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Article

Synthesis and Molecular Structures of (*E*)-non-2-enoic Acid and (*E*)-dec-2-enoic Acid

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Abstract: The molecular structures of (*E*)-non-2-enoic acid (**C9**) and (*E*)-dec-2-enoic acid (**C10**) are reported. The title compounds were crystallized by slow evaporation of ethanolic solutions at −30 °C. **C9** crystallizes in the monoclinic space group $P2_1/c$ and **C10** in the triclinic space group *P*-1, each with 4 molecules in the unit cell. The unit cell parameters for **C9** are: a = 10.6473(4) Å, b = 5.2855(2) Å, c = 17.0313(7) Å; β = 106.0985(10)° and V = 920.87(6) Å^3. The unit cell parameters for **C10** are: a = 4.1405(2) Å, b = 15.2839(6) Å, c = 17.7089(7) Å; α = 68.3291(11)°, β = 83.3850(13)°, γ = 85.0779(12)° and V = 1033.39(8) Å^3.

Keywords: X-ray crystal structure; hydrogen bond; dimer; unsaturated carboxylic acid; fatty acid

1. Introduction

Unsaturated fatty acids and particularly essential fatty acids are compounds with great interest in food, nutrition and health sciences due to their role in biological processes especially for humans [1–4]. They can be classified as monounsaturated and polyunsaturated, or trans-unsaturated and cis-unsaturated fatty acids, which exhibit long nonpolar unsaturated alkyl chains and polar carboxylic head groups. For example, oleic acid (*Z*)-octadec-9-enoic acid, or linoleic acid (9*Z*,12*Z*)-9,12-octadecadienoic acid, respectively, can be found in natural foods, like meat, milk products, nuts, olives and oils. By increasing

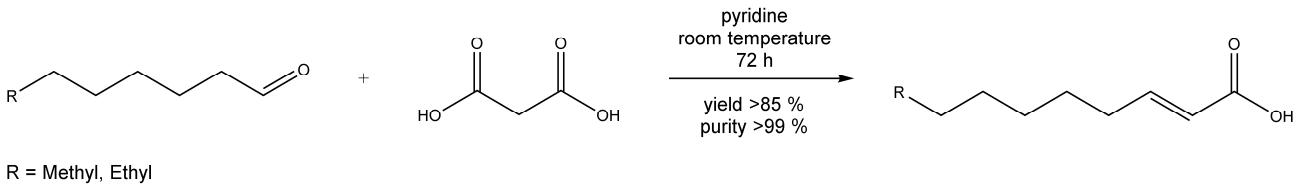
the number of double bonds within the alkyl chain the melting point decreases, yielding liquids, especially in case of polyunsaturated fatty acids at ambient conditions [5]. A certain number of derivatives of the title compounds (*E*)-non-2-enoic acid (**C9**) and (*E*)-dec-2-enoic acid (**C10**) exhibit interesting properties, e.g., 10-Hydroxy-2-decenoic acid isolated from royal jelly shows good antibiotic activity against many bacteria and fungi [6,7]. 4-hydroxy-trans-2-nonenoic acid as a derivate of **C9** acts as a receptor ligand in the cerebral cortex and hippocampus and elevated concentrations can be found in patients with Alzheimer's and Parkinson's disease [8].

Crystal structure determinations of α,β-unsaturated carboxylic acids are still scarce in the literature. The crystal structures of acrylic acid (**C3**) [9–12], and crotonic acid (**C4**) [13] have been known for more than 40 years, while recently the crystal structures of four more members of α,β-unsaturated carboxylic acids, (*E*)-pent-2-enoic acid (**C5**) [14], (*E*)-hex-2-enoic acid (**C6**) [15], (*E*)-undec-2-enoic acid (**C11**) [16], and (*E*)-dodec-2-enoic acid (**C12**) [17], respectively, have been reported. In addition, two crystal structure determinations of co-crystals containing **C6** are also known [18,19]. In comparison a complete set of crystal structures of the corresponding saturated carboxylic acids (propionic acid to pentadecanoic acid) is available from the literature [20–24]. In this contribution, we report on the synthesis, characterization and crystal structure determinations of two members of the series of α,β-unsaturated carboxylic acids: (*E*)-non-2-enoic acid (**C9**) and (*E*)-dec-2-enoic acid (**C10**), respectively, which are linked into centrosymmetric dimers *via* pairs of O–H···O hydrogen bonds in the crystals.

2. Results and Discussion

2.1. Synthesis

One-hundred and thirty years ago, Schneegans reported on the synthesis of nonenoic acid via reaction of heptanal, sodium acetate and acetic anhydride without knowledge of the correct constitution of this α,β-unsaturated carboxylic acid [25]. Later, Harding and Weizmann reported on the correct molecular formula of the received (*E*)-non-2-enoic acid [26]. However, until now there has been no X-ray determination of the crystal structure of **C9** or its homologue **C10**. The syntheses of the two unsaturated α,β-carboxylic acids **C9** and **C10** are conducted by a convenient adapted condensation reaction of malonic acid and the appropriate aldehydes at room temperature in high yields and purities as depicted in Scheme 1 [27–30].



Scheme 1. Reaction sequences for the synthesis of the two title compounds **C9** and **C10**.

2.2. Crystal Structures

Molecular structures of linked acid dimers of the title compounds with pairs of O–H···O hydrogen bonds are shown in Figures 1 and 2, respectively, with atom-labeling scheme and displacement thermal

ellipsoids at the 50% probability level. Crystal and instrumental parameters of the crystal structure determinations of **C9** and **C10** can be found in Table 1.

Selected bond lengths and angles are summarized in Table 2. **C9** crystallizes in monoclinic space group $P2_1/c$ with 4 molecules in the unit cell, whereas **C10** crystallizes in the triclinic space group P-1 with two symmetry independent molecules in the asymmetric unit and $Z = 4$.

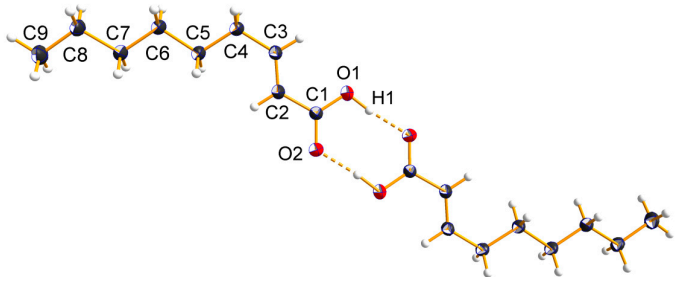


Figure 1. View of acid dimer molecules in the crystal of **C9** showing atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

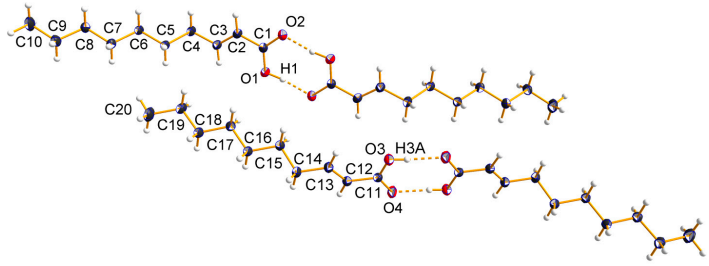


Figure 2. View of acid dimers of the two symmetry independent molecules in the crystal of **C10** showing atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Table 1. Crystal data and structure refinement for compounds **C9** and **C10**.

Compound		**C9**	**C10**
Chemical formula		$C_9H_{16}O_2$	$C_{10}H_{18}O_2$
Formula weight		156.22	170.24
Crystal system		monoclinic	triclinic
Unit cell dimensions	*a*	10.6473(4) Å	4.1405(2) Å
	b	5.2855(2) Å	15.2839(6) Å
	c	17.0313(7) Å	17.7089(7) Å
	α	90°	68.3291(11)°
	β	106.0985(10)°	83.3850(13)°
	γ	90°	85.0779(12)°
Unit cell volume		920.87(6) Å^3	1033.39(8) Å^3
Temperature		150(2) K	150(2) K
Space group		$P2_1/c$	$P\bar{1}$
Z (*Z'*)		4 (1)	4 (2)
μ		0.078 mm^{-1}	0.074 mm^{-1}
No. of reflections measured		8209	20,844
No. of independent reflections		2216	4999

Table 1. *Cont.*

Compound	C9	C10
R_{int}	0.0148	0.0236
Final R_1 values ($I > 2\sigma(I)$)	0.0365	0.0443
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.0992	0.1108
Final R_1 values (all data)	0.0420	0.0619
Final $wR(F^2)$ values (all data)	0.1046	0.1226
Goodness of fit on F^2	1.054	1.057
Density	1.127 g/cm³	1.094 g/cm³

Table 2. Selected atom distances (Å) and angles (°) for compounds **C9** and **C10**.

--	C9	C10	--	--
Atoms	**Distance**	**Distance**	**Atoms**	**Distance**
C1–O1	1.2902(11)	1.2860(15)	C11–O3	1.2757(15)
C1–O2	1.2468(11)	1.2547(15)	C11–O4	1.2575(14)
C1–C2	1.4711(12)	1.4714(17)	C11–C12	1.4697(16)
C2–C3	1.3242(13)	1.3199(18)	C12–C13	1.3197(17)
C3–C4	1.5010(11)	1.4965(16)	C13–C14	1.4927(16)
average C–C (*alkyl chain*)	1.523	1.523	average C–C (*alkyl chain*)	1.523
H-bonds in:	***D*–H···*A***	***D*–H**	**H···*A***	***D*···*A***
C9	O1–H1···O2^{i}	0.960(15)	1.676(15)	2.6319(9)
C10	O1–H1···O2^{ii}	0.967(19)	1.679(19)	2.6400(13)
--	O3–H3A···O4^{iii}	0.84	1.79	2.6209(12)
--	O4–H4A···O3^{iii}	0.84	1.81	2.6209(12)
Atoms	**Angle**	**Angle**	**Atoms**	**Angle**
O1–C1–O2	123.35(8)	123.32(11)	O3–C11–O4	123.55(11)
O1–C1–C2	117.78(8)	117.88(11)	O3–C11–C12	118.30(10)
O2–C1–C2	118.88(8)	118.80(11)	O4–C11–C12	118.14(11)
C1–C2–C3	123.29(8)	123.74(12)	C11–C12–C13	124.19(12)
C2–C3–C4	125.11(8)	124.69(12)	C12–C13–C14	124.11(12)
H-bonds in:	***D*–H···*A***	**Angle**	**Symmetry codes**	--
C9	O1–H1···O2^{i}	173.9(14)	(i) $-x + 2, -y, -z + 2$	--
C10	O1–H1···O2^{ii}	172.4(17)	(ii) $-x + 1, -y, -z + 2$	--
--	O3–H3A···O4^{iii}	170	(iii) $-x + 2, -y + 1, -z$	--
--	O4–H4A···O3^{iii}	162	--	--

Interestingly, all corresponding saturated carboxylic acids crystallize exclusively in monoclinic space groups at low temperatures, either in *C*2/*m* (No. 12) [22], *P*2$_1$/*c* (No. 14) [20,23,24], or *C*2/*c* (No. 15) [24], respectively, whereas only the high-pressure form of propionic acid crystallizes in the triclinic space group *P*-1 (No. 2) [21]. In contrast to this findings, the analogous α,β-unsaturated carboxylic acids crystallize either in orthorhombic (**C3**: *Ibam*) [9–12], monoclinic (**C4**: *C*2/*c*) [13], or triclinic (**C5**, **C6**, **C11**, **C12**: *P*-1) [14–17] space groups. The high-pressure form of acrylic acid crystallizes in the monoclinic space group *P*2$_1$/*c* (No. 14) [12]. Bond lengths and angles in **C9** and **C10** resemble closely values found in other α,β-unsaturated carboxylic acids [9–19]. The crystal structures of **C9** and **C10** are characterized by carboxylic acid inversion dimers linked by pairs of O–H···O

hydrogen bonds. Views of the packing of acid dimer layers in **C9** and **C10** are depicted in Figures 3 and 4, respectively. All non-hydrogen atoms of **C9** lie almost in one plane with a torsion angle between the carboxylic group and the alkyl chain of 8.1° (C2–C3–C4–C5). In contrast, **C10** features two symmetry-independent molecules, which adopt a conformation where the carboxylic group and the following three carbon atoms of the chain of the molecules lie almost in one plane and the remaining carbon atoms of the hydrocarbon chain adopt a nearly fully staggered conformation with torsion angles C2–C3–C4–C5: 127.7° and C12–C13–C14–C15: 122.8°, respectively.

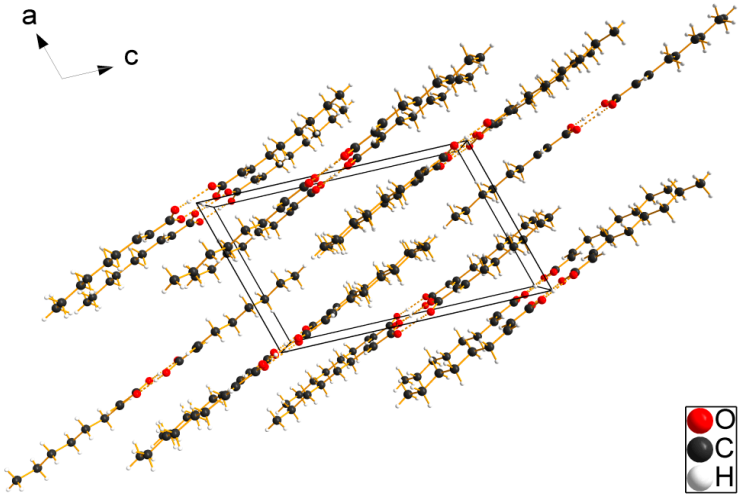


Figure 3. View of the packing of acid dimer layers in the crystal of **C9** along the crystallographic *b* axis.

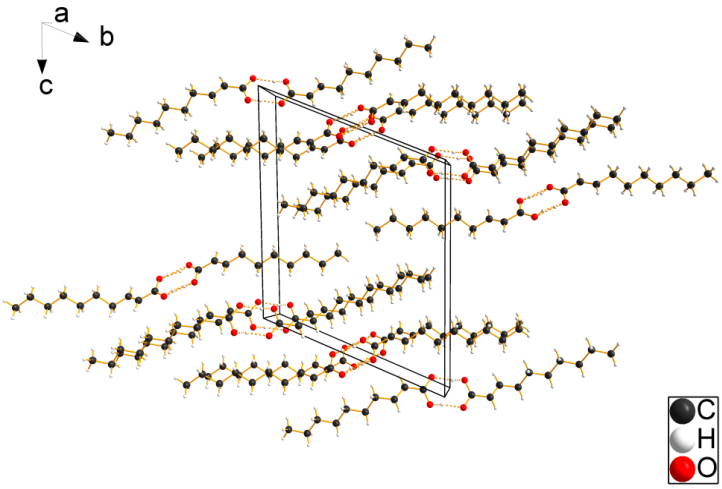


Figure 4. View of the packing of acid dimer molecules in the crystal of **C10** along the crystallographic *a* axis.

3. Experimental Section

3.1. General Considerations

NMR spectra were recorded on a Bruker AV300 or Bruker AV400 and chemical shifts of the ^{1}H, and ^{13}C spectra were reported in parts per million (ppm) using the solvent shifts for ^{1}H and ^{13}C as internal

standard ($CDCl_3$: ^1H δ = 7.26, ^{13}C δ = 77.0). Elemental analysis for C, and H was performed on a Leco Microanalysator TruSpec CHNS device. MS spectra were determined by electron spray ionization using a Thermo Electron Finnigan MAT 95-XP mass spectrometer. Melting points were determined by cyclic differential scanning calorimetry (DSC) using a Mettler Toledo DSC823^e in the range of −50 to 50 °C with a heating rate of 10 K/min (2 cycles, N_2 atmosphere, Al crucible). All melting points are peak temperatures.

3.2. Materials

All chemicals were used as received without further drying or purification unless otherwise noted. *n*-heptanal, *n*-octanal, and malonic acid were purchased from ABCR (purity > 98%).

3.3. General Synthesis of α,β*-unsaturated Carboxylic acids*

Malonic acid (25.0 g, 240.2 mmol, 1.0 eq) was dissolved in dry pyridine (38.0 g, 480.5 mmol, 2.0 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. The appropriate aldehyde (240.2 mmol, 1.0 eq) was then added in one portion and the resulting clear solution was 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. %, 138.5 g, 600.6 mmol, 2.5 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 *in vacuo*. 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 (20.4 g, 192.2 mmol, 0.8 eq in 200 mL). After stirring for 30 min the water phase was separated and extracted three times with 150 mL portions of ethyl acetate. The water phase was then acidified with concentrated hydrochloric acid (37.0 wt. %, 35.5 g, 360.4 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 *in vacuo*. The resulting raw product was further purified by distillation *in vacuo* yielding the product in purity > 99% (GC).

(E)-non-2-enoic acid (**C9**). M. p. 7 °C (lit. 1 °C) [31]; ^1H NMR (400 MHz, $CDCl_3$): δ = 12.05 (br s, 1H, OH); 7.09 (dt, ^3J = 15.6 Hz, ^3J = 7.0 Hz, 1H, –CH–); 5.82 (dt, ^3J = 15.6 Hz, ^4J = 1.6 Hz, 1H, –CH–); 2.26–2.19 (m, 2H, –CH_2–); 1.50–1.432(m, 2H, –CH_2–); 1.35–1.25 (m, 6H, 3x –CH_2–); 0.91–0.85 (m, 3H, –CH_3–). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.56 (CO); 152.57 (CH); 120.78 (CH); 32.46 (CH_2); 31.71 (CH_2); 28.95 (CH_2); 27.98 (CH_2); 22.67 (CH_2); 14.17 (CH_3). MS (EI, 70 eV): *m/z* = 156 (M^+, 0), 99 (17), 97 (11), 96 (25), 95 (10), 86 (26), 84 (23), 81 (24), 73 (49), 71 (11), 70 (12), 69 (23), 68 (27), 67 (19), 57 (14), 56 (28), 55 (53), 54 (16), 53 (31), 45 (37), 43 (83), 42 (33), 41 (100), 40 (20), 39 (89), 38 (11), 29 (75). HRMS (ESI-TOF/MS): calculated for $C_9H_{16}O_2$ (M^+) 155.10759, found 155.10759. Elemental analysis for $C_9H_{16}O_2$% (calc.): C 69.27 (69.19); H 10.28 (10.32).

(E)-dec-2-enoic acid (**C10**): M. p. 12 °C (lit. 9–12 °C) [32,33]; ^1H NMR (400 MHz, $CDCl_3$): δ = 12.13 (br s, 1H, OH); 7.09 (dt, ^3J = 15.6 Hz, ^3J = 7.0 Hz, 1H, –CH–); 5.82 (dt, ^3J = 15.6 Hz, ^4J = 1.6 Hz, 1H, –CH–); 2.26–2.19 (m, 2H, –CH_2–); 1.50–1.43 (m, 2H, –CH_2–); 1.34–1.25 (m, 8H, 4x–CH_2–); 0.90–0.85 (m, 3H, –CH_3–). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.56 (CO); 152.67 (CH); 120.78 (CH); 32.46 (CH_2); 31.87 (CH_2); 29.22 (CH_2); 29.18 (CH_2); 28.02 (CH_2); 22.76 (CH_2); 14.20 (CH_3).

MS (EI, 70 eV): *m/z* = 170 (M^+, 0), 110 (14), 99 (15), 98 (12), 86 (19), 84 (17), 82 (10), 81 (21), 73 (43), 70 (16), 69 (25), 68 (26), 67 (19), 57 (20), 56 (28), 55 (48), 54 (14), 53 (27), 45 (29), 43 (78), 42 (23), 41 (100), 40 (16), 39 (74), 29 (75). HRMS (ESI-TOF/MS): calculated for $C_{10}H_{18}O_2$ (M^+) 169.123340, found 169.12335. Elemental analysis for $C_{10}H_{18}O_2$% (calc.): C 70.62 (70.55); H 10.57 (10.66).

3.4. Crystal Structure Determinations

Data were collected on a Bruker Kappa APEX II Duo diffractometer [34]. The structures were solved by direct methods and refined by full-matrix least-squares procedures on F^2 with the SHELXTL software package [35]. Data were corrected for absorption effects using the multi-scan method (SADABS) [36]. All non-hydrogen atoms were refined anisotropically. H1 could be found from the difference Fourier map and was refined with U_{iso}(H) fixed at 1.5 U_{eq}(O) for both compounds. For compound **C10** the carboxyl group of the second molecule is disordered, therefore H3A and H4A were placed using AFIX 83 instruction with site occupancy factors of 0.6 for H3A and 0.4 for H4A (d(O–H) = 0.84 Å, U_{iso}(H) fixed at 1.5 U_{eq}(O)). The disorder concerning O3 and O4 was not resolved. 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. Crystal data, data collection, and refinement parameters are collected in Table 1. DIAMOND program package was used for graphical representations [37]. Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC-1408770 for *(E)*-non-2-enoic acid (**C9**), and CCDC-1408769 for *(E)*-dec-2-enoic acid (**C10**). These data can be obtained free of charge via http://www.ccdc.cam.ac.uk or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; Fax: (+44)-1223-336-033; or E-Mail: deposit@ccdc.cam.ac.uk.

4. Conclusions

The crystal and molecular structures of two homologues in the series of α,β-unsaturated carboxylic acids, namely (*E*)-non-2-enoic acid (**C9**) and (*E*)-dec-2-enoic acid (**C10**), are reported. In analogy to other known crystal structures of this class of substances **C9** and **C10** are characterized by carboxylic acid inversion dimers linked by pairs of O–H···O hydrogen bonds. However, in contrast to previously reported structures, **C10** features two symmetry-independent molecules.

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Author Contributions

All authors contributed equally to this contribution.

Conflicts of Interest

The authors declare no conflict of interest.

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