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Refinement of the Structure of β-Tricaprin*

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The β-phase of tricaprin ($C_{33}O_6H_{62}$) is triclinic, space group $P\bar{1}$, with the following unit-cell dimensions:

$$\begin{array}{ll} a = 5\cdot488 \pm 0\cdot01 \text{ Å} & \alpha = 85\cdot35 \pm 0\cdot15^\circ \\ b = 12\cdot176 \pm 0\cdot03 & \beta = 87\cdot27 \pm 0\cdot15 \\ c = 26\cdot93 \;\; \pm 0\cdot07 & \gamma = 79\cdot28 \pm 0\cdot15 \end{array}$$

Three-dimensional data collected at ambient temperature, which is only a few degrees below the melting point of 31·9°C, have been used to refine the structure to a final $R = 5\cdot1$ %. The molecules are packed as a modified 'tuning fork' in which two chains with two glyceryl carbon atoms and two ester oxygen atoms form a nearly straight chain, while the third chain branches off by way of the third glyceryl carbon atom and an ester oxygen atom, bends and packs into the structure essentially parallel to the other chains. The chains are packed in the $T\|$ subcell and appear to be slightly bent. The thermal motions of most atoms are quite anisotropic and the motions of those toward the terminal ends of the chains are considerably greater than that of those near the glyceryl moiety. All 62 hydrogen atoms were located by means of a ΔF synthesis. Standard deviations of the 33 C atoms range from 0·0036 to 0·0063 Å, for the 6 O atoms from 0·0023 to 0·0039 Å, and for the 62 H atoms from 0·029 to 0·057 Å.

Tricaprin is the trisubstituted glyceride of capric acid. It is thus a member of the series of simple symmetrical triglycerides which belong to the large class of compounds, the lipids. These are widely distributed in biological systems and have both structural and functional significance. In spite of the interest in the role of the triglycerides in such systems, no complete single-crystal structural analyses have been reported until recently. Vand & Bell (1951) have reported the structure of the chains in β-trilaurin, and a preliminary account of the work on β-tricaprin has appeared (Jensen & Mabis, 1963). Larsson (1964*a*, *b*) has recently reported the structure of β-trilaurin as well as the structures of some mono- and diglycerides.

The structure of β-tricaprin became somewhat of a challenge because of the difficulties involved in collecting sufficient data of adequate quality. The reflections are dominated by relatively few which are intense with most of the rest weak or absent. Furthermore, data were collected within a few degrees of the melting point and the structure is large, 101 atoms in the molecule. Nevertheless, precision is good, but the parameters suffer from large systematic errors, presumably due to thermal motion.

Experimental

High purity tricaprin (m.p. 31·9°) was supplied by Dr E. S. Lutton, and crystals of the β form were grown by slow cooling from the melt as poorly shaped laths elongated along *a*. The only prominently developep faces are {001}.

Linear unit-cell parameters were determined from oscillation and Weissenberg photographs with Cu $K\alpha$ radiation ($\lambda = 1\cdot5418$ Å) and NaCl as an internal standard. Angular parameters were determined from precession and Weissenberg photographs taken with Mo $K\alpha$ and Cu $K\alpha$ radiation respectively. The parameters of the triclinic unit cell chosen according to Dirichlet (Balashov & Ursell, 1957) are as follows:

$$\begin{array}{ll} a = 5\cdot488 \pm 0\cdot01 \text{ Å} & \alpha = 85\cdot35 \pm 0\cdot15^\circ \\ b = 12\cdot176 \pm 0\cdot03 & \beta = 87\cdot27 \pm 0\cdot15 \\ c = 26\cdot93 \;\; \pm 0\cdot07 & \gamma = 79\cdot28 \pm 0\cdot15 \end{array}$$

where the precision indices are estimated standard deviations.

Statistical treatment of the data by Ramachandran & Srinivasan's (1959) modification of the method of Howells, Phillips & Rogers indicated a center of symmetry. The space group was assumed, therefore, to be $P\bar{1}$.

Although absorption of Cu $K\alpha$ radiation by β-tricaprin is not large ($\mu = 4\cdot16$ cm^{-1}), nevertheless, for the best precision, the lath-shaped crystals must be cut to a more nearly ideal shape. Since the crystals are soft and deform readily, it was not possible to cut or grind in the usual way. Accordingly, a solvent lathe was constructed (Peterson, Steinrauff & Jensen, 1960) and served well to cut a crystal without distortion (Fig. 1).

A relatively large, slightly wedge-shaped crystal of approximate dimensions 1·1 × 0·25 × 0·12 mm was cho-

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sen and cut initially into four pieces, two of dimensions 0·35 × 0·25 × 0·12 mm for mounting with a as rotation axis and two of dimensions 0·15 × 0·25 × 0·12 mm for mounting with b as rotation axis. These crystals were shaped further by a combination of planing with a razor blade controlled by means of a micromanipulator and dressing with the cutting filament of the solvent lathe. The specimen finally chosen for collecting data was essentially a cylinder with an elliptic cross section of dimensions approximately 0·15 × 0·12 mm and axis coincident with a.

It is worth noting that no particular difficulty was experienced in using the solvent lathe with a single nylon filament 30 μ in diameter as the cutting tool and ethanol as solvent. Indeed, for cutting easily deformed crystals such as β-tricaprin, this device has been found superior to any other tried.

Unidimensionally integrated equi-inclination Weissenberg data (Jensen, 1954) for $0kl$–$5kl$ were collected by the multiple film technique. Because the lattice is triclinic, all reciprocal lattice points within a hemisphere are unique. To record a complete set of unique data, it is, therefore, necessary either to take two sets of photographs for each upper level or to record by the anti-equi-inclination as well as equi-inclination technique. The first alternative was followed in this work. Two sets of Weissenberg photographs were taken for each level with two 40° regions of overlap between sets. Thus every reflection on the upper levels was recorded at least twice.

Intensities of most reflections were measured by a recording microdensitometer scanning at right angles to the direction of integration on the camera. The response of the recorder was logarithmic so that, within the linear range of the film, the area above background of each trace is proportional to the integrated intensity. Some of the weakest reflections were estimated by eye, which is better than the densitometer near the limit of detection because integration is over the entire spot. For reflections of greater intensity, however, the densitometer is much superior; and for a good set of photographs, it can be expected to about halve the error in the data.

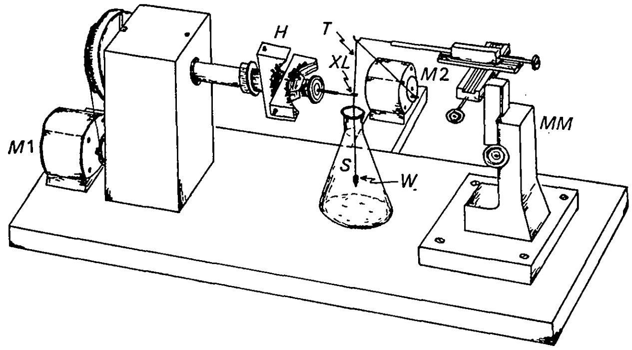


Fig. 1. Solvent lathe. H, goniometer head; $M1$ and $M2$, 1 r.p.s. motors; MM, micromanipulator; S, solvent; W, weight; XL, crystal; T, thread.

A determined effort was made to maximize the number of reflections. Nevertheless, even with 100 hour exposures (Kodak No-Screen film, tube operated at 20 mA, 40 p.k.v.) it was possible to record with measurable intensity only 2220 of the approximately 7500 independent reflections which were in position to reflect. The relatively small percentage observed, 29·6%, stems mainly from two factors:

(1) Room temperatures were in the range 21–27°C when the data were collected and the crystal temperature ran several degrees above this. Thus the crystal is within a few degrees of its melting point and for structures such as this the mean thermal motion of the atoms is large.

(2) β-Tricaprin has a pronounced subcell with a few groups of intense reflections separated by large regions of weak or unobserved reflections. To compensate in part for the relatively few observable reflections, all those recorded in the appropriate intensity range were measured on each of the multiple films.

One of the shaped crystals mounted for rotation about b was used to collect several hundred $h0l$, $h1l$ and $h2l$ data. These were used solely to determine a set of inter-level scale factors in order to bring the data to a common scale.

Though stringent precautions were taken, the data for this compound were subject to more than the usual scatter as judged from the constancy of the inter-film factors and inter-set scale factors for each level. This is probably due in part at least to experimental difficulties encountered in collecting data so close to the melting point.

Solution and initial refinement of the structure

The data initially available for the solution of the structure (Fe $K\alpha$ radiation) were eye estimated and of both limited extent and precision (Jensen & Mabis, 1963). It was decided to attempt the solution in projection along [100] rather than in three dimensions not only because of the more modest computing requirements (the initial computations were done on an IBM 650 computer) but also because fewer parameters are involved. From the dimensions of the a axis, it is evident that virtually all nonhydrogen atoms must be resolved.

The positions of the hydrocarbon chains in β-trilaurin were already available (Vand & Bell, 1951) but the published evidence for the correctness of their positions did not seem conclusive. Accordingly, a Patterson projection along [100] was computed, and it showed clearly the direction of the hydrocarbon chains. Together with packing considerations, this information led directly to a 'tuning fork' configuration for the molecule and chain positions essentially as given by Vand. No attempt was made to deduce an exact model. In fact, C atoms were distributed uniformly along a molecular skeleton with the chain atoms lying along straight lines. Calculation of a set of structure factors resulted in an $R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$ of 79%. An F_o

synthesis showed relatively large corrections in the y coordinate for atoms in one of the chains with only small corrections for the remaining atoms. Recalculation of the structure factors dropped R only to a disappointing 76% although there was no indication that the structure was grossly wrong.

At this point the two-dimensional data were checked and a number of errors corrected and additional reflections at high sin θ estimated to increase the resolution. Recalculation of the structure factors with the corrected and extended data resulted in an R of 60%.

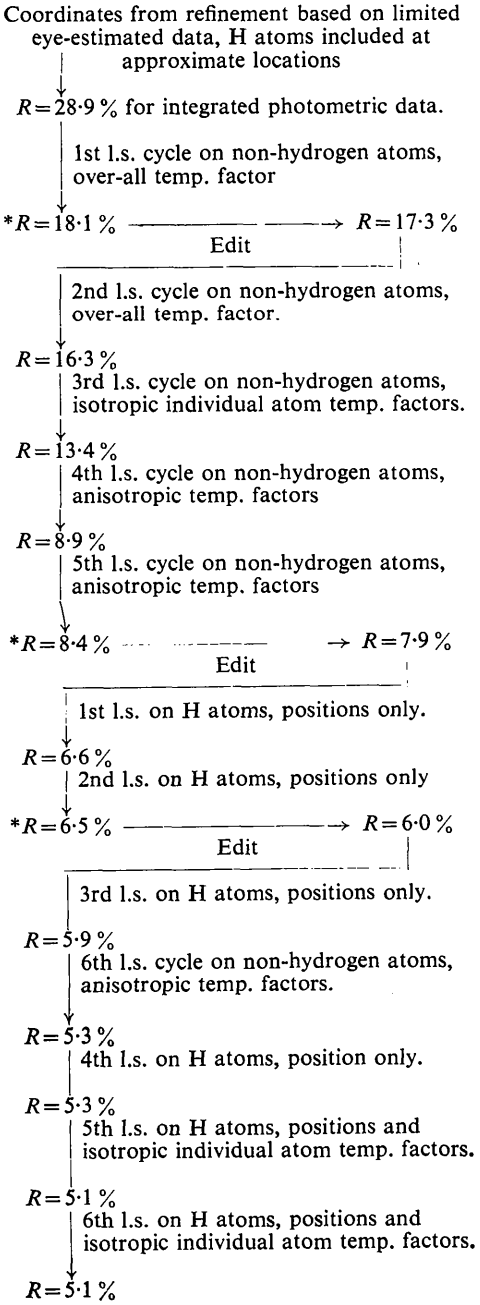


Fig. 2. Flow diagram outlining refinement of structure based on photometric data.

From this point on refinement proceeded by F_o and ΔF syntheses. Convergence was so slow, however, that there was serious doubt as to the correctness of the structure, and a reappraisal of both chain position and glyceride orientation was initiated. Chain positions were checked by F_o and ΔF syntheses based on the *subcell reflections only*, and a careful search was made for other possible positions compatible with the subcell. Validity of the glyceride orientation was checked by a Patterson synthesis based only on the *non-subcell* reflections. The net result of all the checking was to confirm the structure as correct. Refinement was continued, but it was a matter of attrition – 29 cycles were required to reduce R for the $0kl$ reflections to 26·1%. At this point the structure was apparently established in projection and refinement was terminated.

From the y,z coordinates and packing considerations, it was possible to deduce the relative positions of the three chains along a and to fix their approximate tilt and positions with respect to symmetry centers. The chain tilt was confirmed by a three-dimensional Patterson synthesis where the double zigzag to be expected for a straight chain showed to advantage. The apparent bond lengths in projection were also confirmatory and were used to fix the x parameters for the nonchain atoms.

Calculation of the structure factors for the approximately 600 observed hkl reflections resulted in $R=$ 54·5%. One refinement cycle based on an F_o synthesis decreased R to 51·3%. The second three-dimensional F_o synthesis indicated that the x coordinates for atoms C(30) and O(6), which had been derived on the basis of a regular chain, were probably wrong. A ΔF synthesis showed unequivocally that they were wrong and indicated where they should go. Beyond this point refinement proceeded by ΔF syntheses and was uneventful. As in the two-dimensional work, convergence was slow and it required 15 refinement cycles to reduce R to 23·8%. An additional 4 full-matrix least-squares refinement cycles (Busing & Levy, 1959) reduced R to 17·9%. The function minimized was $\Sigma\, w(|F_o|-k|F_c|)^2$. Standard deviations of the C atoms at this stage were about 0·04 Å.

Refinement based on photometric data

Coordinates from the last least-squares cycle based on the eye estimated data were used to begin the refinement with photometric data. The initial R was 28·9% for the full data. Fig. 2 is a summary of the course of refinement in flow diagram form. The weighting used was essentially that of Hughes (1941) where $\sqrt{w}=1\cdot0$ if $F_o\le 15$, $\sqrt{w}=15/F_o$ if $F_o>15$.

Because of the magnitude of the structure, hydrogen atoms were fixed during the refinement of the non-hydrogen atoms and conversely. Furthermore, in refinement cycles involving hydrogen atoms or when the non-hydrogen atoms were given anisotropic thermal parameters, two or three passes were required per cycle. In all multipass refinement cycles, parameters varied in each pass always included the scale factor and parameters of atoms in the glyceride moiety. Because of the extent of the binary output, it was necessary to compute on a 7094 with high density tape those cycles involving nonhydrogen atoms with anisotropic temperature factors. In each pass on the 7094, 136 parameters were varied. The time per pass was just under 20 minutes in contrast to the estimated 200 minutes on the 709.

It should be noted in Fig. 2 that on three separate occasions in the course of refinement, the data were edited. After the first refinement cycle, some gross errors were corrected, in particular indexing errors where a group of high order reflections widely separated from other reflections were in error by $l = \pm 1$.

After the fifth refinement cycle on the nonhydrogen atoms, the data were carefully edited, some minor corrections were made and about 30 very weak reflections which had been included as observed were coded as unobserved.

Before the sixth and last refinement cycle on the nonhydrogen atoms, a final stringent editing was done in which several hundred reflections were checked and about 150 corrections were made, mostly in weak reflections. In most, but not all cases, the corrections were in a direction which improved the agreement between observed and calculated structure factors. In addition, the criteria for classing very weak reflections as observed were altered so as to decrease the probability of including as observed a reflection which was in fact below the limit of detection. It was judged less serious to omit a reflection which might really be an 'observed' than to include one which was, in fact, an 'unobserved'. This resulted in coding about 30 additional very weak reflections as unobserved and a final list of 2220 observed reflections.

There can be little doubt that editing did improve the data and standard deviations in the parameters improved appreciably. There were, however, no pronounced shifts in atomic positions of the nonhydrogen atoms. The mean change in their positional parameters in the last cycle was only 0·004 Å with a maximum change of 0·018 Å, even though the data had been edited twice and the hydrogen atoms refined (mean positional change 0·36 Å).

Table 1. *Parameters for the nonhydrogen atoms and, in parentheses, their standard deviations*

All values have been multiplied by 10^5.

Thermal parameters, β_{ij}, are defined by the equation:

$$\text{T.F.} = \exp -(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})$$

	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	52969(44)	9401(23)	43462(11)	3109(98)	1016(28)	146(5)	−493(41)	−50(19)	−150(10)
O(2)	84365(52)	5028(32)	38091(11)	3096(123)	2296(49)	194(6)	−351(61)	−6(21)	−301(14)
O(3)	25539(42)	80722(22)	45681(10)	2795(92)	911(27)	100(5)	−403(39)	−86(17)	−58(9)
O(4)	55588(47)	77011(26)	39814(10)	2580(108)	1450(35)	135(6)	−661(48)	54(18)	−96(11)
O(5)	64209(48)	33730(24)	45527(11)	3571(111)	532(26)	149(6)	−38(42)	73(20)	−18(10)
O(6)	39018(53)	50239(25)	44142(12)	3618(122)	763(28)	206(7)	12(49)	−103(22)	23(11)
C(1)	99092(116)	82606(51)	2175(20)	9439(342)	1950(72)	218(11)	−1190(126)	−384(52)	−263(23)
C(2)	18458(96)	82290(49)	5970(18)	7138(260)	1879(71)	172(10)	−1121(110)	−168(42)	−133(21)
C(3)	10532(85)	88749(44)	10414(18)	5236(208)	1375(58)	168(9)	−270(87)	−196(36)	−163(18)
C(4)	30000(79)	88383(41)	14226(16)	4831(190)	1303(52)	130(8)	−350(80)	−59(32)	−152(16)
C(5)	21533(74)	95083(39)	18621(16)	4287(177)	1148(48)	143(8)	−91(72)	−144(32)	−99(16)
C(6)	40967(73)	94708(38)	22516(16)	4101(173)	1090(46)	143(8)	−273(71)	−105(31)	−126(16)
C(7)	32369(70)	1421(36)	26913(15)	3857(167)	1013(44)	138(8)	−76(68)	−100(29)	−124(15)
C(8)	51992(66)	972(34)	30752(15)	3266(146)	918(40)	127(7)	−312(61)	−61(27)	−42(14)
C(9)	42998(66)	7269(36)	35256(16)	3202(146)	1050(44)	149(8)	−72(63)	−34(28)	−188(15)
C(10)	62501(75)	6950(34)	38922(18)	3085(170)	715(40)	183(10)	−267(64)	−48(31)	−96(15)
C(11)	71348(107)	50585(52)	4948(19)	8055(302)	1876(72)	177(10)	−820(118)	−212(46)	−218(21)
C(12)	90246(91)	50165(48)	8891(18)	5915(231)	1588(63)	168(10)	−696(97)	−116(39)	−219(20)
C(13)	81221(81)	57030(43)	13229(17)	4383(190)	1362(54)	146(9)	−424(81)	−187(34)	−125(18)
C(14)	172(75)	56837(40)	17099(16)	4051(174)	1221(51)	150(8)	−72(74)	−97(31)	−151(17)
C(15)	91698(74)	63733(40)	21420(16)	3667(173)	1184(50)	132(8)	−177(74)	−74(31)	−96(16)
C(16)	11108(70)	63614(37)	25247(16)	3650(161)	975(46)	144(8)	−156(68)	−144(30)	−107(15)
C(17)	2506(71)	70386(36)	29648(15)	3527(164)	953(43)	122(8)	−280(67)	−108(29)	−43(15)
C(18)	22524(68)	70464(34)	33268(15)	3161(151)	922(42)	122(8)	−109(63)	−109(28)	−137(14)
C(19)	13525(68)	76599(37)	37835(15)	2931(150)	1030(46)	145(8)	−438(65)	−162(28)	−97(15)
C(20)	34012(73)	78068(34)	41017(16)	3354(176)	754(39)	100(7)	−419(63)	−9(28)	−2(13)
C(21)	43026(105)	19435(54)	8025(21)	7052(268)	1817(76)	216(11)	−677(116)	−320(46)	−171(23)
C(22)	61855(102)	18925(47)	11958(20)	6099(267)	1362(59)	183(11)	−440(102)	−10(45)	−146(21)
C(23)	52996(81)	26074(44)	16235(18)	4202(188)	1204(55)	174(10)	−112(81)	−44(36)	−62(19)
C(24)	71816(83)	25626(41)	20232(19)	4198(199)	993(49)	173(10)	−206(79)	−13(37)	−46(18)
C(25)	62756(82)	32734(42)	24530(18)	4562(191)	998(52)	163(9)	−124(81)	18(35)	−124(18)
C(26)	81576(80)	32407(40)	28469(18)	3968(191)	1030(48)	164(9)	−114(75)	−62(35)	−77(17)
C(27)	72226(77)	39659(39)	32687(17)	3804(177)	903(45)	159(9)	−115(70)	−109(33)	−46(16)
C(28)	91099(74)	39800(40)	36585(17)	3460(170)	951(48)	148(9)	−406(72)	16(33)	−26(17)
C(29)	81244(74)	47470(39)	40721(16)	3667(173)	890(45)	155(8)	−523(71)	−43(31)	−16(16)
C(30)	58963(79)	44352(38)	43649(16)	3910(191)	629(42)	127(8)	−140(69)	−204(31)	−58(14)
C(31)	70348(67)	10252(33)	47162(14)	3464(151)	800(39)	134(7)	−74(61)	−220(29)	−108(14)
C(32)	56090(67)	17968(33)	50802(14)	3106(140)	725(38)	129(7)	−337(58)	−138(26)	−69(14)
C(33)	44751(68)	29533(34)	48456(14)	3558(150)	787(38)	135(7)	−431(60)	−63(26)	−54(13)

The final parameters for the nonhydrogen atoms appear in Table 1 and for the hydrogen atoms, in Table 2. The observed and calculated structure factors and their differences are listed in Table 3. The final R is 5·1%.

Table 2. *Parameters of hydrogen atoms and, in parentheses, their standard deviations*

Positional parameters have been multiplied by 10^4

	x/a	y/b	z/c	B
H(1)	401 (90)	7612 (41)	−3 (19)	7·8 (1·3) Å^2
H(2)	8696 (91)	8002 (44)	417 (20)	8·5 (1·4)
H(3)	9000 (92)	9199 (45)	152 (19)	9·7 (1·5)
H(4)	2336 (89)	7448 (43)	753 (19)	9·2 (1·4)
H(5)	3255 (77)	8523 (38)	441 (17)	5·8 (1·1)
H(6)	9606 (74)	8546 (36)	1213 (16)	5·8 (1·1)
H(7)	200 (77)	9691 (38)	927 (17)	6·1 (1·2)
H(8)	3579 (71)	8043 (34)	1536 (15)	4·9 (1·0)
H(9)	4323 (66)	9101 (32)	1286 (14)	3·8 (0·9)
H(10)	694 (76)	9205 (36)	2036 (16)	6·0 (1·2)
H(11)	1651 (70)	232 (34)	1733 (15)	5·0 (1·0)
H(12)	4804 (77)	8570 (36)	2378 (16)	6·1 (1·1)
H(13)	5455 (65)	9698 (32)	2090 (14)	4·1 (0·9)
H(14)	1858 (61)	9921 (30)	2842 (13)	3·2 (0·8)
H(15)	2602 (65)	895 (32)	2620 (14)	3·6 (0·9)
H(16)	5829 (67)	9240 (32)	3185 (14)	4·3 (1·0)
H(17)	6541 (61)	364 (30)	2920 (13)	3·6 (0·9)
H(18)	3047 (56)	424 (27)	3693 (12)	2·7 (0·8)
H(19)	3646 (70)	1559 (33)	3417 (15)	4·9 (1·0)
H(20)	7810 (86)	4474 (41)	246 (17)	7·2 (1·2)
H(21)	5609 (82)	4825 (40)	654 (18)	6·6 (1·2)
H(22)	6435 (95)	5829 (47)	377 (20)	9·5 (1·5)
H(23)	9734 (80)	4173 (39)	981 (17)	7·1 (1·3)
H(24)	512 (70)	5341 (33)	713 (15)	4·5 (1·0)
H(25)	6692 (65)	5432 (32)	1480 (14)	4·1 (1·0)
H(26)	7462 (77)	6470 (38)	1190 (16)	6·0 (1·2)
H(27)	644 (72)	4965 (35)	1823 (16)	5·1 (1·1)
H(28)	1533 (66)	5899 (32)	1554 (14)	4·0 (0·9)
H(29)	7789 (60)	6131 (29)	2313 (13)	2·9 (0·8)
H(30)	8596 (65)	7094 (32)	2023 (14)	4·0 (0·9)
H(31)	1761 (72)	5475 (34)	2654 (15)	5·4 (1·1)
H(32)	2595 (61)	6684 (30)	2321 (13)	3·3 (0·9)
H(33)	8939 (63)	6797 (31)	3144 (14)	3·8 (0·9)
H(34)	9621 (63)	7794 (31)	2837 (13)	3·6 (0·9)
H(35)	3025 (66)	6228 (32)	3453 (14)	4·1 (1·0)
H(36)	3736 (64)	7357 (31)	3125 (14)	3·8 (0·9)
H(37)	203 (71)	7286 (34)	3988 (15)	5·4 (1·1)
H(38)	710 (54)	8422 (27)	3661 (12)	2·5 (0·8)
H(39)	4868 (79)	1431 (38)	519 (17)	6·3 (1·2)
H(40)	2850 (85)	1546 (41)	946 (18)	7·0 (1·3)
H(41)	3689 (82)	2729 (41)	658 (18)	7·6 (1·3)
H(42)	6753 (74)	1102 (36)	1327 (16)	5·7 (1·1)
H(43)	7633 (79)	2180 (38)	1034 (17)	6·3 (1·2)
H(44)	3794 (69)	2386 (33)	1787 (15)	4·4 (1·0)
H(45)	4596 (67)	3422 (32)	1473 (14)	4·2 (1·0)
H(46)	7575 (66)	1761 (32)	2143 (14)	4·2 (1·0)
H(47)	8582 (63)	2799 (30)	1853 (14)	3·4 (0·9)
H(48)	4796 (65)	3014 (32)	2628 (14)	3·9 (0·9)
H(49)	5679 (66)	3994 (33)	2330 (14)	4·4 (1·0)
H(50)	8720 (60)	2441 (29)	3006 (13)	3·1 (0·8)
H(51)	9719 (65)	3512 (31)	2676 (14)	4·0 (0·9)
H(52)	5740 (59)	3682 (29)	3422 (13)	3·1 (0·8)
H(53)	6603 (59)	4730 (29)	3151 (13)	3·1 (0·8)
H(54)	9717 (66)	3186 (31)	3836 (14)	4·2 (0·9)
H(55)	491 (54)	4282 (26)	3489 (12)	2·5 (0·7)
H(56)	7616 (58)	5487 (27)	3944 (13)	2·9 (0·8)
H(57)	9382 (70)	4768 (33)	4290 (15)	4·6 (1·0)
H(58)	7555 (64)	300 (31)	4919 (14)	3·8 (0·9)
H(59)	8474 (57)	1360 (28)	4587 (12)	2·3 (0·8)
H(60)	4163 (58)	1508 (27)	5242 (12)	2·6 (0·8)
H(61)	3123 (53)	2888 (26)	4607 (12)	1·9 (0·8)
H(62)	3825 (58)	3498 (27)	5094 (12)	3·0 (0·8)

In this work, the scattering factors of McWeeny (1951) were used for hydrogen and those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for carbon and oxygen.

Discussion

The configuration of the molecule is shown in the composite F_o synthesis (Fig. 3). It may be described as an unsymmetrical 'tuning fork' in which two hydrocarbon chains, I and II, along with two glyceride carbon atoms and two ester oxygen atoms form one long, almost straight, chain. The third chain, III, branches off by way of the third glyceryl C atom and an ester oxygen atom, bends and packs into the structure essentially parallel to the other chains.

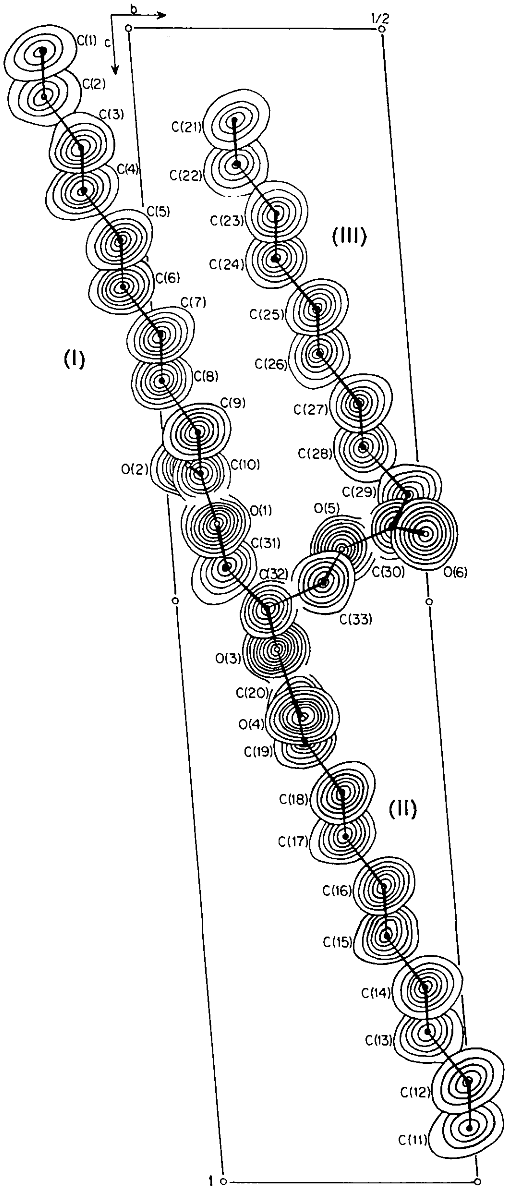


Fig. 3. Composite F_o synthesis showing electron density in sections through C and O atoms and parallel to bc plane. Contours at 1·0 e.Å^{-3}, beginning at 1·0 e.Å^{-3}.

Although the hydrocarbon chains appear to be quite regular as viewed along [100], comparison of the configuration of the chains in the region of the functional end will show that chain III differs from the other two. The difference is seen to advantage when the molecule is viewed along [010] (Jensen & Mabis, 1963) where it is evident that the zigzag of all three chains is regular except for the third chain at the functional end. There is rotation about the C(28)–C(29) bond so that C(30) and O(6) lie on opposite sides of the chain from the

Table 3. *Observed and calculated structure factors*

	0,0,L	
3	273	-270
4	348	-345
5	48	51
6	329	-325
8	121	-112
9	33	-38
10	74	-66
11	74	77
12	221	-211
13	180	-179
14	102	-103
16	61	59
17	101	-103
18	108	107
19	91	-91
20	67	60
20	72	-78
	0,1,L	
-26	278	271
-25	128	-123
-24	190	-185
-20	77	-81
-19	82	90
-18	112	-113
-17	100	99
-16	60	-55
-13	58	-45
-12	64	-62
-11	92	-90
-10	68	69
-9	119	-118
-7	42	-42
-6	141	-147
-4	184	-183
-3	44	-46
-1	140	-137
0	69	67
2	41	-43
3	119	130
4	67	73
6	419	445
7	248	-261
8	645	666
9	215	-224
10	577	563
11	255	231
12	518	-470
17	83	-77
18	185	-164
20	114	-143
21	352	-371
22	127	140
	0,6,L	
-23	65	-63
-22	60	47
-21	72	-68
-10	56	-66
-9	69	67
-8	71	-76
-6	222	-212
-5	665	-672
-4	757	754
-2	217	212
0	72	75
3	62	50
6	108	-104
7	122	113
8	126	-117
10	60	-71
11	50	44
12	78	-73
13	83	77
14	91	-85
15	86	74
16	51	-45
20	120	123
21	62	-51
30	90	-83
31	73	-71
	0,7,L	
-9	71	-68
-8	114	110
-7	199	-207
-6	188	186
-5	204	-196
-4	87	82
-3	82	68
-2	191	-187
-1	200	195
0	223	-221
1	130	139
2	107	-104
3	249	-245

corresponding atoms of chain I. It is this conformational feature of chain III that permits sufficient clearance between it and chain I. The predominant chain packing is in the parallel triclinic ($T\|$) subcell, as found for β-trilaurin by Vand & Bell (1951).

Bond lengths and angles *uncorrected* for thermal motion are shown in Fig. 4. The mean C–C bond length is between 0·03 and 0·04 Å less than the 1·544 Å C–C bond length in diamond, and the C–C–C bond angles are appreciably greater than the 112–113° to be expected for a paraffin chain. These systematically short bond lengths and large bond angles are probably attributable in part to relatively large thermal anisotropy of the chain and in part to rotary oscillation of the chains about their long axes (Cruickshank, 1956; Busing & Levy, 1964).

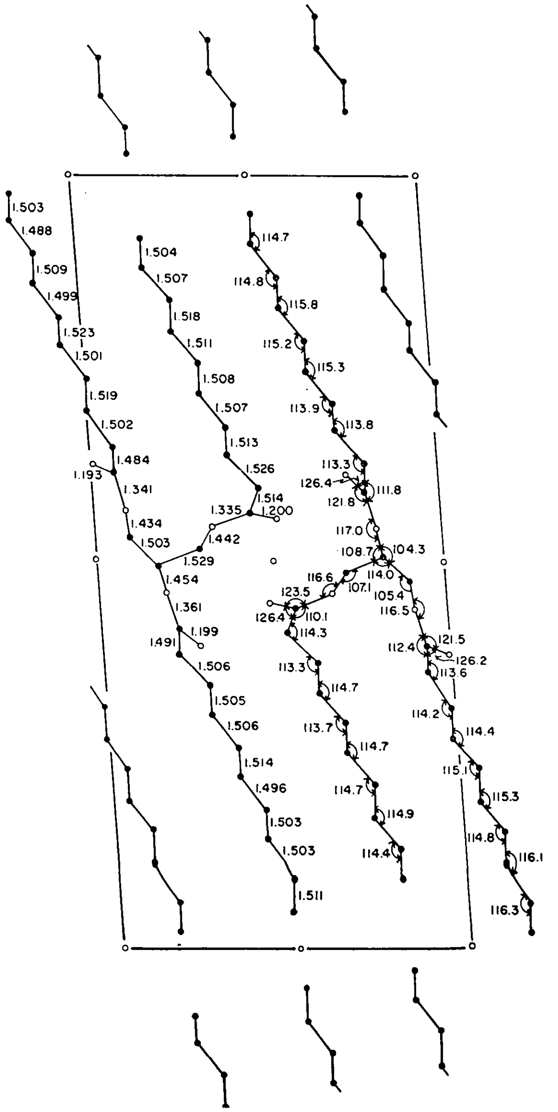


Fig. 4. Molecular outline showing bond lengths and angles not involving hydrogen atoms. Standard deviations in bond lengths about 0·005–0·009 Å, in bond angles about 0·3–0·5°.

In Fig. 5(*a*) the bond lengths for each chain are plotted for comparative purposes against the number of the bond beginning at the terminal end of the chains. The following points may be noted:

(1) The C–C bonds involving the carbonyl C atom are in each case from 0·012–0·018 Å shorter than the adjacent C–C bonds. This is to be expected for a $C(sp^2)$–$C(sp^3)$ bond.

(2) The C–C bond lengths in chain I alternate in length. This effect probably stems from thermal anisotropy and is to be expected, for example, unless one axis of the thermal ellipsoids lies parallel to the chain axis and another in the plane of the chain. The apparent alternation in the C–C bond lengths in hexamethylenediamine (Binnie & Robertson, 1950) is probably accounted for in this way.

(3) For chain I there is a slight trend in bond lengths toward shorter values for bonds toward the terminal end of the chain. This is to be expected since the thermal motion is greater toward the end of the chain.

(4) There is little indication of either alternation or a trend in the C–C bond lengths in chains II and III. This is consistent with the fact that the thermal parameters for the carbon atoms in these two chains are somewhat less than those for the carbon atoms in chain I.

(5) The most pronounced difference in bond lengths between chains involves the C(28)–C(29) and C(29)–C(30) bonds in chain III. These are 0·02–0·03 Å longer than corresponding bonds in chains I and II. The dif-

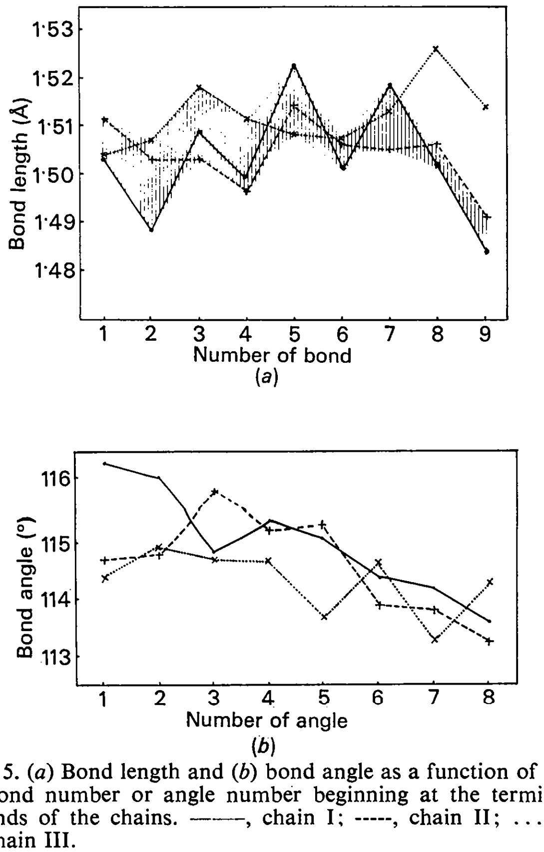


Fig. 5. (*a*) Bond length and (*b*) bond angle as a function of the bond number or angle number beginning at the terminal ends of the chains. ——, chain I; -----, chain II; ....., chain III.

ference is probably due in part to the particular orientation of the thermal ellipsoid for C(29) resulting from the peculiar twist about the C(28)–C(29) bond which destroys the regularity of chain III and interferes with possible rotary oscillation of the chain. The extreme bond lengths (except for the two end bonds of chain III) and possible systematic variation in them are emphasized by the shading in Fig. 5(*a*).

The mean C–C bond length for chain I is 1·506 ± 0·010 Å, for chain II, 1·506 ± 0·005 Å. For both chains, the C–C bond next to the carbonyl group has not been included. The average bond length in chain III is 1·510 ± 0·004 if both C–C bonds involving C(29) are excluded. The precision indices given are simple r.m.s. values derived by neglecting the fact that adjacent bond lengths are not independent.

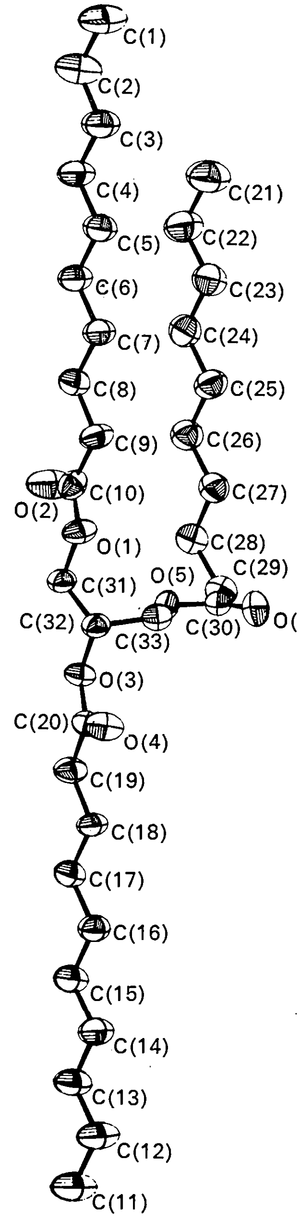


Fig. 6. Plot of thermal ellipsoids for C and O atoms as viewed along the vector [0·162, 0·023, 0·010] in triclinic fractional coordinates. Calculation and plot done by Dr C. K. Johnson, Oak Ridge National Labotarory.

A more satisfactory measure of the precision in this work may be derived from the internal agreement of the results not by averaging over the bond lengths in each chain but by averaging the lengths of the first bonds in the three chains, then the second, *etc.*, and calculating a simple r.m.s. deviation. This was done for the 23 bonds used in the averages cited in the preceding paragraph. The r.m.s. deviation of 0·0056 Å derived in this way is to be compared with the average standard deviation of 0·0066 Å calculated for these bonds from the inverse matrix of the least-squares refinement. However, since the r.m.s. value includes errors not covered in the results from least squares (*e.g.* irregularities caused by thermal motion), it could justifiably have been expected to be greater rather than less. Furthermore, standard deviations from least squares are too low in this case since they are based on calculations involving three passes per refinement cycle. In each pass, the matrix was 136 × 136 (9 parameters for each of the 10 carbon and 2 oxygen atoms of an acyl group along with the 3 carbon atoms of the glyceride plus the scale factor) rather than a single 352 × 352 matrix. Thus over half the off-diagonal terms were neglected. These terms should be small, however, so that the standard deviations from least squares can be taken as reasonable estimates for carbon and oxygen atoms.

In other parts of the molecule, irregularities in bond lengths are probably also attributable, in part, to differences in thermal motion of the atoms involved. For example, the C(31)–C(32) bond in the glyceride group is 0·026 Å shorter than the adjacent C(32)–C(33) bond. While the thermal parameters of the atoms involved in these bonds do not differ greatly, it is evident on inspecting Fig. 6 that the orientation of the thermal ellipsoids for C(31) and C(32) is quite different from that for C(33). However, thermal motion in a molecule such as *β*-tricaprin is complex and it is impossible from the present data to analyze it in detail. Thus only qualitative or semiquantitative corrections based on simplifying assumptions can be given.

The C–C–C bond angles in the chains range from 113·3° to 116·3°, and there is a trend toward smaller values toward the functional ends of the chains where thermal motion is smaller [Fig. 5(*b*)]. The mean value of 114·6° is 1·5–2·0° greater than expected. It can be shown that apparent changes in bond length and bond angle in a chain due to thermal motion or rotary oscillation should be related approximately by the equation

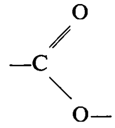
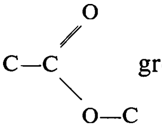
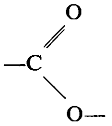
$$-\delta l = \delta\varphi \, \frac{(l)[\cos(\varphi/2)]}{[\sin(\varphi/2)](57{\cdot}3)}$$

where l and φ may be taken as the observed bond length and angle respectively and δl and $\delta\varphi$ are the changes in bond length and angle. In the present case,

$$-\delta l = \delta\varphi(0{\cdot}018)$$

so that a change of $+1{\cdot}6°$ in bond angle would correspond to a change in bond length of $-0{\cdot}029$ Å. Thus, for example, if a mean bond angle of 113° is assumed in β-tricaprin, the mean bond length would be corrected to 1·536 Å.

Although effects on bond angles are particularly evident for systematic thermal motion such as rotary oscillation, angles in general are also affected. However, unless the motion can be analyzed in detail, it is probably preferable to consider angles based on uncorrected distances (Busing & Levy, 1964). Corresponding bond angles about the three carbonyl C atoms agree well in spite of obvious differences in packing. Furthermore, the angles agree well with corresponding angles in propionic acid (Strieter, Templeton, Scheuerman & Sass, 1962) and butyric acid (Strieter & Templeton, 1962) though not so well with those in valeric acid (Scheuerman & Sass, 1962). The angles at the three ester oxygen atoms are all within 0·5° of 117° and, therefore, exceed the bond angle in water by more than 10°. The 'tetrahedral' angles in the glyceryl C atoms (excluding those involving hydrogen atoms) range from 104·3° to 114·0°. Variations here must be mainly attributable to strain. That there are also strains and packing irregularities in the chains is plainly evident when least-squares planes are calculated for each. The deviations for each atom from the planes calculated for the indicated atoms are shown in Table 4(*a*). It is evident that significant deviations from planarity occur for atoms in each of the chains. Furthermore, the deviations are systematic for chains I and III and indicate a significant bend in the same sense for each. The very large deviation of C(30) from the plane for chain III is due to the twist about the C(28)–C(29) bond already indicated. The irregularities noted here are in marked contrast to the very regular planar chains, for example, in n-nonanoic acid hydrazide (Jensen & Lingafelter, 1961), a structure where there is close packing in a monoclinic unit cell.

Planes have been calculated through each  group and the deviations of adjacent bonded atoms are shown in Table 4(*b*). It is evident that each  group is nearly planar, but there are significant deviations. The carbon atoms bonded to  groups are displaced on the same side of the plane through these atoms for chains I and II and on opposite sides for chain III.

The principal axes of the thermal ellipsoids for the nonhydrogen atoms and their orientations are listed in Table 5. A plot of the thermal ellipsoids in the molecule as viewed along the vector [0·162, 0·023, 0·010] is shown in Fig. 6. The greater thermal motion to be expected toward the terminal ends of the chains shows plainly in Fig. 6 and may also be inferred from decreased peak height of the terminal carbon atoms in Fig. 3.

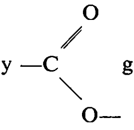
Fig. 7 is a composite of sections from a ΔF synthesis showing the 62 hydrogen atoms as viewed along [010]. The same decrease in peak density observed for carbon atoms toward the ends of the chains is also evident, in general, for hydrogen.

Table 4. *Deviations of atoms from various planes*

Deviations of atoms not used in determining planes are enclosed in parentheses.

(*a*) Least-squares planes for chains

Atom	δ	Atom	δ	Atom	δ
C(1)	(0·012) Å	C(11)	(0·070) Å	C(21)	(0·038) Å
C(2)	0·009	C(12)	−0·006	C(22)	0·008
C(3)	0·005	C(13)	0·016	C(23)	0·019
C(4)	0·007	C(14)	−0·027	C(24)	−0·012
C(5)	−0·015	C(15)	0·013	C(25)	−0·012
C(6)	−0·002	C(16)	−0·009	C(26)	−0·025
C(7)	−0·022	C(17)	0·006	C(27)	−0·009
C(8)	−0·012	C(18)	0·041	C(28)	0·031
C(9)	0·031	C(19)	−0·035	C(29)	(0·085)
C(10)	(0·012)	C(20)	(0·200)	C(30)	(−1·100)

(*b*) Planes determined by  groups

C(9)	(−0·036) Å	C(19)	(0·019) Å	C(29)	(0·055) Å
C(10)	0	C(20)	0	C(30)	0
O(1)	0	O(3)	0	O(5)	0
O(2)	0	O(4)	0	O(6)	0
C(31)	(−0·082)	C(32)	(0·025)	C(33)	(−0·059)

Table 5. *Description of thermal ellipsoids*

$B_i = 8\pi^2\bar{u}_i^2$ where $\bar{u}_i$ is the r.m.s. displacement corresponding to the ith axis of the ellipsoid. C_{ia}, C_{ib}, C_{ic} are the direction cosines of the i axis with respect to the orthogonal coordinate axes by **a**, **c*** × **a** and **c***.

Atom	Axis i	B_i	C_{ia}	C_{ib}	C_{ic}
O(1)	1	2·70	−0·504	0·051	0·862
	2	3·86	−0·786	−0·441	−0·433
	3	6·59	0·358	−0·896	0·262
O(2)	1	3·58	−0·883	−0·455	0·115
	2	4·18	−0·081	−0·093	−0·992
	3	14·20	−0·462	0·886	−0·045
O(3)	1	2·36	−0·380	−0·206	0·902
	2	3·48	−0·877	−0·230	−0·422
	3	5·26	−0·295	0·951	0·093
O(4)	1	2·70	−0·964	−0·262	−0·029
	2	3·75	0·003	0·098	−0·995
	3	8·34	−0·264	0·960	0·093
O(5)	1	2·85	0·096	0·949	0·301
	2	3·86	−0·799	0·254	−0·545
	3	5·05	−0·593	−0·188	0·783
O(6)	1	3·42	0·331	0·930	−0·159
	2	5·22	0·940	−0·341	−0·035
	3	6·23	0·086	0·132	0·987
C(1)	1	3·30	−0·255	0·056	0·965
	2	11·58	−0·936	−0·266	−0·231
	3	12·13	−0·244	0·962	−0·120
C(2)	1	4·18	−0·141	−0·096	0·985
	2	8·04	−0·989	−0·038	−0·145
	3	11·04	−0·051	0·995	0·090
C(3)	1	3·62	−0·197	−0·026	0·980
	2	6·10	0·691	0·705	0·159
	3	8·91	0·695	−0·708	0·121
C(4)	1	3·00	0·028	0·067	0·997
	2	5·63	0·839	0·540	−0·060
	3	7·98	−0·543	0·839	−0·041
C(5)	1	3·55	−0·162	−0·164	0·973
	2	4·82	−0·647	−0·727	−0·231
	3	7·39	−0·745	0·666	−0·012
C(6)	1	3·29	−0·170	0·034	0·985
	2	4·78	0·769	0·629	0·111
	3	6·92	0·616	−0·776	0·133
C(7)	1	3·19	−0·054	0·112	0·992
	2	4·25	0·684	0·728	−0·045
	3	6·82	0·728	−0·676	0·116
C(8)	1	3·42	−0·370	−0·311	0·875
	2	3·96	−0·833	−0·306	−0·461
	3	5·26	−0·411	0·900	0·146
C(9)	1	2·76	0·155	0·397	0·905
	2	3·72	0·794	0·495	−0·353
	3	7·44	0·588	−0·773	0·238
C(10)	1	3·42	−0·699	0·250	0·670
	2	3·65	−0·554	−0·782	−0·286
	3	5·71	0·453	−0·571	0·685
C(11)	1	3·68	−0·104	0·013	0·994
	2	9·56	0·890	0·447	0·087
	3	11·34	−0·444	0·894	−0·058
C(12)	1	3·35	−0·148	0·080	0·986
	2	6·92	−0·908	−0·406	−0·103
	3	9·89	−0·392	0·910	−0·132
C(13)	1	3·13	−0·306	−0·158	0·939
	2	5·54	−0·802	−0·488	−0·344
	3	8·14	−0·513	0·858	−0·022
C(14)	1	3·45	−0·025	0·090	0·996
	2	4·55	0·742	0·670	−0·042
	3	8·04	−0·670	0·737	−0·083
C(15)	1	3·42	−0·080	−0·122	0·983
	2	4·25	−0·807	−0·575	−0·136
	3	7·15	−0·585	0·809	0·053
C(16)	1	3·19	−0·335	−0·109	0·936
	2	4·29	−0·615	−0·727	−0·305
	3	6·41	0·714	−0·678	0·176
C(17)	1	3·16	−0·254	−0·309	0·916
	2	4·36	−0·816	−0·440	−0·375
	3	5·50	−0·519	0·843	0·140
C(18)	1	2·47	−0·188	0·079	0·979
	2	3·62	0·726	0·683	0·084
	3	6·32	0·662	0·727	0·185
C(19)	1	2·45	−0·638	−0·204	0·742
	2	4·47	−0·629	−0·418	−0·656
	3	6·19	−0·443	0·886	−0·139
C(20)	1	2·82	0·415	−0·324	0·850
	2	3·68	0·900	0·012	−0·435
	3	4·47	0·131	0·946	0·296
C(21)	1	4·51	−0·294	−0·110	0·950
	2	8·86	−0·753	−0·585	−0·301
	3	10·99	−0·589	0·803	−0·089
C(22)	1	4·55	0·110	0·189	0·976
	2	7·11	0·831	0·521	−0·195
	3	8·34	−0·545	0·832	−0·098
C(23)	1	4·74	−0·749	−0·656	−0·090
	2	4·90	−0·045	0·185	−0·982
	3	7·35	−0·661	0·731	0·168
C(24)	1	4·66	0·672	0·572	0·470
	2	5·01	0·209	0·462	−0·862
	3	5·98	−0·710	0·677	0·191
C(25)	1	3·68	0·262	0·451	0·853
	2	5·38	0·618	0·601	−0·507
	3	6·69	0·742	−0·660	0·121
C(26)	1	4·40	−0·425	−0·466	0·776
	2	4·44	0·547	0·551	−0·631
	3	6·50	−0·822	0·692	−0·021
C(27)	1	4·00	−0·495	−0·650	0·577
	2	4·59	−0·282	−0·508	−0·814
	3	5·76	−0·822	0·566	−0·068
C(28)	1	3·86	0·946	0·154	0·286
	2	4·40	0·223	0·332	−0·917
	3	5·42	−0·237	0·931	0·279
C(29)	1	3·93	−0·943	−0·011	0·334
	2	4·44	−0·310	0·400	−0·863
	3	5·26	0·124	0·917	0·380
C(30)	1	2·73	−0·316	0·007	0·949
	2	3·55	−0·074	−0·997	−0·017
	3	5·46	0·946	−0·076	0·315
C(31)	1	2·47	−0·380	−0·086	0·921
	2	3·79	−0·411	−0·876	−0·252
	3	6·10	0·829	−0·474	0·298
C(32)	1	2·56	−0·533	−0·035	0·846
	2	4·00	−0·479	−0·812	−0·335
	3	4·66	0·698	−0·583	0·416
C(33)	1	3·35	−0·462	0·057	0·885
	2	4·25	−0·884	−0·104	−0·456
	3	4·70	−0·066	0·993	−0·098

The C–H bond lengths range from 0·90–1·16 Å with a mean value of 0·99 Å Table 6(*a*). This value is to be compared with the mean C–H bond length of 1·037 Å in *N*,*N*′-hexamethylenebispropionamide and 1·062 Å in n-nonanoic acid hydrazide where the thermal motion for the nonhydrogen atoms is considerably less than that in *β*-tricaprin. It might have been expected that C–H bonds near the terminal ends of the chains would be shorter than those farther removed, but there seems to be no evidence for such an effect.

The standard deviations in position of the hydrogen atoms from the least-squares refinement range from 0·029 to 0·057 Å with a r.m.s. value of 0·040 Å. This may be compared directly with the r.m.s. deviations in the lengths of the 62 C–H bonds since the standard deviations in these bonds are essentially the same as the standard deviations of the hydrogen atoms. As expected, the r.m.s. value from the C–H bond lengths, 0·055 Å, is considerably larger than the value from the least-squares refinement which should represent a lower limit. This follows from the necessity of refining hydrogen atoms separately so that all cross terms between hydrogen and nonhydrogen atoms were neglected.

In Table 6(*b*) are listed the H–C–H bond angles. The mean value is 107·5°, range 97–135°. It is not surprising that the extreme values are associated with hydrogen atoms of the terminal methyl groups.

A plot of mean $B'_{\rm H}$ for the hydrogen atoms bonded to a given C atom *vs.* mean B_{ii} for that atom is shown in Fig. 8. The plot shows that $B'_{\rm H}$ tends to be lower than $B_{\rm nonH}$, a tendency found in other compounds when $B_{\rm nonH}$ is less than 4·5 Å^2, and may be taken as evidence supporting the idea that the scattering factor for the bonded hydrogen atom exceeds that for the isolated atom used in this refinement. However, in two other compounds investigated (Jensen & Sundaralingam, 1964; Jensen, 1965), $B'_{\rm H}$ for the hydrogen atoms tend markedly to exceed $B_{\rm nonH}$ at larger values, whereas in β-tricaprin there is little indication of such an effect. This is probably due to triclinic packing in β-tricaprin which produces an effect similar to that found for the *terminal* hydrogen atoms in the other chain

Table 6.

(*a*) C–H Bond lengths

C(1)—H(1)	1·02 Å	C(16)-H(32)	1·08 Å
C(1)—H(2)	0·92	C(17)-H(33)	0·93
C(1)—H(3)	1·16	C(17)-H(34)	0·96
C(2)—H(4)	1·00	C(18)-H(35)	1·04
C(2)—H(5)	0·97	C(18)-H(36)	1·07
C(3)—H(6)	1·03	C(19)-H(37)	0·97
C(3)—H(7)	1·04	C(19)-H(38)	0·97
C(4)—H(8)	0·99	C(21)-H(39)	1·03
C(4)—H(9)	0·90	C(21)-H(40)	1·05
C(5)—H(10)	1·02	C(21)-H(41)	1·00
C(5)—H(11)	0·92	C(22)-H(42)	1·00
C(6)—H(12)	1·12	C(22)-H(43)	1·00
C(6)—H(13)	0·92	C(23)-H(44)	1·01
C(7)—H(14)	0·92	C(23)-H(45)	1·05
C(7)—H(15)	0·92	C(24)-H(46)	0·99
C(8)—H(16)	1·06	C(24)-H(47)	0·96
C(8)—H(17)	0·93	C(25)-H(48)	1·01
C(9)—H(18)	0·92	C(25)-H(49)	0·92
C(9)—H(19)	1·03	C(26)-H(50)	1·03
C(11)-H(20)	1·02	C(26)-H(51)	1·05
C(11)-H(21)	1·00	C(27)-H(52)	1·00
C(11)-H(22)	0·98	C(27)-H(53)	0·96
C(12)-H(23)	1·04	C(28)-H(54)	1·05
C(12)-H(24)	1·05	C(28)-H(55)	0·98
C(13)-H(25)	0·97	C(29)-H(56)	0·94
C(13)-H(26)	0·98	C(29)-H(57)	0·93
C(14)-H(27)	0·91	C(31)-H(58)	1·00
C(14)-H(28)	0·98	C(31)-H(59)	0·99
C(15)-H(29)	0·95	C(32)-H(60)	1·00
C(15)-H(30)	0·91	C(33)-H(61)	1·02
C(16)-H(31)	1·10	C(33)-H(62)	0·99

(*b*) H–C–H Bond angles

H(1)—C(1)—H(2)	100°	H(31)-C(16)-H(32)	110°
H(1)—C(1)—H(3)	135	H(33)-C(17)-H(34)	106
H(2)—C(1)—H(3)	100	H(35)-C(18)-H(36)	105
H(4)—C(2)—H(5)	112	H(37)-C(19)-H(38)	116
H(6)—C(3)—H(7)	102	H(39)-C(21)-H(40)	97
H(8)—C(4)—H(9)	107	H(39)-C(21)-H(41)	109
H(10)-C(5)-H(11)	111	H(40)-C(21)-H(41)	112
H(12)-C(6)—H(13)	104	H(42)-C(22)-H(43)	109
H(14)-C(7)—H(15)	99	H(44)-C(23)-H(45)	101
H(16)-C(8)—H(17)	107	H(46)-C(24)-H(47)	111
H(18)-C(9)—H(19)	109	H(48)-C(25)-H(49)	105
H(20)-C(11)-H(21)	106	H(50)-C(26)-H(51)	108
H(20)-C(11)-H(22)	120	H(52)-C(27)-H(53)	105
H(21)-C(11)-H(22)	100	H(54)-C(28)-H(55)	111
H(23)-C(12)-H(24)	105	H(56)-C(29)-H(57)	106
H(25)-C(13)-H(26)	105	H(58)-C(31)-H(59)	112
H(27)-C(14)-H(28)	100	H(61)-C(33)-H(62)	111
H(29)-C(15)-H(30)	105		

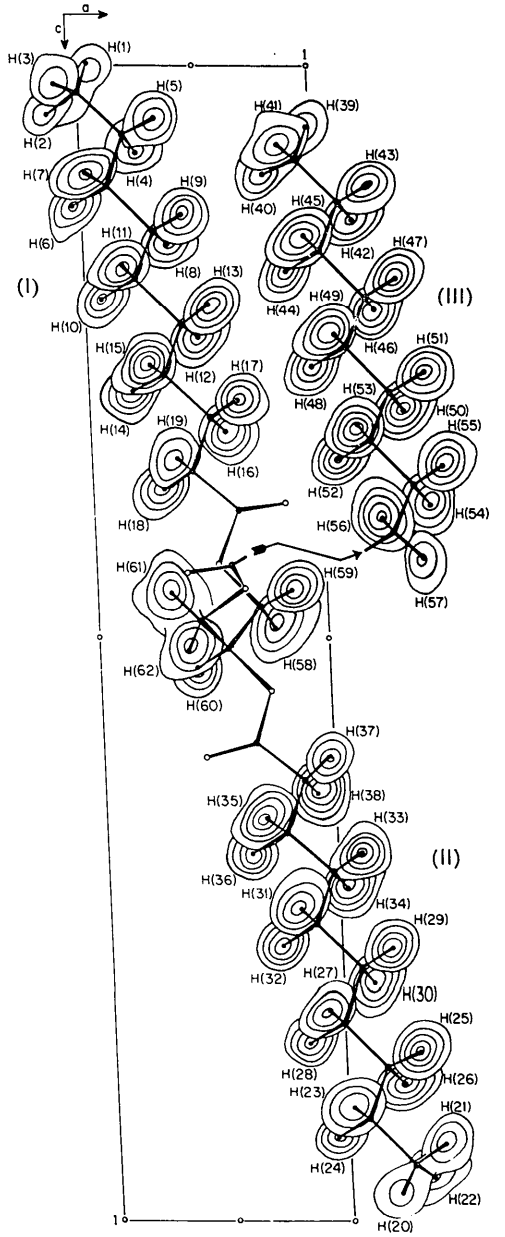


Fig. 7. Composite ΔF synthesis showing electron density in sections through H atoms and parallel to *ac* plane. Contours at 0·1 e.Å^{-3}, beginning at 0·1 e.Å^{-3}. Chain III displaced as indicated by arrow.

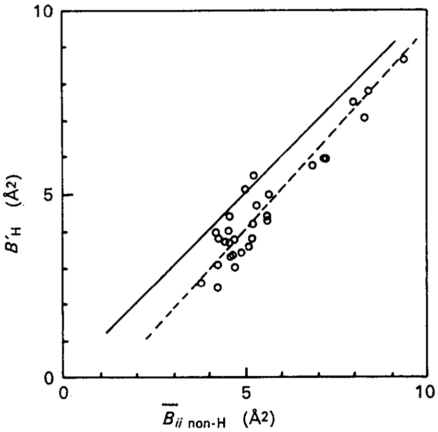


Fig. 8. Plot of apparent thermal parameters B'_H for the H atoms bonded to a given C atom *vs.* mean B_{ii} for that atom. Except for B'_{H60}, all others are average B'_H for the H atoms bonded to a given C atom.

compounds investigated. Furthermore, much of the scatter in the results for β-tricaprin probably stems from irregularities of the triclinic packing of the chains.

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