538 (14) | 0.0474 (13) | 0.0032 (13) | 0.0259 (11) | 0.0004 (12) |
| C6 | 0.0517 (13) | 0.0586 (15) | 0.0521 (14) | 0.0045 (11) | 0.0273 (11) | 0.0110 (12) |
| C4 | 0.0594 (14) | 0.0537 (13) | 0.0451 (12) | 0.0034 (11) | 0.0236 (11) | -0.0002 (11) |
| C7 | 0.0616 (16) | 0.082 (2) | 0.092 (2) | -0.0168 (16) | 0.0366 (16) | -0.0043 (17) |
| C1 | 0.0593 (14) | 0.0745 (19) | 0.0787 (19) | -0.0163 (14) | 0.0241 (14) | -0.0068 (15) |
| C2 | 0.0567 (15) | 0.099 (2) | 0.095 (2) | -0.0099 (15) | 0.0392 (15) | -0.0218 (19) |
| C8 | 0.0683 (19) | 0.126 (3) | 0.103 (3) | -0.012 (2) | 0.0241 (19) | 0.006 (2) |

Geometric parameters (Å, °)

| | | | |
|-------|-----------|--------|-----------|
| O3—C6 | 1.344 (3) | C5—C4 | 1.438 (4) |
| O3—C7 | 1.459 (3) | C5—H5 | 0.9300 |
| N2—C6 | 1.347 (3) | C7—C8 | 1.479 (5) |
| N2—N1 | 1.380 (2) | C7—H7A | 0.9700 |
| N2—H2 | 0.8600 | C7—H7B | 0.9700 |
| O1—C2 | 1.371 (4) | C1—C2 | 1.324 (4) |

| | | | |
|----------|-------------|------------|-----------|
| O1—C4 | 1.366 (3) | C1—H1 | 0.9300 |
| O2—C6 | 1.221 (3) | C2—H2A | 0.9300 |
| N1—C5 | 1.279 (3) | C8—H8A | 0.9600 |
| C3—C4 | 1.341 (4) | C8—H8B | 0.9600 |
| C3—C1 | 1.415 (4) | C8—H8C | 0.9600 |
| C3—H3 | 0.9300 | | |
| | | | |
| C6—O3—C7 | 117.0 (2) | O3—C7—C8 | 110.2 (3) |
| C6—N2—N1 | 118.71 (18) | O3—C7—H7A | 109.6 |
| C6—N2—H2 | 120.6 | C8—C7—H7A | 109.6 |
| N1—N2—H2 | 120.6 | O3—C7—H7B | 109.6 |
| C2—O1—C4 | 105.7 (2) | C8—C7—H7B | 109.6 |
| C5—N1—N2 | 115.90 (18) | H7A—C7—H7B | 108.1 |
| C4—C3—C1 | 107.7 (2) | C2—C1—C3 | 105.7 (2) |
| C4—C3—H3 | 126.1 | C2—C1—H1 | 127.1 |
| C1—C3—H3 | 126.1 | C3—C1—H1 | 127.1 |
| N1—C5—C4 | 121.9 (2) | C1—C2—O1 | 111.5 (3) |
| N1—C5—H5 | 119.1 | C1—C2—H2A | 124.3 |
| C4—C5—H5 | 119.1 | O1—C2—H2A | 124.3 |
| O2—C6—O3 | 124.6 (2) | C7—C8—H8A | 109.5 |
| O2—C6—N2 | 125.6 (2) | C7—C8—H8B | 109.5 |
| O3—C6—N2 | 109.7 (2) | H8A—C8—H8B | 109.5 |
| C3—C4—O1 | 109.4 (2) | C7—C8—H8C | 109.5 |
| C3—C4—C5 | 132.7 (2) | H8A—C8—H8C | 109.5 |
| O1—C4—C5 | 117.9 (2) | H8B—C8—H8C | 109.5 |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H2 \cdots O2 ⁱ | 0.86 | 2.08 | 2.916 (3) | 164 |

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