Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Ethoxycarbonyl-3-furoic acid

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.098
Data-to-parameter ratio = 16.4

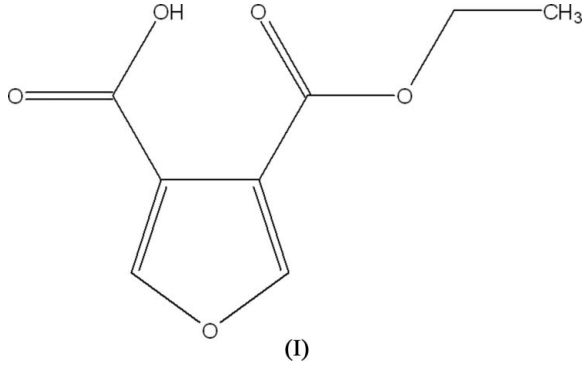
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 February 2006
Accepted 6 February 2006

The structure of 4-ethoxycarbonyl-3-furoic acid, $C_8H_8O_5$, has been determined at 100 K.

Comment

The title compound, (I), represents the first example of a crystal structure of a mono-ester of furoic acid. Previous related crystal structures observed in the Cambridge Structural Database (CSD, Version 5.26; Allen, 2002) include the cyclohexane (CSD refcode CIHNIQ; Baldwin *et al.*, 1997) and methyl (CSD refcode FURCAM; Okada *et al.*, 1971) diesters, and the diacid (CSD refcode FURDCB and derivatives), although there has been some discussion as to the correct space group of this compound (Williams & Rundle, 1964; Semmingsen *et al.*, 1986).



(I)

The molecular structure of the title compound is essentially flat, with all the non-H atoms coplanar. No intermolecular hydrogen bonding is observed, although there are a number of

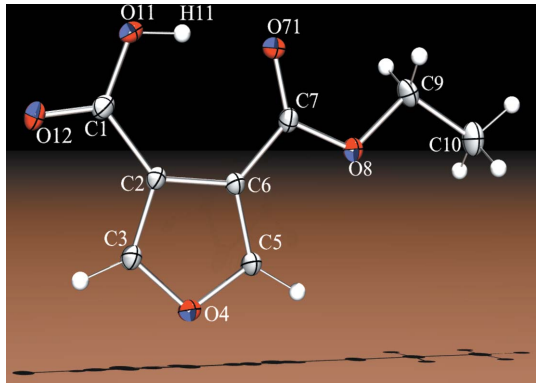


Figure 1
The molecular structure of (I) observed in the crystal structure, showing anisotropic displacement parameter ellipsoids and the numbering scheme used. The displacement ellipsoids are drawn at the 50% probability level.

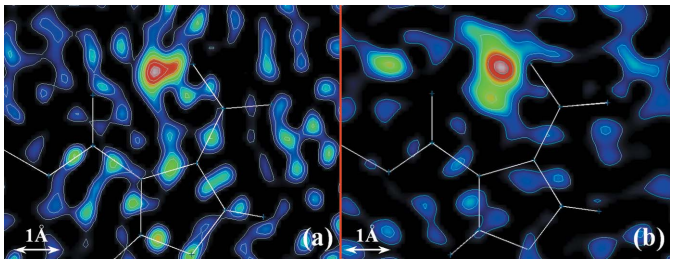


Figure 2
Difference Fourier map sections through the intramolecular hydrogen bond in the same crystal at (*a*) 100 K and (*b*) 293 K.

non-classical C—H···O interactions observed between molecules. The single hydrogen bond observed in the title structure is an intramolecular O11—H11···O71 hydrogen bond (Table 2 and Fig. 1), lying approximately perpendicular to the (021) plane. Much of our interest in such materials lies in the possibility of hydrogen-bond disorder. The difference Fourier map (Fig. 2) shows that no disorder is observed in this hydrogen bond at this temperature; there is also no disorder observed in the hydrogen bond at higher temperatures, as a difference Fourier map of the hydrogen bond from a data set collected at 293 K on the same crystal shows (Fig. 2). The data for the 293 K structure have been deposited with the Cambridge Crystallographic Data Centre.

The packing of the molecule in this structure is layered, with each layer having rows of the title molecule in alternating directions (Fig. 3*a*). The layers are then superimposed on each other, with alternating rows lying on top of one another (Fig. 3*b*). These layers are quite distinct throughout the structure (Fig. 3*c*).

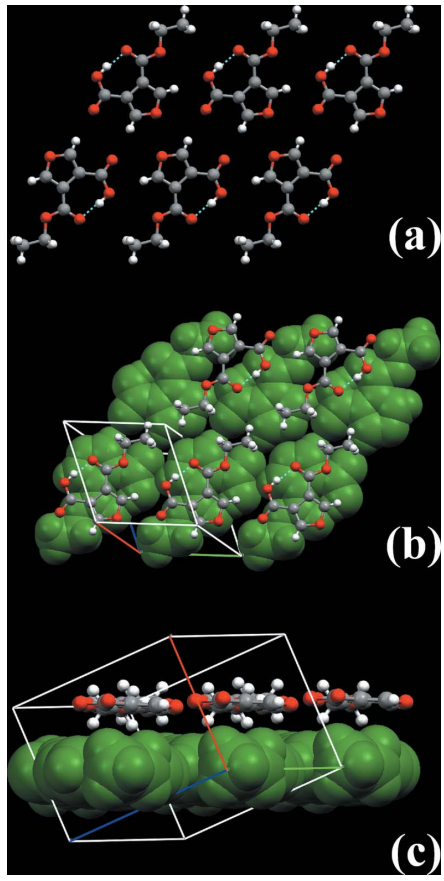


Figure 3
Packing diagrams showing (*a*) the layered structure of the molecule, (*b*) how the layers fit together and (*c*) a side view of the layers.

Experimental

The title material was prepared from diethyl-3,4-furandicarboxylate after exposure to moist air, the crystals being observed floating in the parent material a few days after exposure.

Crystal data

$C_8H_8O_5$
M_r = 184.15
Triclinic, $P\overline{1}$
a = 7.0424 (11) Å
b = 7.4653 (12) Å
c = 9.0724 (14) Å
α = 111.236 (4)°
β = 93.207 (5)°
γ = 109.601 (4)°
V = 410.18 (11) Å^3
Z = 2
D_x = 1.491 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2341 reflections
θ = 2–31°
μ = 0.13 mm^{-1}
T = 100 K
Block, colourless
0.50 × 0.40 × 0.20 mm

Data collection

Brüker APEX2 CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1996)
T_{min} = 0.91, T_{max} = 0.98
5968 measured reflections
2461 independent reflections
1763 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 30.7°
$h = -10 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.098
S = 0.93
2461 reflections
150 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F^2) + 0.05]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—C2	1.4527 (18)	C3—O4	1.3713 (16)
C6—C5	1.3621 (19)	O4—C5	1.3634 (17)
C6—C7	1.4669 (19)	C7—O8	1.3311 (16)
C2—C1	1.4874 (18)	C7—O71	1.2289 (16)
C2—C3	1.3557 (19)	O8—C9	1.4635 (17)
C1—O12	1.2182 (17)	C9—C10	1.506 (2)
C1—O11	1.3327 (17)		
C2—C6—C5	106.07 (12)	C2—C3—O4	110.72 (12)
C2—C6—C7	129.92 (12)	C3—O4—C5	106.99 (10)
C5—C6—C7	124.01 (12)	O4—C5—C6	110.44 (12)
C6—C2—C1	132.51 (12)	C6—C7—O8	111.88 (11)
C6—C2—C3	105.77 (11)	C6—C7—O71	124.32 (12)
C1—C2—C3	121.70 (12)	O8—C7—O71	123.79 (12)
C2—C1—O12	121.56 (12)	C7—O8—C9	116.73 (11)
C2—C1—O11	118.18 (12)	O8—C9—C10	107.16 (12)
O12—C1—O11	120.26 (12)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O11—H11···O71	0.94 (2)	1.70 (2)	2.6267 (16)	172 (2)

All H atoms were found in difference density syntheses and were refined isotropically without restraints.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *MERCURY* (Bruno *et al.*, 2002), *ORTEP-3 for Windows* (Farrugia, 1997) and *WinGX* (Farrugia, 1999); software used to prepare material for publication: *CRYSTALS*.

The authors thank the Nuffield Foundation for funding for JLK.

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supporting information

Acta Cryst. (2006). E**62**, o987–o989 [https://doi.org/10.1107/S1600536806004429]

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$\beta = 93.207$ (5)°
$\gamma = 109.601$ (4)°
$V = 410.18$ (11) Å^3
$Z = 2$
$F(000) = 192$
$D_x = 1.491$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2341 reflections
$\theta = 2$–31°
$\mu = 0.13$ mm^{-1}
$T = 100$ K
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5968 measured reflections
2461 independent reflections
1763 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 30.7°$, $\theta_{min} = 2.5°$
$h = -10\rightarrow9$
$k = -10\rightarrow10$
$l = -13\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.098$
$S = 0.93$
2461 reflections
150 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F^2) + 0.05]$
$(\Delta/\sigma)_{max} = 0.000192$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C6	0.70076 (19)	0.1024 (2)	0.41479 (16)	0.0154
C2	0.65366 (19)	−0.1080 (2)	0.29745 (16)	0.0153
C1	0.6760 (2)	−0.2927 (2)	0.31147 (17)	0.0178
O12	0.62826 (16)	−0.45688 (15)	0.19436 (13)	0.0236
O11	0.74836 (16)	−0.27534 (16)	0.45702 (13)	0.0216

C3	0.5715 (2)	−0.1164 (2)	0.15520 (17)	0.0178
O4	0.56311 (16)	0.07133 (15)	0.17399 (12)	0.0209
C5	0.6426 (2)	0.2022 (2)	0.33238 (16)	0.0191
C7	0.7916 (2)	0.1998 (2)	0.58785 (16)	0.0164
O8	0.81253 (15)	0.39804 (15)	0.65420 (12)	0.0205
C9	0.9021 (2)	0.5115 (2)	0.82699 (18)	0.0227
C10	0.9139 (3)	0.7307 (2)	0.8735 (2)	0.0276
O71	0.84080 (15)	0.10958 (15)	0.66351 (12)	0.0199
H31	0.517 (2)	−0.230 (3)	0.049 (2)	0.022 (4)*
H51	0.654 (2)	0.346 (3)	0.3662 (19)	0.019 (4)*
H91	1.036 (3)	0.504 (3)	0.844 (2)	0.024 (4)*
H92	0.811 (2)	0.442 (3)	0.881 (2)	0.022 (4)*
H101	0.976 (3)	0.819 (3)	0.990 (3)	0.044 (6)*
H102	1.010 (3)	0.804 (3)	0.817 (2)	0.039 (5)*
H103	0.780 (3)	0.733 (3)	0.857 (2)	0.035 (5)*
H11	0.781 (3)	−0.143 (3)	0.538 (3)	0.052 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0192 (6)	0.0132 (6)	0.0130 (6)	0.0059 (5)	0.0026 (5)	0.0048 (5)
C2	0.0190 (6)	0.0122 (6)	0.0153 (6)	0.0069 (5)	0.0036 (5)	0.0052 (5)
C1	0.0187 (6)	0.0151 (6)	0.0208 (7)	0.0075 (5)	0.0033 (5)	0.0080 (5)
O12	0.0316 (5)	0.0137 (5)	0.0225 (6)	0.0105 (4)	0.0007 (4)	0.0032 (4)
O11	0.0318 (5)	0.0168 (5)	0.0190 (5)	0.0121 (4)	0.0009 (4)	0.0082 (4)
C3	0.0238 (7)	0.0137 (6)	0.0160 (7)	0.0087 (5)	0.0040 (5)	0.0048 (5)
O4	0.0333 (5)	0.0165 (5)	0.0142 (5)	0.0118 (4)	0.0014 (4)	0.0062 (4)
C5	0.0288 (7)	0.0140 (6)	0.0137 (7)	0.0089 (5)	0.0021 (5)	0.0042 (5)
C7	0.0182 (6)	0.0132 (6)	0.0164 (7)	0.0055 (5)	0.0037 (5)	0.0047 (5)
O8	0.0296 (5)	0.0137 (5)	0.0147 (5)	0.0089 (4)	−0.0007 (4)	0.0022 (4)
C9	0.0266 (7)	0.0195 (7)	0.0145 (7)	0.0070 (6)	−0.0002 (6)	0.0012 (6)
C10	0.0299 (8)	0.0187 (7)	0.0251 (8)	0.0073 (6)	0.0049 (6)	0.0009 (6)
O71	0.0252 (5)	0.0178 (5)	0.0165 (5)	0.0091 (4)	−0.0006 (4)	0.0067 (4)

Geometric parameters (Å, º)

C6—C2	1.4527 (18)	C5—H51	0.978 (16)
C6—C5	1.3621 (19)	C7—O8	1.3311 (16)
C6—C7	1.4669 (19)	C7—O71	1.2289 (16)
C2—C1	1.4874 (18)	O8—C9	1.4635 (17)
C2—C3	1.3557 (19)	C9—C10	1.506 (2)
C1—O12	1.2182 (17)	C9—H91	0.971 (17)
C1—O11	1.3327 (17)	C9—H92	0.957 (16)
O11—H11	0.94 (2)	C10—H101	1.00 (2)
C3—O4	1.3713 (16)	C10—H102	1.004 (19)
C3—H31	0.971 (17)	C10—H103	0.955 (19)
O4—C5	1.3634 (17)		

C2—C6—C5	106.07 (12)	C6—C7—O8	111.88 (11)
C2—C6—C7	129.92 (12)	C6—C7—O71	124.32 (12)
C5—C6—C7	124.01 (12)	O8—C7—O71	123.79 (12)
C6—C2—C1	132.51 (12)	C7—O8—C9	116.73 (11)
C6—C2—C3	105.77 (11)	O8—C9—C10	107.16 (12)
C1—C2—C3	121.70 (12)	O8—C9—H91	107.5 (10)
C2—C1—O12	121.56 (12)	C10—C9—H91	112.9 (10)
C2—C1—O11	118.18 (12)	O8—C9—H92	106.8 (10)
O12—C1—O11	120.26 (12)	C10—C9—H92	111.5 (10)
C1—O11—H11	113.3 (13)	H91—C9—H92	110.7 (14)
C2—C3—O4	110.72 (12)	C9—C10—H101	112.0 (12)
C2—C3—H31	131.4 (10)	C9—C10—H102	111.4 (11)
O4—C3—H31	117.8 (10)	H101—C10—H102	103.1 (15)
C3—O4—C5	106.99 (10)	C9—C10—H103	111.0 (11)
O4—C5—C6	110.44 (12)	H101—C10—H103	106.1 (15)
O4—C5—H51	117.8 (10)	H102—C10—H103	112.9 (16)
C6—C5—H51	131.7 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···O71	0.94 (2)	1.70 (2)	2.6267 (16)	172 (2)