

Chemistry

## PRODUCING OF TARTARIC AND GRAPE ACIDS ESTERS WITH STEARIC, PALMITIC, LAURIC AND OLEIC ACIDS, AND ALSO PENTADECYL, DODECYL AND DECYL ALCOHOLS

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Syntheses of non-glyceride oils and fats-derivatives of tartaric and grape acids by esterification with palmitic, stearic, lauric and oleic acids and pentadecyl, dodecyl and decyl alcohols were carried out. The physical and chemical constants of the obtained compounds are determined. New non-glyceride oil-like products can be used to screen dietary oils and fats.

**Keywords:** esterification, tartaric, grape, palmitic, stearic, lauric and oleic acids, pentadecyl, dodecyl and decyl alcohols.

**Introduction.** Natural oils and fats are mainly esters of glycerol and higher fatty acids, usually called triglycerides [1, 2]. In the human body, metabolic disorders of fats and oils can be a source of various diseases [3], of them the most common disease is obesity [4]. Obesity is usually led by a sedentary lifestyle and excessive consumption of delicious–fatty and/or sweet food. At the same time, the taste of food is largely determined by oils and fats and, as a result, it is usually difficult for a person to degrade the taste of food consumed by reducing fat content.

Obesity is treated with diets, anorectic medicines, in an operative way, but the successes of medicine are still insignificant [3, 4].

The problem of treating obesity can be approached in a new way: removing fats and oils–triglycerides from food and replacing them with non-digestible fat-like substances with similar physico-chemical and culinary properties. In our opinion, if food fatty acids were esterified with various triatomic or polyhydric alcohols (with the exception of glycerol), then substances structurally related to fats and oils (non-glyceride oils and fats) will be obtained. But unlike triglycerides, these new substances in the human body do not have specific digestive and tissue enzymes (lipases and lipogenases) and therefore will be more stable in hydrolysis and/or biosynthesis reactions of fats. This means that the new non-glyceride oils and fats will probably have dietary properties (food ballast), while preserving the physico-chemical, rheological and taste similarities with natural fats and oils of triglyceride structure [5].

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For the screening of new oil and fat substitutes it is advisable to use two-, three- and four-substituted esters of fatty acids and polyhydric alcohols of natural origin and present in food products. Such are polyols: sorbitol, mannitol, xylitol, erythritol etc., as well as fatty acids that are part of natural oils: palmitic, stearic, oleic etc. It should be noted that mannitol, xylitol, erythritol and other polyols are widely used as food sugar substitutes, and their esters with fatty acids (palmitic, stearic, etc.) with a degree of substitution of 1–1÷2 are used in the food industry as emulsifiers [6–8].

In our previous publications, data on the producing of mannitol, sorbitol, erythritol and 1,4-butanediol esters with higher fatty acids [9–11] and also citric acid with higher alcohols ( $C_6$ – $C_{18}$ ) [12] are given. In the present study the results of the synthesis of new non-glyceride esters by the esterification of tartaric and grape acids by certain higher carboxylic acids and higher aliphatic alcohols in a solution of N-methylpyrrolidone are given. The choice of this solvent is due to its effectiveness in the esterification reaction of erythritol [9].

**Results and Discussion.** The individuality of the synthesized esters is confirmed by  $^1H$  NMR and IR spectra (Fig. 1 and 2). In the IR spectra of carboxylic acids the intense band at  $940\text{ cm}^{-1}$  indicates extraneous plane deformation vibrations of OH in carboxyl groups. In addition, the presence of carboxyl groups is confirmed by wide absorption bands in the range  $2500$ – $2700\text{ cm}^{-1}$  and  $3000$ – $3300\text{ cm}^{-1}$ . Absorption in the  $1230$  and  $1290\text{ cm}^{-1}$  regions also refers to the deformation vibrations of the OH group or to the stretching vibrations of C=O in the carboxyl groups of acids.

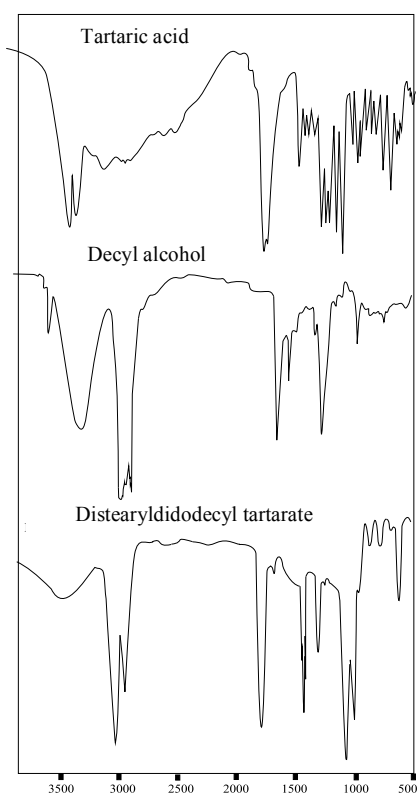


Fig. 1. IR spectra of tartaric acid, decyl alcohol and distearyl didodecyl tartarate.

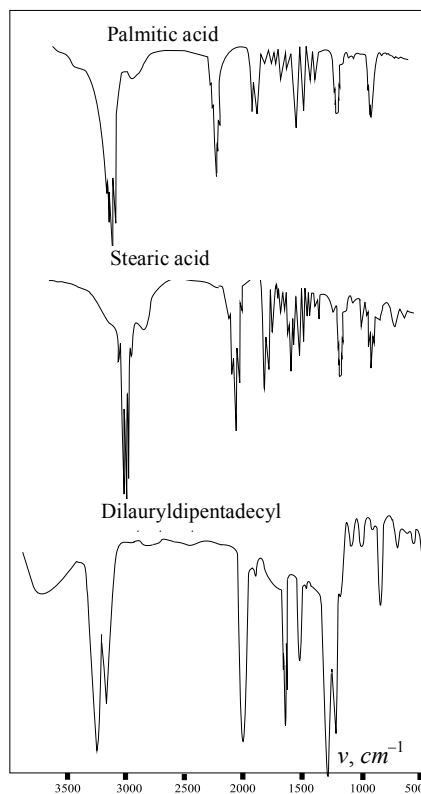
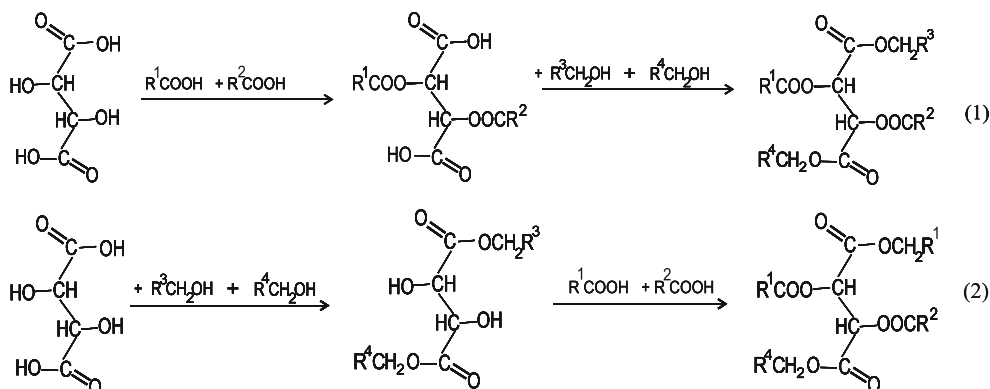


Fig. 2. IR spectra of palmitic acid, stearic acid and dilauryl dipentadecyl tartarate.

After obtaining the esterification product, tetra-substituted tartaric or grape acids, the bands of hydroxyl groups disappear, but the bands at 1740, 1230 and 1290  $cm^{-1}$  remain. This result indicates that the reaction between tartaric acid and fatty acid, and then with alcohol, is complete, and all the hydroxyl groups and carboxyl groups of tartaric acid are converted to ester bonds. The peaks of the carbonyl groups at 1740  $cm^{-1}$  of the tetra-substituted tartaric acid esters formed confirm the esterification reaction.

Unlike esterification of citric acid by higher alcohols [12] and erythritol by higher acids [11], the structure of tartaric and grape acids suggests the possibility of esterification of two hydroxyl groups by acids and then of two carboxyl groups by alcohols according to Scheme (1). The similar products, tetra-substituted tartaric and grape acids, can be obtained by reverse synthesis: first, two carboxyl groups are esterified with alcohols, then two hydroxyl groups with acids, Scheme (2).



where  $R^1$  and  $R^2$ :  $C_{17}H_{35}$  (a),  $C_{15}H_{31}$  (b),  $C_{11}H_{23}$  (c),  $C_{15}H_{29}$  (d);  $R^3$  and  $R^4$ :  $C_{15}H_{31}$  (d),  $C_{12}H_{25}$  (e),  $C_{10}H_{21}$  (f).

Accordingly, various kinds of di-substituted or tri-substituted products can be obtained, by esterification of a different number of carboxyl or hydroxyl groups. The experimental data are summarized in Tabs. 1 and 2.

As can be seen from Tab. 1, the esterification reactions of tartaric acid along route (1) with higher carboxylic acids successfully proceed at temperatures of 130–180°C for 10 h (Tab. 1, item 1, 15, 16). The yields of di-substituted ether acids are observed in the range of 93–99%. Heating the reaction mixture at a temperature of 180 or at 150°C for more than 10 h leads to a darkening of the resulting ether acids. In the first stage of the reaction for the preparation of ether acids, the yields of mono-substituted products are usually higher (96–99%) than di-substituted products (Tab. 1, items 2, 5, 8, 10, 18 and 20). The esterification of intermediate ether acids with higher aliphatic alcohols (decyl, dodecyl and penta-decyl) was also successfully realized at temperatures of 130–180°C for 6–16 h. To the degree of substitution 4, the reaction proceeds more slowly due to steric hindrance in the hydrocarbon skeleton of the partially substituted products. The heating of the reaction mixture at a temperature of 180°C for 10 h results in a blackening of the resulting oil. Darkening is partially prevented by pumping out air to 20–40 mm Hg or by passing inert gases (nitrogen, helium) under reduced

pressure, but it can not be completely avoided. Distearyl didodecyl tartarate (III (a, a, f, f) (Tab. 1, item 1) is a solid wax-like product with a softening temperature of 93–96°C. The di-substituted (Tab. 1, items 10, 18, 20, 21) and tri-substituted products (Tab. 1, item 2, 3, 5, 6, 8, 11) have lower softening temperatures. The color of the products obtained, depending on the reaction conditions, varies from milky white to brown.

Table 1

Preparation of esters along route (1): esterification of tartaric acid (TA) by carboxylic acids and intermediate ether acids by alcohols in a solution of *N*-methylpyrrolidone

№	R <sup>1,2</sup>	Molar ratio of tartaric acid to R <sup>1</sup> COOH	Reaction duration, h	T, °C	Product II	Yield of II, %	R <sup>3,4</sup>	Product III	Yield of III, %
1	a	1:2; 1:2	10+10	150	II (a, a)	95	f	III (a, a, f, f)	90
2	a	1:1; 1:2	10+10	150	II (a)	96	f	III (a, f, f)	91
3	a	1:2; 1:1	10+10	150	II (a, a)	95	f	III (a, a, f)	90
4	b	1:2; 1:2	10+10	150	II (b, b)	95	f	III (b, b, f, f)	91
5	b	1:1; 1:2	10+10	150	II (b)	97	f	III (b, f, f)	92
6	b	1:2; 1:1	10+10	150	II (b, b)	95	f	III (b, b, f)	91
7	c	1:2; 1:2	10+10	150	II (c, c)	97	e	III (c, c, e, e)	92
8	c	1:1; 1:2	10+10	150	II (c)	99	e	III (c, e, e)	94
9	c	1:2; 1:2	10+10	150	II (c, c)	97	c	III (c, c, f, f)	93
10	c	1:1; 1:2	10+10	150	II (c, f, f)	98	f	III (c, f)	94
11	c	1:2; 1:1	10+10	150	II (c, c)	98	f	III (c, c, f)	94
12	c	1:2; 1:2	8+8	150	II (c, c)	90	e	III (c, c, e, e)	81
13	c	1:2; 1:2	6+6	150	II (c, c)	87	d	III (c, c, d, d)	78
14	c	1:2; 1:2	16+16	150	II (c, c)	97	d	III (c, c, d, d)	98
15	c	1:2; 1:2	10+10	130	II (c, c)	75	e	III (c, c, e, e)	56
16	c	1:2; 1:2	10+10	180	II (c, c)	98	e	III (c, c, e, e)	96
17	d	1:2; 1:2	10+10	150	II (d, d)	94	a	III (d, d, a, a)	90
18*	d	1:1; 1:2	10+10	150	II (d, a, a)	96	a	III (d, a)	90
19*	d	1:2; 1:2	10+10	150	II (d, d)	94	e	III (d, d, e, e)	89
20*	d	1:1; 1:2	10 + 10	150	II (d)	96	e	III (d, e)	91
21*	d	1:2; 1:1	10 + 10	150	II (d)	96	e	III (d, e)	93
22*	a	1:2; 1:2	10 + 10	150	II (a, a)	95	f	III (a, a, f, f)	90
23*	b	1:2; 1:2	10 + 10	150	II (b, b)	95	f	III (b, b, f, f)	91

\* Grape acid was used in the experiments.

Tab. 2 gives data on the production of tartaric esters along Scheme (2): first, by esterification of carboxyl groups with higher aliphatic alcohols, mono- and di-substituted ether-acides are obtained. Then, by reaction of ether alcohols with higher carboxylic acids, tri- and tetra-substituted esters are obtained.

As can be seen from the Tab. 2, the main regularities of esterification along route (2) correspond to route (1): the reaction proceeds at 130–180°C in two stages for 20 h, slowing down the reactions and reducing yields as the degree of substitution increases. Dilauryl dipentadecyl ester of tartaric acid (III (d, d, c, c)) (Tab. 2, item 1) is a paraffin-like product with a softening point of 58–65°C.

Grape acid is a racemic mixture of D(–) and L(+) stereoisomeric forms of tartaric acid. A number of experiments on the esterification of grape acid

(Tab. 1, item 18–23 and Tab. 2, item 16–20) confirmed the identity of their properties in esterification reactions.

Table 2

*Preparation of esters along route (2): esterification of tartaric acid with alcohols and esterification of intermediate ether alcohols with carboxylic acids in a solution of N-methylpyrrolidone*

No	R <sup>3,4</sup>	Molar ratio of tartaric acid to R <sup>1</sup> COOH	Reaction duration, h	T, °C	Product II	Yield of II, %	R <sup>1,2</sup>	Product III	Yield of III, %
1	d	1:2; 1:2	10+10	150	II (d,d)	95	c	III (d, d, c, c)	90
2	d	1:1; 1:2	10+10	150	I (d)	96	c	III (d, c, c)	91
3	d	1:2; 1:1	10+10	150	II (a, a)	95	c	III (d, d, c)	90
4	e	1:2; 1:2	10+10	150	II (e, e)	96	c	III (e, e, c, c)	92
5	e	1:2; 1:1	10+10	150	II (e, e)	95	b	III (e, e, f)	91
6	f	1:2; 1:2	10+10	150	II (f, f)	97	a	III (f, f, a, a)	92
7	f	1:1; 1:2	10+10	150	II (f)	98	e	III (f, e, e)	94
8	f	1:2; 1:2	10+10	150	II (f, f)	97	b	III (f, f, b, b)	93
9	f	1:1; 1:2	10+10	150	II (f)	98	c	III (f, c, c)	94
10	f	1:2; 1:2	10+10	150	II (f, f)	94	a, b	III (f, f, a, b)	91
11	f	1:2; 1:2	10+10	150	II (f, f)	94	a, c	III (f, f, a, c)	91
12	f	1:2; 1:2	10+10	150	II (f, f)	95	a, d	III (f, f, a, d)	92
13	f	1:2; 1:2	10+10	150	II (f, f)	93	d	III (f, f, d, d)	90
14	f	1:2; 1:2	10+10	150	II (f, f)	94	b, d	III (f, f, b, d)	91
15	f	1:2; 1:2	10+10	180	II (f, f)	97	c, d	III (f, f, c, d)	93
16*	d	1:2; 1:2	10+10	150	II (d, d)	95	c	III (d, d, c, c)	90
17*	e	1:2; 1:2	10+10	150	II (e, e)	96	c	III (e, e, c, c)	92
18*	f	1:2; 1:2	10+10	150	II (f, f)	97	a	III (f, f, a, a)	92
19*	f	1:2; 1:2	10+10	180	II (f, f)	97	c, d	III (f, f, c, d)	93
20*	f	1:2; 1:2	10+10	130	II (f, f)	97	c, d	III (f, f, c, d)	93

\* Grape acid was used in the experiments.

**Experimental Part.** <sup>1</sup>H NMR spectra were recorded on device Varian Mercury 300 with 300 MHz operating frequency in DMSO-*d*<sub>6</sub>/CCl<sub>4</sub> (1:3) with an internal TMS standard. IR spectra are obtained on device IRS 29. Spectra of solid samples are taken in vaseline oil on KBr plates in the 4000–400 cm<sup>-1</sup> region. The individuality of the obtained substances was controlled by thin layer chromatography in a toluene+butanol (4:1) system on filter paper, iodine vapor used as a developer [13]. Tartaric and grape acids of the grade “pure for analysis”, stearic, palmitic, lauric and oleic acids, decyl, dodecyl, pentadecyl alcohols and the solvents for chromatography were used standard reagents, grades “clean” without further purification.

**General Procedure for the Esterification.** The reaction was carried out on a combined reaction-distillation installation comprising a thermostated reactor with a magnetic stirrer and a column with a reflux condenser to condense the water vapor released by the esterification reaction. The reactor was charged 30 mL N-methylpyrrolidone, 3.0 g (20 mmol) of tartaric acid and 11.93 g (42 mmol) of stearic acid. With the magnetic stirrer turned on, the air was evacuated (20–40 mm Hg) and reactor heated to the set temperature. To control the course of the reaction, every 4 h, a sample was taken and chromatographically content of starting materials and products have been determined. The reaction was carried out for 10 h, after which the flask was purged with nitrogen until the moisture was completely removed.

Then 6.64 g (42 mmol) of 1-decanol was added to the reaction flask and stirring was continued for another 10 h. After completion of the reaction, the flask was cooled, washed with water (4×50 mL) to remove reagent and solvent residues, the product was recrystallized from 50 mL of 80% ethanol, and the resulting oil was dried to constant weight. The yield of product was 17.34 g (97%). The reaction product, distearyl didecyl tartarate, III (a, a, f, f) was a hard paraffin-like material of yellow color. The described experiment is shown in the Tab. 1, for № 1, the results of the remaining experiments are tabulated. In experiments containing oleic acid (d), 0.3 g of 2,6-di-*tert*-butyl-4-methylphenol (P-23) was added to the reaction mixture to inhibit the polymerization.

The structures of the obtained tri-substituted esters of tartaric acid were confirmed by the data of IR and  $^1\text{H}$  NMR spectra.

*Distearyl Didecyl Tartarate, III (a, a, f, f)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 ( $\text{CH}_2$ ); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (92H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Dipalmitinyl Didecyl Tartarate, III (b, b, f, f)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (80H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Dilauryl Didodecyl Tartarate, III (c, c, e, e)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (76H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Dilauryl Didecyl Tartarate, III (c, c, f, f)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (68H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Dilauryl Dipentadecyl Tartarate, III (c, c, d, d)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (92H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Distearyl Dipentadecyl Tartarate, III (d, d, a, a)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (112H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Dioleyl Didodecyl Tartarate, III (d, d, e, e)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–2.02 m (92H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH); 5.33–5.35 dd (4H,  $\text{CH}=\text{}$ ).

*Palmitinyl Stearyl Didecyl Tartarate, III (f, f, a, b)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (88H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Lauryl Stearyl Didecyl Tartarate, III (f, f, a, c)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 ( $\text{C}=\text{O}$ ); 1480, 1435 ( $\text{CH}_2$ ); 1185, 1130 ( $\text{C}-\text{O}$ ).  $^1\text{H}$  NMR,  $\delta$ , ppm: 0.95 (12H,  $\text{CH}_3$ ); 1.18–1.86 m (78H,  $\text{CH}_2$ ); 2.62–2.86 dd (4H,  $\text{CH}_2\text{CO}$ ); 4.08–4.14 t (4H,  $\text{CH}_2\text{O}$ ); 4.64 s (2H, CH).

*Stearyl Oleyl Didecyl Tartarate, III (f, f, a, d)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 (C=O); 1480, 1435 (CH<sub>2</sub>); 1185, 1130 (C–O). <sup>1</sup>H NMR,  $\delta$ , ppm: 0.95 (12H, CH<sub>3</sub>); 1.18–1.86 m (88H, CH<sub>2</sub>); 2.62–2.86 dd (4H, CH<sub>2</sub>CO); 4.08–4.14 t (4H, CH<sub>2</sub>O); 4.64 (2H, CH), 5.33–5.35 dd (2H, CH=).

*Palmitinil Oleyl Didecyl Tartarate, III (f, f, b, d)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 (C=O); 1480, 1435 (CH<sub>2</sub>); 1185, 1130 (C–O). <sup>1</sup>H NMR,  $\delta$ , ppm: 0.95 (12H, CH<sub>3</sub>); 1.18–1.86 m (84H, CH<sub>2</sub>); 2.62–2.86 dd (4H, CH<sub>2</sub>CO); 4.08–4.14 t (4H, CH<sub>2</sub>O); 4.64 (2H, CH), 5.33–5.35 dd (2H, CH=).

*Lauryl Oleyl Didecyl Tartarate, III (f, f, c, d)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 (C=O); 1480, 1435 (CH<sub>2</sub>); 1185, 1130 (C–O). <sup>1</sup>H NMR,  $\delta$ , ppm: 0.95 (12H, CH<sub>3</sub>); 1.18–1.86 m (76H, CH<sub>2</sub>); 2.62–2.86 dd (4H, CH<sub>2</sub>CO); 4.08–4.14 t (4H, CH<sub>2</sub>O); 4.64 (2H, CH), 5.33–5.35 dd (2H, CH=).

*Dioleyl Dipentadecyl Tartarate, III (f, f, d, d)*. IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2920, 2850 (CH); 1773 (C=O); 1480, 1435 (CH<sub>2</sub>); 1185, 1130 (C–O). <sup>1</sup>H NMR,  $\delta$ , ppm: 0.95 (12H, CH<sub>3</sub>); 1.18–2.02 m (104H, CH<sub>2</sub>); 2.62–2.86 dd (4H, CH<sub>2</sub>CO); 4.08–4.14 t (4H, CH<sub>2</sub>O); 4.64 s (2H, CH); 5.33–5.35 dd (4H, CH=).

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