Azimuthal scans of several reflections indicated no need for an absorption correction. The H atoms were located from a difference Fourier map and refined isotropically. The structure was solved by direct methods (*MITHRIL*; Gilmore, 1984) utilizing the *TEXSAN* (Molecular Structure Corporation, 1985) system.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55926 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1004]

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Structure of the γ Phase of Erucic Acid

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Abstract

The hydrocarbon chains on either side of the *cis*-olefin group in 13-docosenoic acid adopt all-*trans* conformation and form an orthorhombic subcell with parallel skeletal planes. Both the methyl- and carboxyl-terminal polymethylene chains are inclined toward the interface of the dimer layers by 34°. The torsion angles of the *cis*-olefin group, C—C—C=C—C—C, are −129, 0 and 127° from the carboxyl side.

Comment

The structure determination of the γ phase of erucic acid was undertaken to confirm that this phase is isomorphous to the low-melting phase of oleic acid (Abrahamsson & Ryderstedt-Nahringbauer, 1962), and to elucidate the dynamic features of the acyl chain. We have shown that *cis*-mono-unsaturated fatty acids crystallize in various phases, among which some reversible or irreversible solid-state phase transitions take place. In the case of erucic acid, four polymorphic phases (α, $\alpha 1$, γ and $\gamma 1$) and two types of reversible phase transition ($\gamma \Leftrightarrow \alpha$ and $\gamma 1 \Leftrightarrow \alpha 1$) have been found. We found that the γ phase is quite similar to the low-melting phase of oleic acid in its vibrational spectrum, crystal habit and thermal behaviour (Suzuki, Sato, Yoshimoto,

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Tanaka & Kobayashi, 1988; Kobayashi & Kaneko, 1990).

The obtained structure of the γ phase is essentially the same as the low-melting phase of oleic acid, as we expected. The hydrocarbon chains on either side of the *cis*-olefin group adopt all-*trans* conformation and form an orthorhombic subcell with a parallel arrangement of the skeletal planes (O′‖), found in oleic and linoleic acids (Ernst, Sheldrick & Fuhrhop, 1979). The average subcell parameters of γ are $a_s = 7.85$, $b_s = 4.67$ and $c_s = 2.55$ Å for the methyl-terminal chain and $a_s = 7.90$, $b_s = 4.67$ and $c_s = 2.55$ Å for the carboxyl-terminal chain. The b_s axis is parallel to the unique b axis of the main lattice, and the c_s axis tilts toward the a axis by 34° for both methyl and carboxyl sides; in other words, the (201) plane of the subcell is parallel to the interface of the dimer layers. The C(11)—C(12)—C(13)=C(14)—C(15)—C(16) torsion angles are −129.1 (2), −0.1 (4) and 127.5 (2)°, being recognized as skew, *cis*, skew′ type.

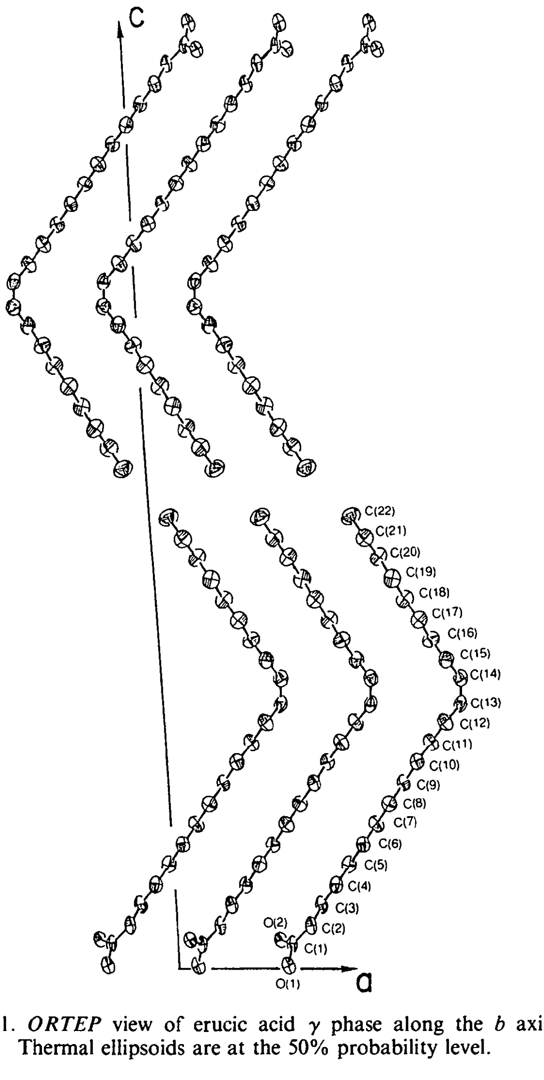

Fig. 1. *ORTEP* view of erucic acid γ phase along the b axis. Thermal ellipsoids are at the 50% probability level.

Both the γ and γ1 phases show a reversible solid-state phase transition accompanied with conformational disordering in the methyl-terminal chain on heating (Kobayashi & Kaneko, 1990). However, thermal factors of the acyl chains suggest that the methyl-terminal chain is less mobile in the γ phase than in the γ1 phase. A steep increase of the thermal factors of C atoms toward the terminal methyl occurs at the C(13)=C(14) bond in the γ1 phase (Kaneko, Kobayashi, Kitagawa, Matsuura, Sato & Suzuki, 1992), while such a change is not observed in the γ phase. The thermal factors increase gradually toward the terminal methyl similarly to those of *n*-fatty acid *E*-form (Kaneko, Kobayashi, Kitagawa & Matsuura, 1990) in which no conformational disorder takes place. This is consistent with our recent result obtained by spectroscopic study, where we indicated a difference in transition behaviour between the γ–α and γ1–α1 transitions; the γ phase holds a regular conformation until the transition point, whereas the conformational disordering starts at a temperature about 30° below the transition point in the γ1 phase (Kaneko, Yamazaki, Kobayashi, Sato & Suzuki, 1991).

The carboxyl-group plane (O—C=O) is twisted by about 20° with respect to the skeletal plane of the carboxyl-terminal hydrocarbon chain. The molecules are dimerized *via* hydrogen bonds of the carboxyl groups [O···O 2.659 (2) Å]. The carbonyl group C(1)=O(2) and the C(2)—C(3) bond adopt *cis* geometry about C(1)—C(2).

Experimental

Crystal data

$C_{22}H_{42}O_2$
$M_r = 338.57$
Monoclinic
$P2_1/a$
$a = 9.492$ (2) Å
$b = 4.674$ (6) Å
$c = 49.39$ (1) Å
$\beta = 93.65$ (2)°
$V = 2186.8$ (8) Å^3
$Z = 4$
$D_x = 1.03$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 19$-31°
$\mu = 0.45$ mm^{-1}
$T = 221$ K
Plate
0.60 × 0.40 × 0.10 mm
Colorless
Crystal source: grown from an acetonitrile solution

Data collection

4-Circle diffractometer
ω scans
Absorption correction: none
12964 measured reflections
3346 independent reflections
2789 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int} = 0.045$
$\theta_{\rm max} = 120$°
$h = -10 \rightarrow 10$
$k = -5 \rightarrow 5$
$l = -55 \rightarrow 55$
3 standard reflections
frequency: 80 min
intensity variation: 3%

Refinement

Refinement on F
Final $R = 0.051$
$wR = 0.067$
$S = 1.52$
2789 reflections
385 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F)+0.008F^2]$
$(\Delta/\sigma)_{\max} = 0.026$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
O(1)	0.1191 (2)	0.7973 (3)	0.00320 (3)	3.65
O(2)	0.0826 (2)	0.4245 (3)	0.03029 (3)	3.64
C(1)	0.1444 (2)	0.6478 (4)	0.02559 (4)	3.13
C(2)	0.2510 (3)	0.7824 (5)	0.04530 (4)	3.70
C(3)	0.3193 (2)	0.5856 (4)	0.06672 (4)	3.54
C(4)	0.4078 (2)	0.7535 (4)	0.08820 (4)	3.47
C(5)	0.4865 (3)	0.5683 (5)	0.10940 (4)	3.69
C(6)	0.5707 (2)	0.7428 (5)	0.13103 (4)	3.61
C(7)	0.6521 (3)	0.5628 (5)	0.15237 (4)	3.77
C(8)	0.7356 (2)	0.7399 (5)	0.17382 (4)	3.66
C(9)	0.8175 (3)	0.5618 (5)	0.19522 (4)	3.88
C(10)	0.9012 (3)	0.7391 (5)	0.21665 (4)	3.76
C(11)	0.9837 (3)	0.5591 (5)	0.23792 (4)	4.03
C(12)	1.0693 (3)	0.7350 (5)	0.25909 (4)	4.09
C(13)	1.1618 (3)	0.5516 (5)	0.27798 (4)	4.06
C(14)	1.1719 (3)	0.5543 (5)	0.30481 (4)	4.23
C(15)	1.0942 (3)	0.7413 (5)	0.32366 (4)	4.44
C(16)	1.0213 (3)	0.5700 (5)	0.34495 (5)	4.60
C(17)	0.9557 (3)	0.7565 (5)	0.36622 (4)	4.39
C(18)	0.8856 (3)	0.5860 (5)	0.38793 (5)	4.74
C(19)	0.8198 (3)	0.7724 (5)	0.40912 (5)	4.65
C(20)	0.7521 (3)	0.6045 (6)	0.43126 (5)	5.05
C(21)	0.6847 (3)	0.7931 (6)	0.45203 (5)	5.53
C(22)	0.6150 (4)	0.6246 (9)	0.47396 (7)	7.1

Table 2. *Selected geometric parameters* (Å, °)

C(1)—O(1)	1.317 (2)	C(12)—C(13)	1.507 (3)
C(1)—C(2)	1.497 (3)	C(13)—C(14)	1.322 (3)
C(1)—O(2)	1.227 (2)	C(14)—C(15)	1.504 (3)
C(2)—C(1)—O(1)	123.1 (2)	C(12)—C(13)—C(14)	128.1 (2)
O(1)—C(1)—O(2)	123.1 (2)	C(13)—C(14)—C(15)	128.2 (2)
C(10)—C(11)—C(12)	113.8 (2)	C(14)—C(15)—C(16)	112.6 (2)
C(2)—C(1)—O(2)	113.8 (2)	C(15)—C(16)—C(17)	113.5 (2)
C(11)—C(12)—C(13)	112.5 (2)		

O(1)—C(1)—C(2)—C(3)	−161.1 (2)
O(2)—C(1)—C(2)—C(3)	21.5 (3)
C(1)—C(2)—C(3)—C(4)	−171.1 (2)
C(10)—C(11)—C(12)—C(13)	−173.8 (2)
C(11)—C(12)—C(13)—C(14)	−129.1 (2)
C(12)—C(13)—C(14)—C(15)	−0.1 (4)
C(13)—C(14)—C(15)—C(16)	127.5 (2)
C(14)—C(15)—C(16)—C(17)	173.6 (2)

Data collection: Rigaku AFC-5 with software *AFCM* from the Protein Research Center, Osaka University. Programs used to refine structure: *HBLS*-V and *FMLS* (Ashida, 1979). Programs used to plot structure: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *POTP* (Yasuoka, Kimura & Mizuma, 1979) and *DAPH* (Ashida, 1979). The following two types of ω-scan width were used in order to avoid the overlapping of neighbouring reflections, with an ω-scan rate of 4.0° min^{-1} and background counts for 4 s on each side of every scan: $(1.6-0.17\tan\theta)°$ for reflections with $\theta \leq 100°$ and $(1.6-0.4\tan\theta)°$ for reflections with $100 < \theta < 120°$. The molecular model was initially built for 13 C and two O atoms by reference to the structure of the γ phase of oleic acid. Refinement was by full-matrix least-squares methods.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55950 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1011]

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Structure of a Coupled Carbohydrate and Terpene†

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Abstract

In (1S-{1α,1[3R*,5R*(R*)],2α,5β})-5-[1,2-bis(benzoyloxy)ethyl]-4,5-dihydro-3-[1-hydroxy-5-methyl-2-(1-methylethyl)cyclohexyl]-2(3H)-furanone, the tetrahydrofuran (THF) ring exhibits a shallow conformation with atom C3 (C-4 by IUPAC numbering)

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† Part 2. Part 1: Abboud, Jiang & Enholm (1993).

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