[C(9)—C(11)] = 1·2°. The best mirror passes through C(11) and C(14), with ΔC_s[C(11)] = 3·1°. Ring B is in the C(8)β—C(9)α-half-chair conformation with C(8) and C(9) −0·360 and +0·343 Å away from the plane formed by C(5), C(6), C(7) and C(10). The asymmetry parameter ΔC_2[C(5)—C(6)] = 1·3°.

The large value for the asymmetry parameter {ΔC_s[C(14)] = 8·5°} shows that ring D is in a distorted C(14)α-envelope conformation. The phase angle of pseudorotation $\Delta = -47·4°$ and maximum angle of torsion $\varphi = 40·3°$ (Altona, Geise & Romers, 1968). Ring E, which has the partial double bond [N(1)—C(20) = 1·33 Å] shows the C(13)—C(17)-half-chair conformation.

The two water molecules are involved in four hydrogen bonds. OW(1) forms three H bonds, two with symmetry-related OW(2) molecules and one with the N(2) atom. OW(2) forms one more H bond with O(1). The torsional angle C(1)—C(10)—C(13)—C(18), which gives the twist of the steroid molecule about the line joining C(10) and C(13), is 50·4°.

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Structure of (±)-*threo*-9,10,16-Trihydroxypalmitic Acid

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Abstract. 9,10,16-Trihydroxyhexadecanoic acid, $C_{16}H_{32}O_5$, $M_r = 304·4$, triclinic, $P\bar{1}$, $a = 4·871$ (1), $b = 8·801$ (1), $c = 21·121$ (2) Å, $\alpha = 89·54$ (1), $\beta = 84·88$ (2), $\gamma = 76·35$ (2)°, $V = 876·14$ (4) Å^3, $Z = 2$, $D_x = 1·154$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·26$ cm^{-1}, $F(000) = 336$, $T = 295$ K. Final $R = 0·055$ for 1556 reflections with $I \geq 3\sigma(I)$. The 16 C atoms form an all-*trans* zigzag chain. Each molecule is linked to seven neighboring molecules *via* eight possible hydrogen bonds with O···O distances varying from 2·591 (6) to 2·673 (3) Å. Each carboxyl group is potentially hydrogen-bonded to the carboxyl and C16—OH groups of other molecules with O···O distances of 2·591 (6) and 2·644 (4) Å. The conformation around the C9—C10 bond is *gauche* with an O3—C9—C10—O4 torsion angle of 66·4 (5)°.

Introduction. 9,10,16-Trihydroxypalmitic acid exists as four stereoisomers. Aleuritic acid (I) [(±)-*threo*-9,-10,16-trihydroxypalmitic acid], a major constituent of natural shellac (Nagel, 1927), has been studied by various groups (*e.g.* Harries & Nagel, 1922; Ames, Goodburn, Jevans & McGhie, 1968; Eswaran, Seshadri, Sriram & Subramanian, 1971; Chatterjea, Sengupta, Mangee & Mukherjee, 1976). The crystal structure of (I) was originally determined with photographic data by conventional techniques to an $R = 0·11$ (Prasad & Gupta, 1975). It has been redetermined with diffractometer data, the results of which are presented here.

$$HO_2C\text{-}(CH_2)_7\text{-}\underset{OH}{C}H\text{-}\underset{OH}{C}H\text{-}(CH_2)_6\text{-}OH$$

(I)
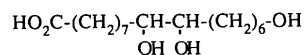
Experimental. Crystals obtained by repeated crystallization from aqueous ethanol of a sample supplied by Fluka Chemical Corp. (catalog No. 05540, compound mislabeled as DL-erythro-9,10,16-trihydroxypalmitic acid); 0·5 × 0·3 × 0·05 mm crystal. Enraf–Nonius CAD-4 diffractometer; Mo radiation with incident-beam monochromator; cell parameters from 25 reflections centered in the range $8·5 < \theta < 15·4°$; $2\theta–\theta$ scan at variable scan speed of 8·24–1·03° min^{-1}; θ scan range of 1·5(1·0° + 0·34° $\tan\theta$); six standards measured every 2 h of X-ray exposure; 3237 data (includes standards) measured from $\theta = 1·5–25°$; index range for h, k, $l = -5$ to 5, -10 to 10, 0 to 25; each scan recorded as 96 steps and subsequently processed with a modified Lehmann–Larsen procedure (Lehmann & Larsen, 1974; Ammon, 1986); 3126 unique reflections; 1556 reflections with $I > 3\sigma(I)$; average change in standard intensities of $-1·6\%$ with range of $-3·5$ to $+0·6\%$. Absorption ignored. All crystallographic calculations performed with the *TEXSAN* (Molecular Structure Corporation, 1985) program system on a DEC MicroVAX II computer; structure resolved with *MITHRIL* (Gilmore, 1983) direct-methods link; refinement by full-matrix least squares with anisotropic temperature factors for C and O; $\sum w(F_o - F_c)^2$ minimized, $w = 1/\sigma^2(F_o)$; C-linked H atoms were positioned from the C skeleton and refined with isotropic terms; O-linked H atoms could not be located definitively in difference maps; ten highest peaks in final difference map (0·302–0·144 e Å^{-3}) were in vicinities of O atoms; secondary-extinction parameter [method of Zachariasen (1968)] refined; final S, R and wR values of 1·9, 0·055 and 0·063; maximum shift/e.s.d. of 0·59; minimum and maximum values in the final difference map of $-0·164$ and 0·302 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.*

Discussion. An *ORTEP* (Johnson, 1965) diagram of the molecule is shown in Fig. 1. Bond lengths and bond angles are listed in Table 2. The 16 C atoms form an all-*trans* zigzag chain typical of long-chain fatty acids. The C—C bond lengths vary from 1·487 (6) to 1·528 (5) Å, average of 1·512 (6) Å; C—C—C angles range from 113·1 (4) to 115·5 (4)°, average of 114·1 (4)°. The maximum deviation of a C atom from the least-squares plane through the 16 C atoms is 0·261 (6) Å (C16), average deviation of 0·107 Å. The plane of the carboxyl group (O1,O2,C1,C2) is rotated out of the 16 C plane by 26·2°. The three C—O bond

lengths not involving the carboxyl group are nearly equal at C9—O3 = 1·431 (4), C10—O4 = 1·437 (5) and C16—O5 = 1·433 (5) Å.

Table 1. *Atomic fractional coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j,$$

	x	y	z	B_{eq}(Å^2)
O1	0.2397 (7)	−0.0951 (4)	0.0094 (1)	5.8 (2)
O2	0.5532 (7)	−0.1942 (4)	0.0766 (1)	5.3 (2)
O3	0.2585 (5)	0.3622 (3)	0.5144 (1)	3.6 (2)
O4	−0.3221 (5)	0.4271 (3)	0.5743 (1)	3.5 (2)
O5	−0.1674 (7)	0.6306 (4)	0.9793 (1)	5.9 (2)
C1	0.318 (1)	−0.1056 (6)	0.0652 (2)	4.5 (3)
C2	0.132 (1)	−0.0138 (7)	0.1187 (2)	4.4 (3)
C3	0.276 (1)	0.0024 (6)	0.1782 (2)	4.0 (3)
C4	0.071 (1)	0.0723 (7)	0.2345 (2)	4.0 (3)
C5	0.219 (1)	0.0915 (6)	0.2935 (2)	4.0 (3)
C6	0.021 (1)	0.1469 (7)	0.3521 (2)	3.7 (3)
C7	0.168 (1)	0.1626 (6)	0.4112 (2)	3.5 (3)
C8	−0.0346 (9)	0.2156 (5)	0.4694 (2)	3.0 (2)
C9	0.1052 (8)	0.2441 (5)	0.5287 (2)	2.9 (2)
C10	−0.1033 (8)	0.2921 (5)	0.5870 (2)	3.0 (2)
C11	0.036 (1)	0.3253 (6)	0.6453 (2)	3.5 (3)
C12	−0.171 (1)	0.3775 (7)	0.7030 (2)	4.0 (3)
C13	−0.035 (1)	0.4169 (8)	0.7605 (2)	4.3 (3)
C14	−0.245 (1)	0.4866 (6)	0.8156 (2)	4.1 (3)
C15	−0.108 (1)	0.5251 (8)	0.8732 (2)	4.7 (3)
C16	−0.314 (1)	0.5970 (7)	0.9273 (2)	4.8 (3)

Table 2. *Bond distances (Å), angles (°) and e.s.d.'s (in parentheses)*

O1	C1		1·266 (5)	C6	C7		1·518 (6)
O2	C1		1·265 (5)	C7	C8		1·508 (5)
O3	C9		1·431 (4)	C8	C9		1·528 (5)
O4	C10		1·437 (5)	C9	C10		1·518 (5)
O5	C16		1·433 (5)	C10	C11		1·520 (5)
C1	C2		1·495 (6)	C11	C12		1·508 (6)
C2	C3		1·517 (6)	C12	C13		1·512 (6)
C3	C4		1·512 (6)	C13	C14		1·508 (6)
C4	C5		1·524 (6)	C14	C15		1·515 (6)
C5	C6		1·507 (6)	C15	C16		1·487 (6)
O2	C1	O1	120·7 (4)	O3	C9	C8	108·1 (3)
O2	C1	C2	119·1 (4)	C10	C9	C8	113·7 (3)
O1	C1	C2	120·1 (4)	O4	C10	C9	110·9 (3)
C1	C2	C3	115·5 (4)	O4	C10	C11	109·1 (3)
C4	C3	C2	113·6 (4)	C9	C10	C11	113·4 (3)
C3	C4	C5	113·1 (4)	C12	C11	C10	113·8 (4)
C6	C5	C4	114·4 (4)	C11	C12	C13	114·1 (4)
C5	C6	C7	114·4 (4)	C14	C13	C12	114·1 (4)
C8	C7	C6	113·4 (4)	C13	C14	C15	113·9 (4)
C7	C8	C9	114·8 (4)	C16	C15	C14	114·1 (5)
O3	C9	C10	110·0 (3)	O5	C16	C15	110·4 (4)

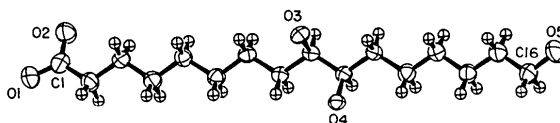
Fig. 1. *ORTEP* drawing of (I). The C and O atoms are shown as 50% boundary ellipsoids; H atoms are drawn as spheres with $B = 1·5$ Å^2.

Each molecule may be linked to seven neighboring molecules with eight possible H$\cdots$O bonds; the O$\cdots$O distances range from 2·591 (6) to 2·673 (4) Å (see Fig. 2 and Table 3). Our inability to locate the O-linked H atoms necessarily makes definite assignment speculative. Unlike the situation in many carboxylic acids, (I) does not form doubly-hydrogen-bonded dimers in which each half of the dimer serves as both a hydrogen-bond donor and acceptor to the other half. Examples of fatty-acid crystals in which carboxylic acid groups face each other across centers of symmetry are isostearic acid (Abrahamsson & Lunden, 1972), lauric acid (Goto & Asada, 1978) and the B' form of heptadecanoic acid (Goto & Asada, 1984); the average carboxyl intermolecular O$\cdots$O distance is 2·67 Å. In (I), however, the carboxyl group [in molecule (i) at x, y, z; see Fig. 2 and Table 3] appears to be simultaneously hydrogen-bonded to the carboxyl of molecule (ii) and to the C16 hydroxyl (O5) of molecule (iii). In view of the virtually identical carboxyl C—O distances [C1—O1 = 1·266 (5), C1—O2 = 1·264 (5) Å], which are between the usual carboxyl C—O single- and double-bond values [e.g. C=O = 1·214 (3), C—O = 1·304 (3) Å in α-ketoglutaric acid; Lis & Matuszewski, 1984], it would appear that both carboxyl carbon—oxygen bond orders in (I) are close to 1·5. This interpretation, together with the end-hydroxyl-to-carboxyl interactions, could lead to a description of the O$\cdots$O linkages, illustrated in Fig. 2, as: (a) O$\cdots$H$\cdots$O, in which the H atoms are located halfway between the O atoms; or (b) O—H$\cdots$O, O$\cdots$H—O, in which the actual space group of P1 averages to $P\bar{1}$ as a consequence of a static or dynamic disorder phenomenon. In the case of (b), the hydrogen-bond network implied in Fig. 2 could be depicted as O5^v—H$\cdots$O5^{iii}—H$\cdots$O2^i=C1—O1—H$\cdots$O1^{ii}=C1—O2—H$\cdots$O5^{vi}—H$\cdots$O5^{iv}⇌O5^v$\cdots$H—O5^{iii}$\cdots$H—O2^i—C1=O1$\cdots$H—O1^{ii}—C1=O2$\cdots$H—O5^{vi}$\cdots$H—O5^{iv}. Such a structure would extend, of course, over many unit cells.
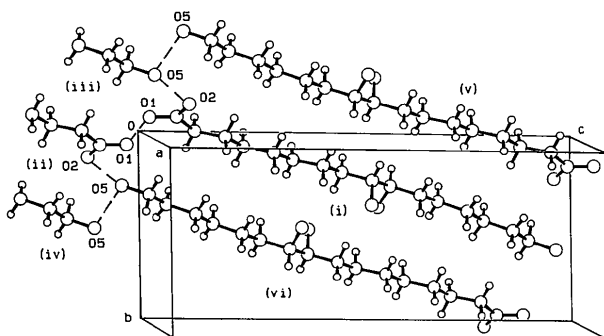

Fig. 2. *ORTEP* packing diagram showing molecules related by symmetry operations (i)–(vi). Only portions of the (ii)–(iv) molecules are shown. The symmetry operations and O$\cdots$O distances are listed in Table 3. The diagram labels were inserted with the program *PLOTMD* (Luo, Ammon & Gilliland, 1989) running on a VAXStation II.

Table 3. O$\cdots$O *distances* (Å) *and symmetry operations*

O1^i$\cdots$O1^{ii}	2·591 (6)	O3^i$\cdots$O4^{viii}	2·673 (4)
O2^i$\cdots$O5^{iii}	2·644 (4)	O5^i$\cdots$O5^{ix}	2·673 (7)
O3^i$\cdots$O4^{vii}	2·669 (4)		

Symmetry operations: (i) x, y, z; (ii) $-x, -y, -z$; (iii) $1+x, -1+y, -1+z$; (iv) $-1+x, y, -1+z$; (v) $1-x, -y, 1-z$; (vi) $-1-x, 1-y, 1-z$; (vii) $1+x, y, z$; (viii) $-x, 1-y, 1-z$; (ix) $-x, 1-y, 2-z$.

The two center-most hydroxyl groups each form two interactions with distances of 2·669 (4) (O3^i$\cdots$O4^{vii}) and 2·673 (4) Å (O3^i$\cdots$O4^{viii}). These distances are listed in Table 3, but are not shown in Fig. 2.

The O3—C9—C10—O4 torsion angle is 66·4 (4)°, a *gauche* conformation. Additionally, the configuration is *threo*, confirming the findings of a number of workers (Ames *et al.*; Wittcoff & Miller, 1947; Gensler & Schlein, 1956).

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