acetamide compound indicates that there is no justification for doubling this spacing on the assumption that the first, and longest, reflection order has not been observed. Under identical conditions of measurement the longer first-order reflections of the myristic, palmitic and stearic acid addition compounds are observed. Furthermore, when the length of the first observed X-ray line in the pattern of the lauric acid–acetamide compound is doubled, only every other reflection order is obtained. Finally, the characteristic short spacings for the lauric acid adduct exhibit a somewhat different pattern from similar spacings for the "C" forms of the other adducts. The three strongest short spacings for the "C" forms of the addition compounds, for example

	I	II	III
Lauric	4.21	3.90	3.76
Myristic	4.43	4.12	3.74
Palmitic	4.45	4.12	3.72
Stearic	4.46	4.06	3.77

reveal the difference in the pattern of the lauric acid compound.

Under "X" in Table I is listed the long spacing for the myristic acid previously shown to agree with the value in parentheses reported by Slagle and Ott[3] for an unstable modification of this acid.

The fact that the two modifications of the fatty acid–acetamide compounds correspond to the "A" and "C" forms of the fatty acids suggested the possibility of preparing the "A" forms of the acids by dissolving the acetamide from the acetamide compound without melting. It was found, however, that when the acetamide was removed from the "A" form of the palmitic acid–acetamide compound by extraction with cold water, the X-ray diffraction pattern of the resulting product was that of the "C" form of the free palmitic acid.

There is a considerable difference between the short spacings listed in Table I for the two polymorphic forms of each of the molecular compounds. A combination of long and short spacings permits a ready and a positive identification of any of the addition compounds and of its polymorphic form.

NEW ORLEANS, LOUISIANA

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF PITTSBURGH]

The Crystallography and Structure of Some C_{19} Cyclopropane Fatty Acids[1]

BY T. BROTHERTON AND G. A. JEFFREY

RECEIVED MARCH 4, 1957

The unit cell dimensions and space groups have been determined for four synthetic fatty acids, *cis*- and *trans*-DL-9,10-methylene-octadecanoic and *cis*- and *trans*-DL-11,12-methylene-octadecanoic, and two natural products, lactobacillic acid and dihydrosterculic acid. The crystals of the two *cis* and two *trans* synthetic acids, respectively, are isomorphous for reasons apparent from the packing shape of the molecules. The crystal structure of the dihydrosterculic acid is identical with that of the *cis*-DL-9,10 synthetic acid. The lactobacillic acid crystal structure is different from that of any of the synthetic products. The crystal data in combination with chemical evidence point to the *cis*-D- or L-11,12-methylene-octadecanoic acid structure for lactobacillic acid. From powder diffraction data the acid amides are shown to have similar crystal structural relationships.

The Crystallography of the C_{19} Cyclopropane Acids.—Six $C_{19}H_{36}O_2$ cyclopropane fatty acids are known from the work of Hofmann and his collaborators[2–5]; the *cis*- and *trans*-DL-9,10-methylene-octadecanoic acids and the *cis*- and *trans*-DL-11,12-methylene-octadecanoic acids, which were prepared synthetically; the lactobacillic and dihydrosterculic acids, which were obtained from natural products.[6]

The crystal habit of these acids is similar to that of the normal long-chain fatty acids. Single crystals are difficult to obtain and always appear in thin plates on (001), with the conventional assignment of the long axis as *c*. The so-called "pseudo single crystals"[7] are commonly obtained from a variety of solvents and mixed solvents. In this form of crystal imperfection, multiple thin platy crystals are aligned with one axis in common and the other more or less regularly disordered, like the pages of a book. Even crystals which appeared to be true single crystals under the microscope nearly always showed evidence of this type of imperfection to a more or lesser degree on the X-ray photographs. Conspicuously, the natural products were more difficult to crystallize than the synthetic compounds and gave poorer powder diffraction patterns. The single crystals used in this research were obtained by recrystallization from 5:1 acetone–water mixture at 0°.

The crystallographic data on these acids are given in Table I, together with those of some normal and branched-chain fatty acids of comparable chain length. The values of a, b, α, γ, and c sin β (the long spacing) are measured directly from the X-ray photographs. The selection of the values for β is arbitrary and it is convenient to choose β so that it corresponds to the projection on (010)

(1) Work done jointly in the Sarah Mellon Scaife Radiation Laboratory and the Chemistry Department and supported by a research grant from the Department of Health, Education and Welfare, Public Health Service, National Institutes of Health.

(2) K. Hofmann and R. A. Lucas, THIS JOURNAL, **72**, 4328 (1950).

(3) K. Hofmann, R. A. Lucas and S. M. Sax, *J. Biol. Chem.*, **195**, 473 (1952).

(4) K. Hofmann, O. Jucker, W. R. Miller, A. C. Young, Jr., and P. Tausig, THIS JOURNAL, **76**, 1799 (1954).

(5) K. Hofmann, S. F. Orochena and C. W. Yoho, *ibid.*, **79**, 3608 (1957).

(6) The specimens used in this research were provided by Dr. K. Hofmann, Biochemistry Department, University of Pittsburgh.

(7) A. Muller, *Proc. Roy. Soc.* (*London*), **A114**, 542 (1927).

TABLE I

UNIT CELL DIMENSIONS AND SPACE GROUPS OF SOME C_{18} AND C_{19} ACIDS

Fatty acid	Crystal system	*a*	*b*	Long spacing *c* sin β	Vol. of cell, Å.^{3}	Density	No. of molecules	Space group	Ref.
trans-DL-9,10-Methylene-octadecanoic acid, m.p. 35°	Monoclinic	10.1, Å.	4.78, Å.	41.4, Å 41.0	1999	Calcd. 0.985 Obsd. 0.96	4	$P2_1/a$	8(a) 4
trans-DL-11,12-Methylene-octadecanoic acid, m.p. 37°	Monoclinic	10.1	4.75	41.3[a] 41.0			4	$P2_1/a$	8(a) 4
cis-DL-9,10-Methylene-octadecanoic acid m.p. 39°	Monoclinic	8.93	5.10	43.9	1999	Calcd. 0.99	4		8(c)
cis-DL-11,12-Methylene-octadecanoic acid, m.p. 37°	As for the 9,10-compound above								
Lactobacillic acid, m.p. 29°	Triclinic	5.64 α88.2°	5.19 β89.0°	41.1 γ56.6° 41.0	1005	Calcd. 0.96 Obsd. 0.97	2	P1	8(b) 4
Dihydrosterculic acid, m.p. 40°	Monoclinic	8.90	5.10	43.7	1984	Calcd. 0.997	4	P2 or P2/m	8(a)
α-Stearic acid, m.p. 69.6°, $C_{18}H_{36}O_2$				39.75					9
β-Stearic acid $C_{18}H_{36}O_2$	Monoclinic	5.546	7.381	43.76			4	P2/a or $P2_1/a$	7
n. $C_{19}H_{38}O_2$ α-form, m.p. 69.5°				44.50					8.5
n. $C_{19}H_{38}O_2$, β-form				43.15					9
Tuberculostearic acid, $C_{19}H_{38}O_2$				37.0 (D(−) or L(+) m.p. 12.5°)					10
10-Methyl-octadecanoic acid				38.5 (DL from solvent, m.p. 21.5°)					
				c, Å.					
14-Ethylhexadecanoic acid	Triclinic	5.58 α = 93°15′	6.79 β = 111°30′	27.96 γ = 100°14′	960	Calcd. 0.982 Obsd. 0.989	2	$P\bar{1}$	11
16-Methyloctadecanoic acid, m.p. 50.5°	Triclinic	5.43 α = 91°16′	6.91 β = 94°16′	28.00 γ = 104°56′	1011	Calcd. 0.979 Obsd. 0.988	2	$P\bar{1}$	11
17-Methyloctadecanoic acid, m.p. 67.5°	Triclinic	5.64 α = 90°24′	9.66 β = 91°38′	37.10 γ = 102°33′	1973	Calcd. 1.004 Obsd. 0.995	4	$P\bar{1}$	11

[a] Not significantly different from 41.4 above.

of the angle of tilt of the molecule. The unit cell as defined by *a*, *b*, *c* and α, β, γ will then circumscribe *N* whole molecules. When the length of the molecule and the mode of packing can be predicted, as in stearic acid, the angle of tilt can be calculated from the long spacing.

The unit cell dimensions of the 9,10- and 11,12-*cis* acids and of the 9,10- and 11,12-*trans*-acids, respectively, are identical within the limits of the experimental measurements. The diffraction spectra on oscillation, Weissenberg and precession photographs can be superimposed exactly with respect to position but differed in intensity distribution. It was not possible to make precise measurements of the lattice dimensions, but it is estimated that the difference in cell dimensions between the two *cis* or two *trans* acids is less than 1%.

The structural isomorphism[12] of these pairs of compounds is a consequence of the similarity in the shape of the molecules. This is illustrated in Fig. 1, which represents one of a number of possible ways of packing the molecules in a crystal lattice, which could give rise to this crystal structural relationship between the two *trans* isomers. The particular arrangement shown in this figure is, in fact, based on a detailed structure analysis of the 9,10-*trans* compound, which will be described elsewhere.

But a study by means of models of the effect of the ring position on the over-all shape of the molecules makes it clear, in retrospect, that this isomorphism is to be expected. It is a characteristic of the molecular structures, rather than a peculiarity of a special mode of molecular packing in the crystal structures. This is borne out by the occurrence of the same relationship between the two *cis* isomers, where again a shift of the ring to the next "odd/even" position makes little difference to the over-all shape of the molecule. It seems likely that in both the *cis* and the *trans* series, the whole range of cyclopropane acids with the rings in an "odd/even" position from 5–6 to 13–14 will belong to these isomorphous series.

The Structure of the Lactobacillic and Dihydrosterculic Acids.—The chemical degradation studies of Hofmann and Marco[13] have recently provided evidence that the cyclopropane rings in lactobacillic and dihydrosterculic acids are in the 11,12- and 9,10-positions, respectively, but do not determine the *cis/trans* stereochemistry. The non-identity crystallographically of lactobacillic acid with either of the synthetic *cis*- or *trans*-11,12 acids does not conflict with the 11,12-configuration for the natural acid. The difference in crystal structure (and melting point) can correspond to the difference between the synthetic racemate crystals and the natural enantiomorphic form. In tuberculostearic acid,[9] for example, the difference in long spacing is 1.5 Å. and in melting point is 10°. These differences are comparable with those between lactobacillic acid and the *cis* and *trans* synthetic compounds (see Table I).

(8) This work; source of compound: (a) K. Hofmann, ref. 4; (b) K. Hofmann, ref. 2. (c) K. Hofmann, ref. 5.

(9) F. Francis and S. H. Piper, THIS JOURNAL, **61**, 577 (1939).

(10) S. Stallberg-Stenhaven, *Arkiv Kemi, Mineral. Geol.*, **23A**, 1 (1946).

(11) G. L. Clark and C. Chu, *Acta Cryst.*, **4**, 470 (1951).

(12) the term "isomorphous" is here applied to compounds, the crystal lattices of which are nearly identical due to a closely related arrangement of geometrically similar structural units; *cf.*, A. F. Wells "Structural Inorganic Chemistry," O.U.P., 1945.

(13) K. Hofmann and G. J. Marco, *Federation Proc.*, **15**, 308 (1956).

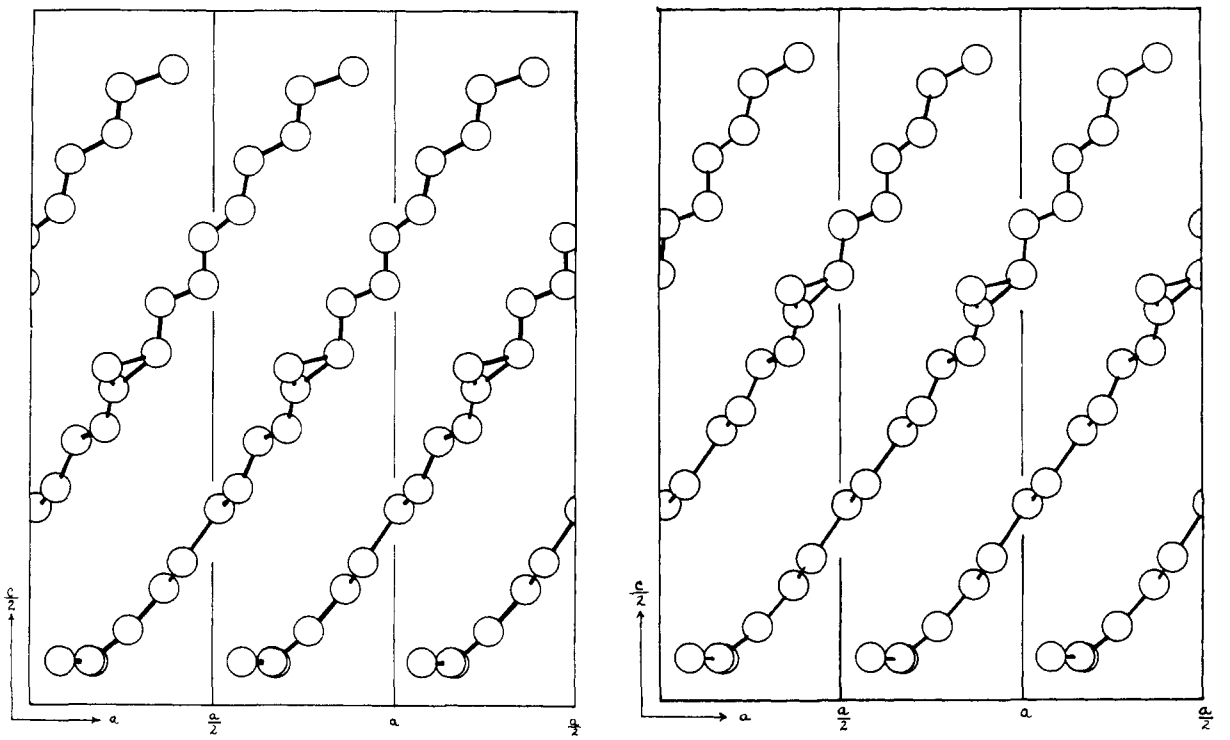

Fig. 1.—Diagram showing the "isomorphous" relationship of the crystal structure of the *trans* 9,10- and 11,12-methylene-octadecanoic acids.

The proof of the structure of lactobacillic acid must come then from either the difficult resolution of the synthetic racemate to give an enantiomorphic crystalline compound identical with the natural product, or the stereochemical identification by means of a detailed electron density distribution map from a single crystal X-ray analysis, which is currently in progress in our laboratory. However, some indirect evidence favoring the *cis* configuration for the lactobacillic acid can be obtained from a comparison of lateral packing of the long chain molecules, as inferred from the unit cell dimensions. In the normal long chain fatty acids, the cell dimensions normal to the chain axis, b, and $a \sin \beta$ are commonly in the ratio of $\sqrt{2}$ to 1; *e.g.*, lauric acid 1.41; palmitic acid, 1.44; stearic acid, 1.49 (with b and $a \sin \beta$ reversed). This is associated with the packing of molecules of elliptical cross-section with major and minor axes in the ratio of 5:4 (based on a methylene van der Waals radius of 2.0 A., C–C = 1.54 Å. and the tetrahedral valence angles). The synthetic *trans*-cyclopropane acids have a cross-sectional $b/a \sin \beta$ ratio of close to $\sqrt{3}$:1, taking $\beta \backsimeq 55°$ from a comparison of the observed value of $c \sin \beta$ with the theoretical length of the molecule. This corresponds to a hexagonal close packing of chain molecules with average circular cross-section, which we believe to be due to the particularly compact structure of the *trans* substituted ring in the chain, and its influence on the relative orientation of the two halves of the molecule (Fig. 1). The synthetic *cis* acids have an elliptical cross-section, as inferred from $b/a \sin \beta = 1.41$, and, if for more direct comparison, the lactobacillic acid dimensions are transformed to a non-primitive cell with nearly orthogonal axes of $a' = 6.80$, $b' = 5.52$, $\gamma' = 91.5°$, the cross-section ratio is 1.21. This corresponds to the packing of molecules with more elliptical cross-section, and is such as might be expected from the packing of the molecules with all left or all right hand *cis* substituted cyclopropane rings.

The dihydrosterculic acid and the *cis*-DL-9,10-methylene-octadecanoic acid are identical compounds as shown conclusively by the single crystal and powder X-ray diffraction data given in Tables I and II. The powder patterns of the synthetic compound and the dihydrosterculic acid, which was prepared by hydrogenation of the naturally occurring sterculic acid, differ only in the degree of crystallinity, remarked upon also in connection with the problem of obtaining single crystals. It is a consequence of this difference that the (002) inner ring of the synthetic compound could not be observed on the photographs of the natural product, which were generally more diffuse. The powder photographs also show quite clearly the structural isomerism between the 9,10 and 11,12 compounds. The strong side spacings, which depend mainly upon the general packing of the long chain molecules, are identical in spacing within the precision of the measurements, and differ only slightly in intensity relationship. The orders of the long spacing, the (00l)s, and some of the outermost lines on the pattern, although also identical in spacing, differ more in their intensity relationship, since they are more sensitive to the detailed arrangement of the carbon atoms along the length of the molecules.

TABLE II

X-RAY POWDER DATA FOR THE SYNTHETIC *cis*-DL-9,10- AND 11,12-METHYLENE-OCTADECANOIC ACIDS, DIHYDROSTERCULIC AND LACTOBACILLIC ACIDS

	d Spacings in Å.					
	Synthetic *cis*-9,10-DL-acid d_{hkl}	Synthetic *cis*-11,12-DL acid d_{hkl}	Dihydrosterculic acid d_{hkl}	(*hkl*)	Lactobacillic acid d_{hkl}	(*hkl*)
	22.0, m.	21.0, m.	.....	(002)		
	.....	14.5, vs.	.....	(003)	13.6, s.	(003)
	8.67, ms.		8.40, ms.	(005)	8.5, vw.	(005)
	7.34, m.		7.34, m.	(006)		
	6.10, w.	6.15, w.		(007)	5.76, vw.	(007)
	5.08, w.				5.14, vw.	(008)
	4.61, vw.				4.61, vs.	
	4.34, vs.	4.39, s.	4.33, s.		4.42, s.	Strong side
	4.05, m.	4.08, ms.	4.10, m.	Strong side	4.38, m.	spacings
	3.87, vs.	3.87, m.	3.89, s.	spacings	3.75, m.	
	3.71, m.	3.71, m.	3.70, m.		3.55, m.	
	3.38, w.	3.40, w.	3.41, w.		3.37, w.	
	3.23, w.		3.26, w.		3.20, vw.	
	3.09, vw.				3.02, vw.	
	2.73, vw.				2.87, vw.	
	2.62, vw.				2.70, vw.	
	2.53, m.	2.56, m.	2.52, m.		2.59, w.	
	2.34, m.		2.33, m.		2.20, w.	
	2.17, m.	2.17, m.	2.17, m.			
	2.09, w.	2.04, w.				
	1.99, w.					
Long spacing (mean value from (00*l*)s), Å.	43.4	43.3	43.7		41.0	

Polymorphism in the Cyclopropane Fatty Acids.—It is known that the long-chain monocarboxylic fatty acids exhibit trimorphism in both the odd and even series. This is a factor which might invalidate inferences drawn from comparison of cell dimensions and packing shapes as in part of the preceding discussion, and careful observations were made for evidence of polymorphism in these cyclopropane acids. In our efforts to obtain crystals suitable for X-ray intensity measurements, a variety of solvents and conditions was used. In no cases did we get any evidence of other than the one crystal form of the acids. In addition, the acids were melted in capillaries and solidified to give powder photographs which were identified as being from the same structures as the corresponding single crystal patterns. Apparently the presence of the cyclopropane ring restricts the number of "economical" modes of packing the long chains to that of a single form, in contrast to the normal straight-chain acids.

The Crystallography of the C_{19} Cyclopropane Acid Amides.—The synthetic *cis*- and *trans*-9,10-DL-, *cis*- and *trans*-11,12-DL-methylene-octadecanoic acid amides, lactobacillic acid amide and dihydrosterculic acid amide were examined as powder specimens since it was not possible to obtain crystals suitable for single crystal X-ray study. The powder diffraction data are shown in Table III. The synthetic compounds gave strikingly sharper diffraction patterns than the derivatives of the natural products.

As in the synthetic acids, the *trans*-9,10- and 11,12-acid amides are structurally isomorphous. The d-spacings agree within the errors of measurement of the X-ray photographs. The first six lines on the photograph of the 9–10 compound could be indexed consistently as (00*l*) spacings to give a value for the long spacing of 39.12 Å. The side spacings for the two compounds were very similar in intensity distribution, and it was only by reason of the detailed intensity distribution of the (00*l*)s and the lines beyond the prominent side-spacings that the photographs were distinguishable. This well illustrates the very close similarity of the two crystal structures, and in poorly crystalline material it could easily occur that the patterns were indistinguishable.

In the *cis* series of amides, in contrast to the acids, the comparison of the powder data was complicated by dimorphism. The original specimen of dihydrosterculic acid amide (form B in Table III), which had been crystallized from acetone at −50°,[4] was not identical with the synthetic 9,10-compound, as was the case for the parent acids. Recrystallization of the natural product amide from methanol at 0° gave, however, a second form, A, the powder pattern of which matched that of the synthetic product (Table III). On melting and solidifying, form B was converted to A.

Two forms of the *cis*-11,12-DL-amide were also obtained, one from methanol at 0° and the other from the melt. Neither of these corresponded with the powder data from the lactobacillic acid amide, in agreement with the conclusions derived from the study of the acids. In general, these amides gave rather poorly crystalline material from the melt and some samples on cooling gave a completely or partly amorphous transparent solid.

Acknowledgments.—The authors are grateful to Dr. K. Hofmann for suggesting this research, for providing specimens and for many stimulating

TABLE III

X-RAY POWDER DATA FOR SOME SYNTHETIC AND NATURAL C_{19} CYCLOPROPANE FATTY ACID AMIDES

	Synthetic *trans*-9,10 m.p. 87° d_{hkl}	Synthetic *trans*-11,12 m.p. 84° d_{hkl}	*hkl*	*Lactobacillic* m.p. 81° d_{hkl}	*hkl*	Dihydrosterculic m.p. 87° (form B) d_{hkl}	*hkl*	Dihydrosterculic m.p. 87° (form A) d_{hkl}	*hkl*	Synthetic *cis*-9,10 m.p. 87° d_{hkl}	*hkl*	Synthetic *cis*-11,12 (form B) m.p. 87° d_{hkl}	*hkl*	Synthetic *cis*-11,12 (form A) m.p. 87° d_{hkl}	*hkl*
	13.1m	12.9s	003	12.5m	003	20.1vw	002	12.8ms	003	12.6ms	003	13.3ms	003	12.6s	003
	9.75vw		004	9.19vvw	004	12.9m	003	9.3vvw	004	9.3vvw	004			7.6vvw	005
	7.83m	7.75vw	005	7.44vw	005	9.65w	004	7.52m	005	7.52m	005	7.8vvw	005		
	6.51vw		006			7.82w	005	6.27w	006	6.32w	006	5.60vw	007	5.5vvw	007
	5.54vvw	5.59w	007	5.42w	007	6.48vw	006	4.85vs		4.84vs		4.86w		4.92vs	
	4.93m	4.88w	(008)	4.89vs		4.91ms	(008)	4.38vs		4.38vs		4.52vvs		4.56vvs	
	4.48vs	4.50vs		4.28s	Side	4.48vvs	Side	4.23w		4.20w	Side	4.40vw	Side	4.18vs	Side
	4.19vs	4.18vs	Side	3.81m	spac-	4.17ms	spac-	3.95m		3.95m	spac-	4.18ms	spac-	3.95s	spac-
	3.98s	4.00s	spac-	3.55ms	ings	3.77m	ings	3.70m		3.70m	ings	3.98ms	ings	3.70m	ings
	3.76s	3.78s	ings					3.45m		3.45m		3.72m		3.56w	
	3.56m									3.28vw		3.22w		3.28w	
	3.26vw	3.23m		3.33m				2.71m		2.71m		2.60w		3.05vw	
	3.06m			3.12w										2.71w	
	2.84m	2.81vw		2.91w										2.71w	
	2.57m	2.58m		2.77mw		2.63m								2.57w	
	2.44vw			2.44mw											
	2.37m	2.38m													
	2.26vw			2.26w		2.29				2.35vw					
	2.22vw	2.22m		2.20w		2.18		2.21m		2.21w		2.19w			
	2.15m	2.18m													
	2.07vw														
	1.95m	1.93m		1.85w											
	1.82w			1.79w											
Long spacing $c \sin \beta$	39.12	39.12		37.5		39.13		37.7		37.6		39.3		38.0	This work
	39.6	39.7		36.3											Ref. (4)

discussions. They wish to thank Mr. K. Momoki for taking some of the powder and single crystal photographs. We are grateful to the Research Corporation for funds with which we obtained the precession camera used in this research.

PITTSBURGH, PA.

[CONTRIBUTION FROM THE RESEARCH AND DEVELOPMENT DEPARTMENT, THE PROCTER & GAMBLE COMPANY, MIAMI VALLEY LABORATORIES]

Acetin Fats. III. The Binary Systems ASA–SAA and APA–PAA, Mixed Symmetrical and Unsymmetrical Stearoyl Diacetins and Palmitoyl Diacetins

BY E. S. LUTTON

RECEIVED APRIL 18, 1957

ASA (2-stearoyldiacetin) and APA (2-palmitoyldiacetin) occur in metastable subalpha and alpha forms and stable beta prime-like forms (alpha being questionable for the palmitic compound). Mixtures of the symmetrical and unsymmetrical isomers in the binary systems ASA–SAA and APA–PAA show greatly increased alpha stability over that of the pure compounds. Melting points of mixtures in the alpha form lie in a straight line joining those of the components. In the composition range 30–90% of unsymmetrical isomer, the alpha phase shows no transformation in 2 months, hence a high degree of stability is implied for this phase of a binary mix of saturated diacetins prepared via random interesterification (2/3 unsymmetrical.)

Waxy forms of acetylated monoglycerides, specifically of 1-stearoyldiacetin (SAA) and 1-palmitoyldiacetin (PAA) have been of interest for the high degree of stability of these metastable forms.[1–6] In practical methods of preparation, mixtures of symmetrical and unsymmetrical compounds may occur and some comparison of mixtures with individual isomers has been made.[5] Pure ASA and APA previously have not been available, however.

Experimental

ASA and APA were made by acetylation, respectively, of 2-monostearin and 2-monopalmitin, prepared as previously reported.[7] SAA and PAA were prepared by familiar methods.[3]

Approximately 0.2 g. mixes (20, 40, 50, 60 and 80% symmetrical) of the symmetrical and unsymmetrical isomers were weighed up and carefully stirred in both solid and liquid states. Capillary samples were prepared and m.p.'s determined as previously described[8] after melting, chilling and appropriately storing. The so-called rapid complete m.p., normally referring to alpha, was determined on fresh samples. The regular complete m.p. was performed on samples stored 2 months below the minimum alpha m.p. of the binary system, *i.e.*, at 28° for ASA–SAA and 15° for APA–PAA. The results appear in Fig. 1.

X-Ray diffraction data were obtained on metastable (subalpha and alpha, if observable) and stable forms of ASA and APA and on the apparently stable subalpha and alpha forms of 50% mixes of ASA and SAA and of APA and PAA. Flat-film diffraction patterns were obtained with a General Electric XRD-1 unit employing nickel-filtered Cu K_α radiation and a 0.025 in. pinhole system. Sample-to-film distance was either 5 or 10 cm. Low-temperature phases were kept in a small cold-block during exposure, subalpha at 0° except for APA at −20°; alpha at 15° for palmitoyl compositions and 25° for stearoyl compositions.

Thermal and X-ray diffraction data are listed in the following order:

Composition.—Polymorphic state: m.p. °C.; long spacing, L.S. (in Å.); short spacing, S.S. (in Å.). Relative intensities of diffraction lines are indicated by (VS) = very strong, (S) = strong, (M) = medium and (W) = weak.

ASA. Subalpha: no m.p.; L.S. 36.7; S.S. 4.20(S), 3.86(M). Alpha: 29.0°; L.S. 36.0; S.S. 4.16(S); stable: 37.0°; L.S. 30.0; S.S. 4.71(M), 4.21(S), 4.06(S), 3.86(M).

APA. Subalpha: no m.p. (?); L.S. 34.0; S.S. 4.20(S), 3.82(M). Alpha: 15.7° (presumably alpha); L.S. —; S.S.—; stable: 37.0°; L.S. 27.5; S.S. 4.68(M−), 4.42(M−), 4.20(S), 4.04(S+), 3.85(M).

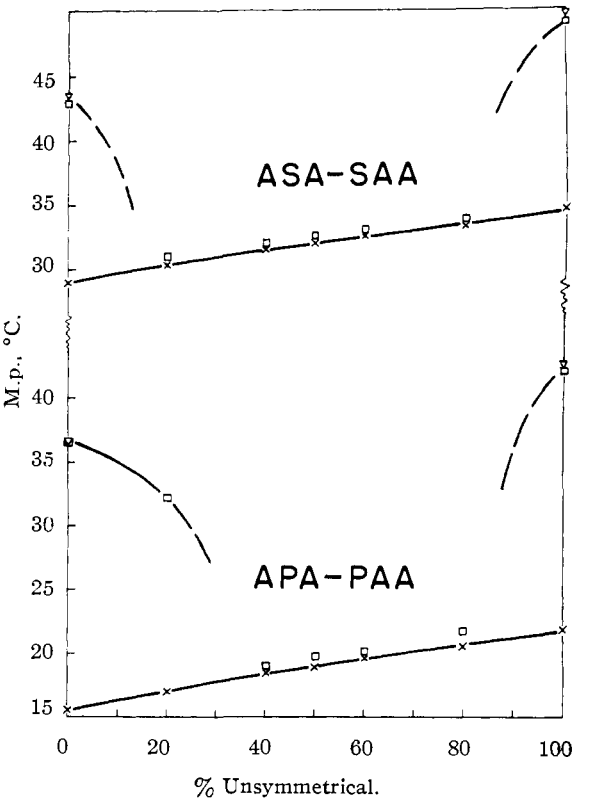

Fig. 1.—M.p.'s of binary mixtures of ASA–SAA and APA–PAA: ×, rapid complete m.p.; □, regular complete m.p. (ASA–SAA stored 2 mon., 28°; APA–PAA stored 2 mon., 15°); ▽, solvent crystals, m.p.

(1) F. L. Jackson (to The Procter & Gamble Co.), U. S. Patent 2,615,159 (1952).

(2) F. J. Baur (to The Procter & Gamble Co.), U. S. Patent 2,615,160 (1952).

(3) F. L. Jackson and E. S. Lutton, THIS JOURNAL, **74**, 4827 (1952).

(4) R. O. Feuge, E. J. Vicknair and N. V. Lovegren, *J. Am. Oil Chem. Soc.*, **30**, 283 (1953).

(5) F. J. Baur, *ibid.*, **31**, 196 (1954).

(6) R. O. Feuge, E. J. Vicknair and K. S. Markley (to the Secretary of Agriculture), U. S. Patent 2,745,749 (1956).

(7) J. B. Martin, THIS JOURNAL, **75**, 5482 (1953).

(8) E. S. Lutton, F. L. Jackson and O. T. Quimby, *ibid.*, **70**, 2441 (1948).