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Crystal structure of (*E*)-pent-2-enoic acid

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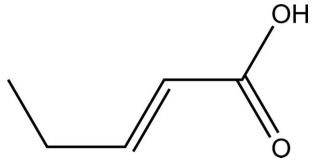
The molecule of the title compound, $C_5H_8O_2$, a low-melting α,β-unsaturated carboxylic acid, is essentially planar [maximum displacement = 0.0239 (13) Å]. In the crystal, molecules are linked into centrosymmetric dimers *via* pairs of O—H···O hydrogen bonds.

Keywords: crystal structure; hydrogen bond; dimer; unsaturated carboxylic acid.

CCDC reference: 1058870

1. Related literature

For the synthesis of unsaturated carboxylic acids including the title compound, see: Shabtai *et al.* (1981); Gastaminza *et al.* (1984); Outurquin & Paulmier (1989). For crystal structure determinations of acrylic acid, see: Higgs & Sass (1963); Chatani *et al.* (1963); Boese *et al.* (1999); Oswald & Urquhart (2011). For the structure of crotonic acid, see: Shimizu *et al.* (1974). For the structure of related hexenoic acid cocrystals, see: Aakeröy *et al.* (2003); Stanton & Bak (2008).



2. Experimental

2.1. Crystal data

$C_5H_8O_2$
M_r = 100.11
Triclinic, $P\overline{1}$
a = 6.7336 (13) Å
b = 6.7821 (13) Å
c = 7.2349 (14) Å
α = 67.743 (2)°
β = 75.518 (2)°
γ = 64.401 (2)°
V = 274.29 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 150 K
0.51 × 0.35 × 0.27 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.81, T_{max} = 0.97
7544 measured reflections
1323 independent reflections
1122 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.113
S = 1.10
1323 reflections
69 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.95 (2)	1.69 (2)	2.6322 (13)	173.3 (19)

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXL2014*; software used to prepare material for publication: *SHELXL2014*.

Acknowledgements

The authors thank P. Thiele (University of Rostock) for DSC measurements and Professor Dr J. G. de Vries (LIKAT) for helpful support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: RZ5155).

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supporting information

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Crystal structure of (*E*)-pent-2-enoic acid

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S1. Synthesis and crystallization

Malonic acid (24.8 g, 237.8 mmol, 1eq) was dissolved in dry pyridine (37.6 g, 475.7 mmol, 2 eq) at room temperature in a three-necked flask equipped with a magnetic stir bar and a reflux condenser under a mild flow of argon. Propanal (13.8 g, 240.2 mmol, 1 eq) was then added in one portion and the resulting clear solution further stirred for 72 h at room temperature under argon. Afterwards, the resulting light yellow to orange solution was brought to an acidic pH value by adding phosphoric acid at 0°C (42.5 wt.-%, 582.7 mmol, 2.45 eq). The resulting two layers were extracted three times with 150 mL portions of ethyl acetate and reduced to a volume of ca. 150 mL. To remove impurities from aldol condensation the raw acid was converted into the corresponding sodium salt by addition of an aqueous solution of sodium carbonate (18.9 g, 178.4 mmol, 0.75 eq in 200 mL). After stirring for 30 minutes the water phase was separated und extracted three times with 150 mL portions of ethyl acetate. The water phase was then acidified with concentrated hydrochloric acid (35.2 g, 356.7 mmol, 1.5 eq), the organic phase was separated and the water phase was again extracted three times with 150 mL portions of ethyl acetate. The combined organic phases were dried over Na_2SO_4 and evaporated to dryness under diminished pressure. The resulting raw product was further purified by distillation *in vacuo* yielding the product in purity >99% (GC). M. p. 10°C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 12.35 (br s, 1H, OH); 7.14 (dt, ^3J = 15.6 Hz, ^3J = 6.3 Hz, 1H, -CH-); 5.82 (dt, ^3J = 15.6 Hz, ^4J = 1.7 Hz, 1H, -CH-); 2.30-2.21 (m, 2H, -CH_2-); 1.08 (t, ^3J = 7.4 Hz, 3H, -CH_3-). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.69 (CO); 153.77 (CH); 119.76 (CH); 25.54 (CH_2); 21.10 (CH_3). MS (EI, 70 eV): *m/z* = 100 (M^+, 50), 83 (13), 82 (23), 81 (10), 58 (11), 57 (17), 56 (23), 55 (100), 54 (43), 53 (35), 52 (12), 51 (25), 50 (28), 45 (77), 41 (36), 40 (13), 39 (99), 38 (25), 37 (11), 29 (61). HRMS (ESI-TOF/MS): calculated for $C_5H_8O_2$ (M^+) 99.04515, found 99.04529. Elemental analysis for $C_5H_8O_2$ % (calc.): C 59.99 (59.98); H 8.05 (8.05). Suitable single crystals were grown by slow evaporation of an ethanolic solution at -30 °C over one week.

S2. Refinement

The carboxylic H atom could be found in a difference Fourier map and was refined freely. All other H atoms were placed in idealized positions with d(C—H) = 0.95 Å (CH), 0.99 Å (CH_2), 0.98 Å (CH_3) and refined using a riding model with U_{iso}(H) fixed at 1.2 U_{eq}(C) for CH and CH_2 and 1.5 U_{eq}(C) for CH_3. A rotating model was used for the methyl group.

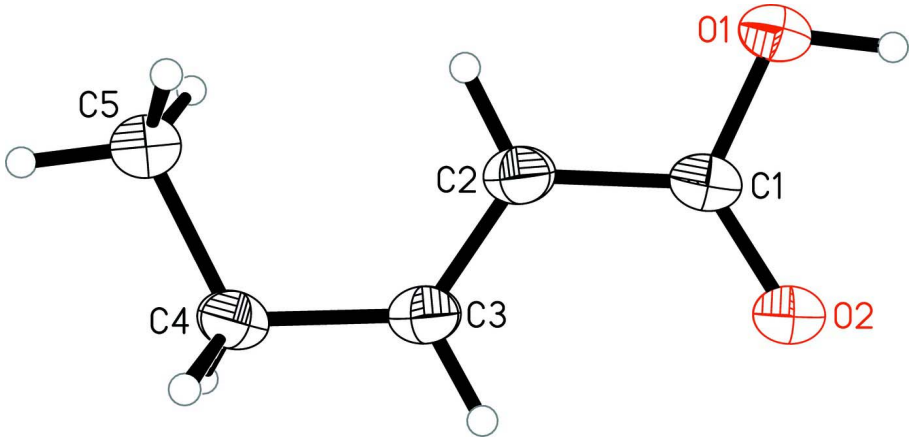

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at 50% probability level.

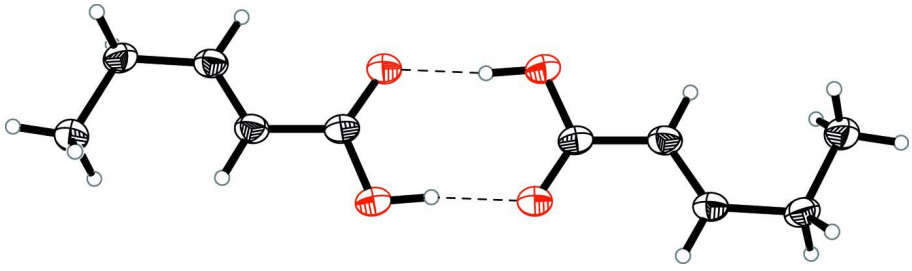

Figure 2

ORTEP representation of a dimer formed by intermolecular O—H···O hydrogen bonds.

(*E*)-Pent-2-enoic acid

Crystal data

$C_5H_8O_2$
M_r = 100.11
Triclinic, $P\overline{1}$
a = 6.7336 (13) Å
b = 6.7821 (13) Å
c = 7.2349 (14) Å
α = 67.743 (2)°
β = 75.518 (2)°
γ = 64.401 (2)°
V = 274.29 (9) Å^3
Z = 2
$F(000)$ = 108
D_x = 1.212 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4399 reflections
θ = 3.1–28.7°
μ = 0.09 mm^{-1}
T = 150 K
Prism, colourless
0.51 × 0.35 × 0.27 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.81, T_{max} = 0.97
7544 measured reflections
1323 independent reflections
1122 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.0°, θ_{min} = 3.1°
h = −8→8
k = −8→8
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.113$
$S = 1.10$
1323 reflections
69 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0602P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.36613 (18)	0.77456 (19)	0.59878 (16)	0.0284 (3)
C2	0.25933 (18)	0.6065 (2)	0.66526 (16)	0.0295 (3)
H2	0.1507	0.6289	0.5893	0.035*
C3	0.31028 (18)	0.4243 (2)	0.82842 (16)	0.0290 (3)
H3	0.4190	0.4066	0.9018	0.035*
C4	0.2111 (2)	0.2446 (2)	0.90640 (17)	0.0323 (3)
H4A	0.1414	0.2401	1.0450	0.039*
H4B	0.3320	0.0930	0.9135	0.039*
C5	0.0389 (2)	0.2791 (2)	0.78294 (19)	0.0372 (3)
H5A	−0.0864	0.4244	0.7812	0.056*
H5B	−0.0133	0.1517	0.8430	0.056*
H5C	0.1055	0.2834	0.6452	0.056*
O1	0.30172 (15)	0.93766 (15)	0.42861 (12)	0.0357 (3)
O2	0.50223 (14)	0.76619 (15)	0.69113 (12)	0.0361 (3)
H1	0.376 (3)	1.039 (4)	0.395 (3)	0.071 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0277 (5)	0.0275 (6)	0.0251 (5)	−0.0060 (4)	−0.0070 (4)	−0.0056 (4)
C2	0.0270 (5)	0.0326 (6)	0.0272 (5)	−0.0093 (5)	−0.0077 (4)	−0.0066 (4)
C3	0.0265 (5)	0.0325 (6)	0.0257 (5)	−0.0086 (4)	−0.0067 (4)	−0.0071 (4)
C4	0.0321 (6)	0.0332 (6)	0.0267 (5)	−0.0121 (5)	−0.0087 (4)	−0.0010 (4)
C5	0.0378 (6)	0.0382 (7)	0.0355 (6)	−0.0177 (5)	−0.0135 (5)	−0.0013 (5)
O1	0.0406 (5)	0.0339 (5)	0.0295 (4)	−0.0147 (4)	−0.0153 (3)	0.0015 (3)
O2	0.0402 (5)	0.0345 (5)	0.0332 (5)	−0.0155 (4)	−0.0166 (4)	−0.0005 (3)

Geometric parameters (Å, º)

C1—O2	1.2337 (14)	C4—C5	1.5239 (16)
C1—O1	1.3223 (13)	C4—H4A	0.9900

C1—C2	1.4723 (16)	C4—H4B	0.9900
C2—C3	1.3301 (16)	C5—H5A	0.9800
C2—H2	0.9500	C5—H5B	0.9800
C3—C4	1.4981 (16)	C5—H5C	0.9800
C3—H3	0.9500	O1—H1	0.95 (2)
O2—C1—O1	122.75 (11)	C5—C4—H4A	108.4
O2—C1—C2	123.99 (10)	C3—C4—H4B	108.4
O1—C1—C2	113.26 (10)	C5—C4—H4B	108.4
C3—C2—C1	122.03 (10)	H4A—C4—H4B	107.5
C3—C2—H2	119.0	C4—C5—H5A	109.5
C1—C2—H2	119.0	C4—C5—H5B	109.5
C2—C3—C4	125.63 (10)	H5A—C5—H5B	109.5
C2—C3—H3	117.2	C4—C5—H5C	109.5
C4—C3—H3	117.2	H5A—C5—H5C	109.5
C3—C4—C5	115.33 (10)	H5B—C5—H5C	109.5
C3—C4—H4A	108.4	C1—O1—H1	108.7 (12)
O2—C1—C2—C3	−3.05 (19)	C1—C2—C3—C4	−179.72 (10)
O1—C1—C2—C3	176.98 (11)	C2—C3—C4—C5	0.45 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.95 (2)	1.69 (2)	2.6322 (13)	173.3 (19)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.