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## Compatibility of Derivatives of 9,10-Dihydroxystearic Acid and 9,10-Dihydroxyoctadecanol With Some Commercial Polymers<sup>1</sup>

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URING the course of our program on the epoxidation and hydroxylation of unsaturated compounds derivable from fats (2, 4, 5, 7) we had occasion also to prepare a large number of esters, ether-esters, and hydroxy-ethers of 9,10-dihydroxystearic acid and 9,10-dihydroxyoctadecanol (3, 6, 8, 9). Since these compounds had low vapor pressures, were insoluble in water, and had a relatively high oxygen content, their possible utilization as plasticizers was considered. This paper briefly describes the results of compatibility tests conducted intermittently during the past five years on many of these compounds with some commercial resins. In one case semiquantitative data are also reported.

The compounds studied were the alkyl (8) and alkenyl (9) esters of low-melting 9,10-dihydroxystearic acid, several 9,10(10,9)-alkoxyhydroxyoctadecanols (6), several esters of 9,10(10,9)-alkoxyhydroxystearic acids (6), and two series of previously unreported compounds, namely, esters of the isomeric 9,10-dihydroxystearic acids with ether-alcohols and polymeric plasticizers prepared by the reaction of selected members of this group of new esters with phthalic anhydride. Commercial polymers employed were ethyl cellulose, cellulose acetate, polyvinyl chloride-acetate copolymers (95:5 and 87:13), polyvinyl butyral (low and high viscosity), and in a few cases, polyvinyl acetate, polystyrene, and a vinylidene chloride-acrylonitrile copolymer.

Experimental

Materials Tested. The preparation of the alkyl (8) and alkenyl (9) esters of low-melting 9,10-dihydroxystearic acid, the 9,10(10,9)-alkoxyhydroxyoctadecanols (6), and the esters of the 9,10(10,9)-alkoxyhydroxystearic acids (6) has already been reported.

A typical preparation of an ester of 9.10-dihydroxystearic acid with an ether-alcohol is described. A mixture of 15.8 g. (0.05 mole) of 9,10-dihydroxy-stearic acid, m.p. 95°, 7.6 g. of ethylene glycol monomethyl ether (0.10 mole), 0.79 g. of naphthalene-2-

<sup>1</sup>Presented at the meeting of the American Oil Chemists' Society, Atlanta, Ga., May 1950.

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sulfonic acid, and 200 ml. of toluene was refluxed for eight hours. The water formed during the reaction was removed azeotropically with the toluene, and the toluene was automatically returned to the reaction mixture. The reaction mixture was evaporated to dryness and the residue was washed three times with hot water. The washed, dried residue, consisting essentially of 2-methoxyethyl 9,10-dihydroxystearate, was obtained in quantitative yield and melted at about 45°. Recrystallization from acetone yielded substantially pure product, m.p. 52.3-53.0°, and saponification number, 153 (calcd. 150). Table I lists the esters prepared by the method just described as well as some of their characteristics. All the recrystallized products were white solids whereas some of the crude reaction products were liquids.

Ethylene glycol monomethyl ether, b.p. 123-4°, ethylene glycol monoethyl ether, b.p. 134°, ethylene glycol monobutyl ether, b.p. 170°, ethylene glycol mono(2-ethylbutyl) ether, b.p. 197°, ethylene glycol monophenyl ether, b.p. 170° at 100 mm., ethylene glycol monobenzyl ether, b.p. 180° at 98.5 mm., and diethylene glycol monobutyl ether, b.p. 138° at 50 mm., were obtained by multiple fractional distillation of the purest commercial grades through efficient columns (helix packed or heligrid). Diethylene glycol monomethyl ether, b.p. 192° and diethylene glycol monoethyl ether, b.p. 200-1°, were obtained from the purest commercial grades by washing a benzene solution of the ether-alcohol (250 g. per 750 ml. of solvent) with small quantities of water (ca. 5 ml.) to remove water-soluble materials (10 washes), and fractionally distilling the dried benzene solution.

The polymeric plasticizers were prepared by the reaction of equimolar quantities of methyl 9,10-dihydroxystearate, m.p. 70°, 2-butyoxyethyl-9,10-dihydroxystearate, m.p. 50°, 2-benzyloxyethyl-9,10-dihydroxystearate, m.p. 45°, and 2-(2'-methoxyethoxy)ethyl-9,10-dihydroxystearate, m.p. 41°, respectively, with phthalic anhydride for one hour at 215-220° and then at 235-240° until the reaction product showed signs of threading. The polymeric plasticizers were pale-yellow to amber colored liquids, insoluble in water.

# TABLE I Esters of the 9,10-Dihydroxystearic Acids With Ether-Alcohols

$$CH_{3}-(CH_{2})_{7}-CH-CH-(CH_{2})_{7}-C-O-R$$
 $OH OH$ 

Number		Crude Esters		Recrystallized Esters			
	R	Sapon. No. Found	М. Р. °С.	Sapon, No.		M, P.	
				Calcd.	Found	°C.	
1	-CH <sub>2</sub> -CH <sub>2</sub> -OCH <sub>3</sub> <sup>1</sup>	164	45-7	150	153	52,3-53,0	
2	$-CH_2-CH_2-OCH_3^2$	160	80-5	150	152	89	
3	$-CH_2-CH_2-OC_2H_5^{-1}$	1		144	153	48	
4	$-CH_2-CH_2-OC_2H_5^2$	1		144	149	-86	
5	-CH2-CH2-OC4H21			135	136	50	
3	$-CH_2-CH_2-OC_4H_9^2$			135	138	82	
7	$-CH_2-CH_2-CC_6H_5^{-1}$	1		129	130	i 78	
3	$-CH_{2}-CH_{2}-CC_{6}H_{5}^{2}$			129	129	100	
9	$-CH_2-CH_2-OCH_2C_6H_5^1$	1		125	140	45	
0	$-CH_2-CH_3-OCH_2C_6H_5^2$			125	135	80	
1	$-CH_2-CH_2-OC_4H_8(C_2H_5)^{\frac{1}{2}}$			126	134	30	
2	$-CH_2-CH_2-OC_4H_8(C_2H_5)^2$	i		126	131	80	
3	$-(CH_2-CH_2-O)_2-CH_3^1$	<b>.</b>		134	139	41	
L	$-(CH_2-CH_2-O)_2-CH_3^2$	1		134	138	82	
5	$-(CH_2-CH_2-O)_2-C_2H_5^1$	· · · · · ·		130	135	38	
3	$-(CH_2-CH_2-O)_2-C_2H_5^2$			130	135	80	
***************************************	$-(CH_2-CH_2-O)_2-C_4H_9^1$			122	137	41	
3	-(CH-CH2-O)2-C4H2	1		122	132	79	

Prepared from low-melting 9,10-dihydroxystearic acid, m.p. 95°, and the ether-alcohols.
 Prepared from high-melting 9,10-dihydroxystearic acid, m.p. 130°, and the ether-alcohols.

The stock solutions of commercial polymers had the compositions tabulated below:

and compressions tabletation boldy.	
Ethyl Cellulose Stock Solution Ethyl cellulose	16 g.
Xylene: butanol (80:20 by volume)	200 ml.
Cellulose Acetate Stock Solution	a= a=
Cellulose acetate	
n-Butanol	
Ethanol (95%)	76.5 ml.
Toluene	140 ml.
Polyvinyl Chloride: Acetate Copolymer (95:5) Stock Solution	
CopolymerButyl acetate: tetrahydropyran (80:20	16 g.
by volume)	250 ml.
Polyvinyl Chloride: Acetate Copolymer (87:13) Stock Solution	
Copolymer	16-20 g.
Dioxane or ethylene dichloride: ethanol (90:10 by volume)	200 ml.
Polyvinyl Butyral Stock Solution	
Polyvinyl butyral (high or low viscosity)	16 ~
Toluene: ethanol (80:20 by volume)	200 ml.
70.7 . 7	
Polyvinyl Acetate Stock Solution	
Polyvinyl acetate	
Xylene: n-butanol (80:20 by volume)	200 ml.
Polystyrene Stock Solution	
Polystyrene	16 g.
Xylene: n-butanol (80:20 by volume)	200 ml.
Vinylidene Chloride-Acrylonitrile Copolymer Stock Solution	
Copolymer	16 g.
Methyl ethyl ketone	400 ml.
<u></u>	

In each case the material, the compatibility of which was being tested, was dissolved in about 20 ml. of polymer stock solution, and the resulting solution was spread on a glass plate (6" x 8" x 1/4") by means of a doctor blade. The thickness of the wet film was adjusted to give a dry film about 0.003-0.004" thick. The solvent was allowed to evaporate slowly (several days) in specially constructed boxes which held the plates perfectly flat. The film was removed from the plate, usually by lifting one corner with a razor blade and then soaking the film and plate for a short time in cold water until the entire film was detached. The films were then allowed to hang in the air at room temperature for several days to ensure complete removal of solvent and water. The films were then examined for compatibility of plasticizers. Incompatibility was detected either by hazing of the film, exudation of liquid, or actual crystallization of the material being tested. Control films containing no plasticizer were prepared in all cases. The compatibility tests are summarized in Table II.

The most promising materials were methyl 9,10-(10,9)-methoxyhydroxystearate, compounds 5, 6, 9, and 10 of Table I, and the four polymeric plasticizers listed near the end of Table II. In addition to determining the compatibility of methyl 9,10(10,9)-methoxyhydroxystearate it was milled with polyvinyl chloride-acetate copolymer (95:5) by a standard technique using the recipe below:

Copolymer	63.5 g.
Basic lead carbonate	1.0
Stearic acid	0.5
Plasticizer	35.0

The test data were as follows:

Plasticizer	Milling Molded characteristics appearance		Compatibility	Elonga- tion, %	Modulus at 100% elongation, psi	Tensile strength, psi
Methyl 9,10 (10,9)-methoxy- hydroxystearate	Milled very well, fumed slightly	White, opaque sheet	Borderline; slight exudation at 23°C.	280	1490	2690
Di-(2-ethylhexyl)phthalate	-(2-ethylhexyi)phthalate Milled very well, fumed White, opaque sheet moderately		O.K.	O.K. 270		3140

These characteristics compare favorably with a control stock plasticized with di-(2-ethylhexyl)phthalate. a well-known commercial plasticizer. Methyl 9,10-(10,9)-methoxyhydroxystearate was also milled with cellulose acetate and was found to be superior to butyl stearate, the control plasticizer, at a concentration of 15%. The flow temperature (1) with the former compound was <103°C., whereas with butyl stearate it was 112°C. Furthermore milling temperatures were about 40°C. lower with methyl 9,10(10,9)methoxyhydroxystearate than with butyl stearate. The polymeric plasticizers were not studied further because of the inability to remove phthalic anhydride completely even though the products were washed and heated under vacuum above the sublimation temperature of phthalic anhydride. These materials are of interest however because they are compatible with polymers of such widely different structure.

No attempt has been made to study mixtures of commercial plasticizers with any of the compounds reported in this paper because of the large number of possible combinations. Such a study may prove especially fruitful in the case of borderline plasticizers.

#### Summary

A study of compatibility with some commercial polymers is reported for some alkyl and alkenyl esters of low-melting 9,10-dihydroxystearic acid, 9,10(10,9)alkoxyhydroxyoctadecanols, esters of 9,10(10,9)-alkoxyhydroxystearic acids, and two series of previously unreported compounds, namely, esters of the isomeric 9,10-dihydroxystearic acids with ether-alcohols and polymeric plasticizers prepared by the reaction of selected members of this group of new esters with phthalic anhydride.

The most promising materials are methyl 9,10-(10,9)-methoxyhydroxystearate, esters of 9,10-dihydroxystearie acid with ethylene glycol monobutyl ether and ethylene glycol monobenzyl ether, and the polymeric plasticizers. The last-named group is compatible with polymers which differ widely in chemical structure.

### Acknowledgment

The authors wish to thank T. J. Dietz for the test data on methyl 9,10(10,9)-methoxyhydroxystearate.

TABLE II Compatibility With Commercial Polymers 1

Material Tested	% by Weight of Dry Film	Compatible	Incompatible	Borderline
Methyl 9,10-dihydroxystearate (L.M.)2.	5	1	2	
Methyl 9,10-dihydroxystearate (L. M.)	15			1
Methyl 9.10-dihydroxystearate (I, M)	. 20	1	1, 3, 4, 5, 6, 9	
Butyl 9,10-dihydroxystearate (L.M.)	15	1		
Butyl 9,10-dihydroxystearate (L.M.)	20	(	1, 3, 4, 5, 6, 9	
Octyl 9,10-dihydroxystearate (L.M.)	5		2	1
Octadecyl 9,10-dihydroxystearate (L.M.)	5 15	1	1	
Octadecyl 9,10-dihydroxystearate (L.M.) Allyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	15 5	1.0	i .	
Allyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	15	1, 2 5, <b>6</b>		1
Allyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	20	5, 0	1, 2, 3, 4, 9	5, 6
Methallyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	20 5		1, 2, 5, 4, 5	3, 0
Methallyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	15	1		-
Methallyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	20	-	1, 3, 4, 5, 6, 9	
9. Chloroally 18 0. 10. diby drovy stoone to (T. M.)	~		l, -, -, -, -, -, -	2
2-Chloroallyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	15	1	-	
2. Chiorogatyr 3,10-dinydroxystearate (11,M.)	20	5, 6	1, 3, 4, 9	
Oleyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	5	1	2	
Oleyl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	15			1
Furfuryl <sup>3</sup> 9,10-dihydroxystearate (L.M.)	5	2		
Furfuryl <sup>3</sup> 9,10-dihydroxystearate (L.M.)		1		5, 6
Furfuryl <sup>3</sup> 9,10-dihydroxystearate (L.M.)		_	2, 3, 4, 5, 6, 9	1
9,10(10,9)-Methoxyhydroxyoctadecanol.	. 5	2		
9,10(10,9)-Methoxyhydroxyoctadecanol		)		2
9,10(10,9)-Ethoxyhydroxyoctadecanol	.8	2	2	
9,10(10,9)-Ethoxyhydroxyoctadecanol	20	2	) z	1
9,10(10,9)-Propoxyhydroxyoctadecanol.	5	. 2		2
9,10(10,9)-Propoxyhydroxyoctadecanol				4
9,10(10,9)-Butoxyhydroxyoctadecanol.	. <b>5</b>	2		9
9,10(10,9)-Alloxyhydroxyoctadecanol <sup>3</sup> .	5	9		4
9,10(10,9)-Alloxyhydroxyoctadecanol <sup>3</sup>	20	_		
Methyl 9,10 (10,9)-Methoxyhydroxystearate	15	1		2
Methyl 9,10 (10,9)-Methoxyhydroxystearate	35	4	•	3
Ethyl 9,10 (10,9) Ethoxyhydroxystearate.	5	$2, \tilde{4}$		_
Ethyl 9.10(10.9)-Ethoxyhydroxystearate.	10	_, _		2.4
Allyl 9,10 (10,9)-Alloxyhydroxystearate <sup>3</sup>	5	1, 2		1 '
Allyl 9.10(10.9)-Alloxyhydroxystearate <sup>3</sup>	20	,	2	1
Compounds 1 and 2, Table I	20		2	1
Compounds 3 and 4, Table I	20		2	1
Compounds 5 and 6, Table I		1, 3, 5, 6	2, 4, 7, 8, 9	
Compounds 7 and 8, Table I	20		2, 4, 7, 8, 9	}
Compounds 9 and 10, Table I	20	1, 3, 5, 6	2, 4, 7, 8, 9	5.0
Compounds 9 and 10, Table I	30		9	5, 6
Compounds 11 and 12, Table I	20	l	$\frac{z}{2}$	1 1
Compounds 15 and 16, Table I	$\frac{20}{20}$		2	1 1
Compounds 17 and 18, Table I	$\frac{20}{20}$		9	1 1
Polymeric Plasticizer A <sup>4</sup>	20		2	_
Polymeric Plasticizer A <sup>4</sup>	30	3, 5, 6		
Polymeric Plasticizer B <sup>5</sup>	20	0, 0, 1	2	
Polymeric Plasticizer B <sup>5</sup>	30	3, 5, 6	_	
Polymeric Plasticizer C <sup>6</sup>	20	, , , , i	2	İ
Polymeric Plasticizer C <sup>6</sup>	30	3, 5, 6	_	
Polymeric Plasticizer D <sup>7</sup>	20	1	2, 8, 9	1
Polymeric Plasticizer D <sup>7</sup>	30	3, 5, 6		Į.

Commercial polymers are listed in the compatibility columns under the following code: 1 = ethyl cellulose; 2 = cellulose acetate; 3 = polyvinyl chloride-acetate copolymer (87:13); 5 = polyvinylbutyral, ligh viscosity; 7 = polyvinyl acetate; 8 = polystyrene; 9 = vinylidene chloride-acrylonitrile copolymer.

2 LM. means prepared from low melting 9,10-dihydroxystearic acid, m.p. 95°.

3 Films darkened on exposure to air.

4 Prepared from phthalic anhydride and compound 9 of Table I.

6 Prepared from phthalic anhydride and compound 5 of Table I.

7 Prepared from phthalic anhydride and compound 5 of Table I.

<sup>7</sup> Prepared from phthalic anhydride and compound 13 of Table I.

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**ABSTRACTS** Don Whyte, Editor

### Oils and Fats

R. A. Reiners, Abstractor

IMPROVEMENT OF THE CLASSICAL METHOD FOR DETERMINATION OF THE INDUSTRIAL OUTPUT OF OLIVE-OIL BY PRESSURE. P. M. Rousseau. Industr. agr. alim., 67, 52(1950). The pressure-juice is collected in a graduated tube of sufficient capacity. The oil is separated from the liquid in the tube by centrifugation. Measurement of the volume gives the results.

DERIVATIVES OF FATTY ACIDS. IDENTIFICATION BY X-RAY DIF-FRACTION POWDER PATTERNS. F. W. Matthews, G. G. Warren, and J. H. Michell. *Anal. Chem.* 22, 514(1950). Three types of derivatives of fatty acids, silver salts, amides, and anilides, were found suitable as a means of identification of the acid by the use of X-ray diffraction powder patterns. For ease of preparation and pattern differentiation the silver salts are preferred.

HEAT CAPACITY OF STABILIZED PEANUT BUTTER. T. L. Ward, W. S. Singleton, and A. F. Freeman (Southern Reg. Res. Lab.). Food Research 15, 146(1950). An equation is derived for expressing the specific heat of stabilized peanut butter from 20 to  $80^{\circ}$ C. which is  $C_p = 0.361 + 0.0012t$  where  $C_p$  is expressed in calories per g. and t is the temperature in °C.

KINETICS OF THE ESTERIFICATION OF OLEIC ACID BY PRIMARY ALCOHOLS AT ROOM TEMPERATURE. M. Loury and Janine Piquard. Oleagineux 4, 505-9(1949). The extent of esterification of oleic acid with various primary alcohols was measured after reacting for 7 days at 18°C. in the presence of sulfuric acid. (Chem. Abs. 44, 3885.)

FATS AND FATTY OILS AS LUBRICANTS. M. Singer. Seifensieder-Ztg. 73, 133-5, 153-5, 173-5(1947). The properties and uses of a large number of animal and vegetable fats and oils and polymerized oils are discussed. (Chem. Abs. 44, 3723.)

CHROMATOGRAPHIC ANALYSIS OF THE UNSAPONIFIABLE MATTER OF MARINE ANIMAL OILS. L. E. Swain. Canadian Chem. and Process Indust. 32 (6), 553-554(1948). A flowing chromatogram is used to separate the unsaponifiable matter into hydrocarbons (petroleum elution), mono-alcohols (methylene chloride elution), di-alcohols (diethyl ether elution), and an unidentified fraction (methanol elution). (Biol. Abs. Sect. G. 23 [3], 21.)

THE ISOLATION OF A CONJUGATED UNSATURATED ACID FROM THE OIL FROM Ximenia caffra KERNELS. S. P. Lighthelm and H. M. Schwartz. J. Am. Chem. Soc. 72, 1868(1950). The acids from X. caffra oil were fractionated and a C1s acid was isolated which contained a double bond and a triple bond either in conjugation with one another or with one of them in conjugation with the carboxyl group. This acid, for which the name ximenynic acid is proposed, was present in about 25% concentration.

ABSORPTION SPECTRUM OF OLIVE OILS IN THE ULTRAVIOLET ZONE. F. Poggio and Maria M. Retortillo. Bol. Inst. Espanol Oceanogr. 14, 1-11(1949). A study of the absorption spectra of 50 samples of olive oil of different qualities revealed that at the wavelength 255 mm. the maximum extinction coefficient for the pressure oils was 87 and that the minimum extinction coefficient for refined extracted oils was 139. This difference can serve as a guide in distinguishing quality. (Biol. Abs. Sect. G. 24 [1], 20.)

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ducts of all n-monocarboxylic acids from C4 to C12, as well as C14, C16, and C18 were formed. Most of these were precipitated from a methyl alcohol solution of urea. (Chem. Abs. 44, 3900.)

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RAPE SELECTION. P. Larroque. Oleagineux 5, 292-295(1950).

Discusses methods for scientific selection of rape.

STUDIES IN CO-SOLVENCY. PART IV. SOLUBILITY OF STEARATES OF LITHIUM, SODIUM, AND POTASSIUM IN GLYCOLIC MIXTURES. Shreepati Rao and Santi R. Palit. J. Indian Chem. Soc. 26, 577-583(1949). The solubility of stearates of lithium, sodium, and potassium was determined at 35° in binary solvent mixtures where one component is propylene or diethylene glycol and the other is chloroform, ethylene dichloride, or benzene. A few measurements have been recorded using alcohols in place of glycols in the above binary mixtures. The glycolic mixtures improve to a great extent the solvent power of the individual solvents and the results are found to be in agreement with previous observations and the already postulated mechanism.

CLASSIFICATION OF SURFACE-ACTIVE AGENTS BY "HLB." Griffin. J. Soc. Cosmetic Chemists 1, 311-326(1949). The HLB system for the choice of emulsifiers based on their hydrophilelipophile balance is described. The system, though it does not indicate the over-all efficiency of the emulsifier, does tell what kind of an emulsion or product to expect, and so enables us to compare various chemical types of emulsifiers at their optimum balance. Estimated HLB values for various types of emulsifiers and fats and oils are presented as well as a method for their determination.

EXPERIMENTAL FORMULATION OF EMULSIFIED CREAMS. S. Druce. Mfg. Chemist, 21, 159-160(1950). Emulsified cosmetic and dermatological creams, particularly of the o/w type, have become very popular during recent years and many medicaments have been incorporated into vanishing-cream type bases. In some cases it is necessary to formulate a base which will remain stable in an acid medium, and for this purpose a soap emulsifying agent cannot be used. The emulsifying properties of the sulphated alcohols and other emulsifying agents when used as separate entities were investigated.

WORLD PRODUCTION OF FAT SUBSTANCES IN 1949. Paul H. Mensier. Oleagineux 5, 284-286(1950). The outlook for production of fat substances and their bearing on world trade is examined.

SELECTIVE HYDROGENATION. M. Loury. Oleagineux, 5, 279-283 (1950). Selective hydrogenation results from a particular heterogeneous catalysis which can be controlled by the reaction conditions.

CONDITIONS FOR STORING RAW AND RENDERED FAT. S. Liberman and E. Mirkin. Myasnaya Industriya 20 (5), 26-9(1949). The effects of temperature, light, and duration of storage on raw and rendered fats are pointed out with tables and charts showing the development of peroxide and acid values. Optimum storage conditions of 3 to 5°C. and relative humidity of