Composition of Representative SALATRIM Fat Preparations

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SALATRIM is a family of structured triacylglycerols containing at least one short-chain fatty acid and one saturated long-chain fatty acid (predominantly stearate) on each glycerol. The mixtures are prepared by the interesterification of hydrogenated vegetable oils such as soy or canola, with triacetin and/or tripropionin and/or tributyrin. Qualitative and quantitative analyses were conducted on five SALATRIM preparations. Qualitatively, several triacylglycerols are common to various members of the SALATRIM family. Approximately 90% of the total mass of the SALATRIM preparations is accounted for in positively identified triacylglycerols. Free fatty acids, phytosterols, and tocopherols account for an additional 0.4–0.8% of the mass of the SALATRIM preparations. The triacylglycerol distribution is in excellent agreement with the predicted distribution based on the fatty acid composition of the starting material and supports the concept of random distribution of the fatty acids in interesterified oils. Interesterified oils were further refined by distillation, to yield final product, which captured nearly 100% acylglycerols. No unexpected compounds were detected. Supporting the predictable nature of the interesterification reaction, it is concluded that the SALATRIM family is composed of a series of randomly esterified triacylglycerols containing a mixture of short- and long-chain fatty acids.

INTRODUCTION

In foods, fat is frequently associated with high quality and a pleasant eating experience. Food texture and flavor delivery are both frequently improved when a high level of fat is present in a product. Increasing concern regarding health and nutrition by consumers has led to interest in products with fewer calories from fat. One approach to providing fewer calories from fat is to replace fat with a low-calorie fat substitute. Nabisco Foods Group has developed a family of low-calorie fats called SALATRIM. SALATRIM fats are mixtures of triacylglycerols formed by the interestification of hydrogenated vegetable oils with triglycerides of short-chain fatty acids (triacetin and/or tripropionin and/or tributyrin). The methods of preparation and the model that predicts the composition are described by Klemann et al. (1994). The family of triacylglycerol compositions reported by Klemann et al. were synthesized by a sodium methoxide catalyzed interesterification of a short-chain triglycerides (SCT) and a saturated long-chain triglyceride (LCT). The process of interesterification results in a random distribution of the short- and long-chain acid esters with respect to the glycerol. The molar ratios of the SCT and LCT reactants determine the relative concentration of the various possible triacylglycerol products and isomeric structures. All excess SCT was removed by steam distillation under high vacuum. This was done to control short- to long-chain fatty acid ratio for specific products. The lots were compared to establish reproducibility of the synthesis procedures. In studies with rats and in human clinical studies it has been demonstrated that SALATRIM provides fewer calories (4.5–5.5 kcal/g) than conventional fats such as corn oil (9 kcal/g) (Finley et al., 1994). One of the goals of this work was to perform a mass balance study to account for as much of the SALATRIM preparation products as possible. The present study was undertaken to support animal studies (Hayes et al., 1994a–c) and human clinical studies (Finley et al., 1994). In this study the chemical composition of SALATRIM preparations is compared to the predicted compositions based on the fatty acid profiles of the starting materials. The analyses of non-triacylglycerol components of the SALATRIM preparations are also reported.

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Table 1. Molar Ratio of Short- and Long-Chain Acid Sources Used To Prepare the SALATRIM Family of Edible Oils

SALATRIM family	short-chain source	long-chain source	mole ratio
SALATRIM 4CA	tributyrin	hydrogenated canola oil	2.5:1
SALATRIM 23CA	triacetin tripropionin	hydrogenated canola oil	11:1:1
SALATRIM 234CS	triacetin tripropionin tributyrin	hydrogenated cottonseed oil	4:4:4:1
SALATRIM 234CA	triacetin tripropionin tributyrin	hydrogenated canola oil	4:4:4:1
SALATRIM 23SO	triacetin tripropionin	hydrogenated soybean oil	11:1:1

MATERIALS AND METHODS

Materials. All materials were prepared by the interesterification of either hydrogenated soybean, hydrogenated canola, or hydrogenated cottonseed oils with appropriate short-chain aliphatic triacyglycerols as described by Klemann et al. (1994). Starting materials and molar ratios for interesterification are summarized in Table 1. The excess short-chain triesters served as "solvents" for the syntheses and were removed during the deodorization process. Materials were distilled in a Pope 2 in. wiped film still wiped film distillation apparatus (Pope Scientific, Menomonee Falls, WI). The body temperature of the apparatus was 190 °C, while the inner core temperature was still 80 °C, and the vacuum was 0.04 Torr.

Methods. The SALATRIM preparations described in Table 1 are characterized using a series of conventional analytical

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Table 2. Chemical Characterization of Selected SALATRIM Compounds

analysis	method	% RSD[a] of method	ref
acylglycerols	qualitative and quantitative composition of the SALATRIM family of compounds	5.0	Huang et al. (1994)
free fatty acids	composition of free fatty acids	21.3	Castellano (1993)[b]
total fatty acid	gas chromatography of methyl esters (FAME)	7.8	Greeley (1992) West (1975)
metals analysis	determination of metal composition	14.9	AOCS Ca 18b-91 (1991a); AOCS Ca 18c-91 (1991b)
non-acyl glycerol organics	tocopherol plant sterols	2.81 4.7	AOCS Ce 8-89 (1991c) AOAC 976.26 (1990)

[a] Percent relative standard deviation. [b] Private communication. See Methods.

Table 3. Fatty Acid Composition (Weight Percent)[a] of Selected Members of the SALATRIM Family

type	canola[b]	soy	cotton	corn oil[c,d]	23CA	23SO	4CA	234CS	234CA
C2:0					25.95 ± 0.12	26.14 ± 2.88		8.39 ± NC[e]	10.06 ± 0.21
C3:0					3.07 ± 0.04	3.38 ± 0.42		9.33 ± NC	10.90 ± 0.43
C4:0							21 ± 2.0	12.38 ± NC	13.68 ± 0.65
C12:0	0.019 ± 0.001	0.026 ± 0.001	<0.015	<0.15	<0.015	<0.015	0.019 ± 0.003	0.025 ± 0.001	<0.015
C14:0	0.061 ± 0.002	<0.015	<0.015	0.047 ± 0.003	0.078 ± 0.071	0.078 ± 0.071	NC	0.534 ± 0.029	0.056 ± 0.004
C16:0	4.952 ± 0.224	12.24 ± 0.73	25.80 ± 0.05	9.681 ± 0.527	2.37 ± 0.04	7.97 ± 0.13	2.75 ± 0.08	12.30 ± 0.14	3.23 ± 0.08
C18:0	81.53 ± 0.43	79.59 ± 4.75	72.30 ± 0.11	2.611 ± 0.118	57.0 ± 0.00	55.68 ± 1.11	58.0 ± 4.0	41.81 ± 0.48	49.11 ± 5.48
C18:1	1.597 ± 0.151	0.13 ± 0.01	0.18 ± 0.00	24.604 ± 1.929	0.572 ± 0.005	0.04 ± 0.01	0.56 ± 0.022	0.06 ± 0.11	0.77 ± 0.08
C18:2	0.0867 ± 0.341	<0.015	0.34 ± 0.07	53.83 ± 2.99	0.066 ± 0.0004	<0.015	0.124 ± 0.003	0.01 ± 0.01	0.13 ± 0.02
C20:0	1.8443 ± 0.0714	0.63 ± 0.03	0.09 ± 0.07	0.464 ± 0.0311	1.50 ± 0.03	0.47 ± 0.07	1.51 ± 0.05	0.26 ± 0.009	2.17 ± 0.13
C22:0	0.629 ± 0.01109	0.38 ± 0.02		0.186 ± 0.0167	0.668 ± 0.007	0.29 ± 0.04	0.64 ± 0.02	0.11 ± 0.01	0.63 ± 0.05
C24:0	0.30 ± 0.061	0.14 ± 0.01		9.681 ± 0.0527	0.335 ± 0.03	0.11 ± 0.02	0.299 ± 0.008	0.08 ± 0.010	0.31 ± 0.02

[a] Means of three determinations, C2:0–C4:0 determined as free acids and converted to methyl esters. [b] Pooled av ± standard deviation, 3 measurements of $n = 3$ each. [c] Pooled av ± standard deviation, 2 measurements of $n = 3$ each. [d] Included as reference only. [e] NC, not calculated.

procedures developed specifically for characterization of these unique triacylglycerols. The methods and the relative standard deviations for the methods are summarized in Table 2.

Volatile fatty acids were quantified by aqueous saponification of the SALATRIM family of compounds with sodium hydroxide, followed by acidification with concentrated hydrochloric acid. Acidified samples were analyzed by gas chromatographically by direct injection.

Seventh–tenth gram quantities were weighed into 100-mL round-bottom flasks containing 5–10 mL of water and were refluxed for 45–60 min. Control samples containing known amount of acetic and propionic and/or butyric were prepared identically as positive controls and to validate the procedure. Upon completion of hydrolysis, 20 mL of water was added through the condensor. After cooling, 5 mL of water was added and the flasks are stoppered immediately and maintained at <40 °F if necessary.

The aqueous phases were added to separatory funnels containing 15 mL of heptane and were extracted with 3 × 20 mL portions of water. The extracts were quantitatively transferred to 200-mL volumetric flasks and acidified to pH <3. Following acidification, the solutions were brought to volume with water. Dilutions were made using hydrochloric acid solution (pH 2) as necessary for GC analysis. Samples and standards were stored in brown glass bottles below 40 °F.

Gas chromatographic analyses for these volatile acids are accomplished using a 30-m stabilized Carbowax 20 M column with a film thickness of 1 μm and a 0.53 mm i.d. The detector is a flame ionization type (FID) set to 250 °C. The injection is cool on-column, and the injection volume is 1–3 μL with temperature tracking. The carrier gas was helium set at 5.0 psig.

RESULTS AND DISCUSSION

The fatty acids in the starting materials (hydrogenated canola, hydrogenated cottonseed, and hydrogenated soy oils) and five different SALATRIM preparations are reported in Table 3. From the data it can be seen that the predominant long-chain fatty acids are palmitic and stearic acids. The differences in fatty acid distribution of the starting materials (hydrogenated canola, hydrogenated cottonseed, and hydrogenated soyoils) are reflected in the fatty acid profiles of the SALATRIM products produced by interesterification. These vegetable oils were highly hydrogenated as evidenced by the low levels of unsaturated fatty acids found in the starting materials and the interesterification products. The short-chain fatty acids in the SALATRIM preparations are reflections of the ratios of short-chain fatty acids in the reaction mixtures. For example, in SALATRIM 23CA the molar ratio of acetic acid to propionic acid in the reaction mixture is 10:1. Analysis of the final mixture revealed weight percentages of acetic and propionic acids to be 25.95% and 3.07%, respectively. When these data are converted to mole ratio, the acetic to propionic acid ratio for 23CA is 10.1:1.0. In 234CA and 234CS conversion of the weight percentages resulted in molar ratios for acetic to propionic to butyric acid of 1.2:1.0:1.0 and 1.1:0.85:1, respectively. The ratios of acetic to propionic to butyric acid approximate the 1:1:1 molar ratio used in the reaction to produce 234CA and 234CS. The ratio of short- to long-chain fatty acids in the products is significantly different from that in the original reaction mixture. The short- to long-chain ratio in the original interesterification reaction mixture was 12:1. As reported by Klemann et al. (1994) the short-chain triglycerides serve as solvents for the interesterification reaction. In the refining process the excess tri-short triacylglycerols in the reaction mixture are removed. The SALATRIM preparations thus contain only triacylglycerols with at least one long-chain fatty acid on the molecule. The combination of triacylglycerols in the mixtures can be summarized as SSL, SLS, LSL, LLS, where S is a short-chain fatty acid and L is a long-chain fatty acid.

Prediction of Distribution of Fatty Acids on Triacylglycerols. The distribution of triacylglycerol species for a random interesterification reaction can be precisely predicted from the mole ratios of the fatty acids present in the reaction mixture. The mole fraction of triacyl-

Table 4. Predicted vs Actual Triacylglycerols in SALATRIM 4CA

acylglycerol	pred	wt %
1,2-dibutyryl-3-stearoylglycerol	37.24	37.32 ± 0.48
1,3-dibutyryl-2-stearoylglycerol	18.62	17.87 ± 0.48
1-butyryl-2,3-distearoylglycerol	19.15	18.54 ± 0.062
1,3-distearoyl-2-butyrylglycerol	9.58	9.62 ± 0.036
tristearoylglycerol	4.53	2.82 ± 0.054
1,2-dibutyryl-3-palmitoylglycerol	1.72	1.71 ± 0.008
1-butyryl-2-palmitoyl-3-stearoylglycerol	0.9	1.59 ± 0.009
1-butyryl-2-stearoyl-3-palmitoylglycerol	0.9	
2-butyryl-1-palmitoyl-3-stearoylglycerol	0.9	0.77 ± 0.019
1,3-dibutyryl-2-palmitoylglycerol	0.86	0.81 ± 0.012
1,2-dibutyryl-3-arachidoylglycerol	0.75	0.86 ± 0.004
2-butyrylstearoyloleoylglycerol	0.685	0.15 ± 0.008
1-palmitoyl-2,3-distearoylglycerol	0.43	<0.01
2-butyryl-1-stearoyl-3-arachidoylglycerol	0.38	
1,3-dibutyryl-2-arachidoylglycerol	0.38	0.4 ± 0.004
1-butyryl-2-stearoyl-3-arachidoylglycerol	0.38	0.69 ± 0.015
1-butyryl-2-arachidyl-3-stearoylglycerol	0.38	0.40 ± 0.015
1,2-dibutyryl-3-behenoylglycerol	0.33	0.33 ± 0.01
1,3-distearoyl-2-palmitoylglycerol	0.21	
1,2-distearoyl-3-arachidoylglycerol	0.18	
2-butyryl-1-stearoyl-3-behenoylglycerol	0.17	
1,3-dibutyryl-2-behenoylglycerol	0.17	0.2 ± 0.008
1-butyryl-2-stearoyl-3-behenoylglycerol	0.17	0.2 ± 0.008
1-butyryl-2-behenoyl-3-stearoylglycerol	0.17	
1,2-dibutyryl-3-oleoylglycerol	0.15	1.1 ± 0.21
2-butyryl-1-stearoyl-3-oleoylglycerol	0.15	
total diacylglycerols	0	1.47
total acylglycerols	99.49	96.85 ± 0.72

glycerol having fatty acid i in the sn-1 position, fatty acid j in the sn-2 position, and fatty acid k in the sn-3 position is given as

$$F_{ijk} = \frac{X_i X_j X_k}{\sum_{l}^{N}\sum_{m}^{N}\sum_{n}^{N} X_l X_m X_n}$$

where X stands for the fatty acid mole fraction in the reaction mixture and i–n are indices for the fatty acid, i.e., 1 for acetic, 2 for propionic, 3 for butyric, 4 for palmitic, etc. The summations are for all fatty acid species from 1 to N. There should be N^3 possible unique triglycerides.

For the purpose of this model we assume that the 1- and 3-positions of the glycerol are indistinguishable; F_{iij} and F_{jii} are thus combined, where i is not equal to j. The denominator is unaffected.

To account for the loss of volatile triacylglycerols during the deodorization process, all terms in the denominator due to species with three "short-chain" fatty acids (acetic, propionic, and butyric) were removed from the sum and the values of F_{ijk} renormalized accordingly. The weight percent of each distinguishable triacylglycerol is then given as

$$\% W_{ijk} = \frac{F_{ijk}'(\mathrm{MW}_{ijk})}{\sum_{l}^{N}\sum_{m}^{N}\sum_{n}^{N} F_{lmn}'(\mathrm{MW}_{lmn})} \times 100$$

where all volatile species were excluded from the sum. MW_{ijk} is the molecular weight of the triacylglycerol with fatty acids i, j, and k, and F_{ijk}' is the renormalized mole fraction.

Proportions of all possible triacylglycerols have been predicted on the basis of the distribution of fatty acids in the starting vegetable oil (Table 3) and the short/long fatty acid ratio of the SALATRIM preparation. The model assumes that all tri-short-chain triacylglycerols are removed in the refining process. In Tables 4–7 the predicted and identified triacylglycerols are reported for SALATRIM preparations. The tables include all triacylglycerols predicted to occur at 0.1% or greater in the product. An assumption was made that all tri-short triacylglycerols are removed in the refining process. This was confirmed by the absence of detectable short-chain triacylglycerols in all materials tested. From the data in the tables it can be seen that there is good correlation between the predicted and the observed triacylglycerols. Regression analyses comparing the predicted vs observed triacylglycerols (for materials greater than 0.1%) are shown in Table 8. From the analyses it can be seen that R^2 (correlation coefficient) values are consistently over 0.99 for all five SALATRIM preparations. The model also predicts that significant amounts of triacylglycerols would be expected at levels

Table 5. Predicted vs Actual Triacylglycerols in SALATRIM 23SO and 23CA

	23CA		23SO	
acylglycerol	pred	wt %	pred	wt %
1,2-diacetyl-3-stearoylglycerol	46.81	38.49 ± 0.281	43.25	39.63 ± 0.054
1,3-diacetyl-2-stearoylglycerol	23.4	19.24 ± 0.069	21.63	19.82 ± 0.035
1-acetyl-2,3-distearoylglycerol	5.92	7.58 ± 0.071	5.15	6.54 ± 0.005
1-acetyl-2-propionyl-3-stearoylglycerol	4.39	3.82 ± 0.098	4.06	3.95 ± 0.023
1-acetyl-2-stearoyl-3-propionylglycerol (1)	4.39	7.67 ± 0.066	4.07	7.87 ± 0.012
2-acetyl-1-propionyl-3-stearoylglycerol (1)	4.39		4.06	
2-acetyl-1,3-distearoylglycerol	2.96	3.67 ± 0.062	2.58	2.82 ± 0.001
1,2-diacetyl-3-palmitoylglycerol	2.15	1.49 ± 0.016	5.92	4.68 ± 0.033
1,3-diacetyl-2-palmitoylglycerol	1.07	0.77 ± 0.011	2.96	2.29 ± 0.182
1-propionyl-2,3-distearoylglycerol	0.55	0.57 ± 0.021	0.48	0.6 ± 0.003
1,2-dipropionyl-3-stearoylglycerol	0.41	0.41 ± 0.007	0.38	0.46 ± 0.109
1,2-diacetyl-3-oleoylglycerol	0.37	<0.1	0.14	ND
tristearoylglycerol	0.33	0.71 ± 0.083	0.27	0.48 ± 0.008
1-acetyl-2-stearoyl-3-palmitoylglycerol	0.28		0.72	2.09 ± 0.0020
1-acetyl-2-palmitoyl-3-stearoylglycerol	0.28	1.22 ± 0.006	0.72	
2-acetyl-1-palmitoyl-3-stearoylglycerol	0.28		0.72	0.89 ± 0.005
2-propionyl-1,3-stearoylglycerol	0.27	0.37 ± 0.128	0.24	0.31 ± 0.004
1,3-dipropionyl-2-stearoylglycerol	0.21	0.18 ± 0.002		0.19 ± 0.020
1-acetyl-2-palmitoyl-3-propionylglycerol	0.2		0.56	1.07 ± 0.191
2-acetyl-1-propionyl-3-palmitoylglycerol	0.2	0.58 ± 0.079		0.59 ± 0.079
1-acetyl-2-propionyl-3-palmitoylglycerol	0.2		0.57	
2-acetyl-1-propionyl-3-palmitoylglycerol	0.2	1.38 ± 0.007	0.56	0.61 ± 0.049
1,3-diacetyl-2-oleoylglycerol	0.18		0.07	
1,2-diacetyl-3-arachidoylglycerol	0.1			0.36 ± 0.076
total diacylglycerols		2.29		2.24
total acylglycerols	99.34	90.44 ± 0.37	99.11	97.49 ± 0.32

Table 6. Predicted vs Actual Triacylglycerols in SALATRIM 234CA

acylglycerols	pred	wt %
2-acetyl-1-butyryl-3-stearoylglycerol	6.25	
1-acetyl-2-butyryl-3-stearoylglycerol	6.25	
1,2-dipropionyl-3-stearoylglycerol	6.25	24.81 ± 0.18
1-acetyl-2-stearoyl-3-butyrylglycerol	6.25	
1,3-dipropionyl-2-stearoylglycerol	3.12	
1-propionyl-2-butyryl-3-stearoylglycerol	6.43	
2-propionyl-1-butyryl-3-stearoylglycerol	6.43	16.93 ± 0.34
1-propionyl-2-stearoyl-3-butyrylglycerol	6.43	
1,2-dibutyryl-3-stearoylglycerol	6.62	
1,3-dibutyryl-2-stearoylglycerol	3.31	9.76 ± 0.02
1,2-diacetyl-3-stearoyl-glycerol	5.87	4.53 ± 0.06
1-acetyl-2-stearoyl-3-propionylglycerol	6.06	
2-acetyl-1-propionyl-3-stearoylglycerol	6.06	15.10 ± 0.20
1-acetyl-2-propionyl-3-stearoylglycerol	6.06	
1,3-diacetyl-2-stearoylglycerol	2.94	2.19 ± 0.02
1-butyryl-2,3-distearoylglycerol	2.13	1.96 ± 0.18
1-acetyl-2,3-distearoylglycerol	2.04	1.7 ± 0.01
1,3-distearoyl-2-butyrylglycerol	1.06	1.09 ± 0.01
1,3-stearoyl-2-propionylglycerol	1.04	0.99 ± 0.018
1,3-distearoyl-2-acetylglycerol	1.02	0.8 ● 0.01
1-propionyl-2-butyryl-3-palmitoylglycerol	0.3	
2-propionyl-1-butyryl-3-palmitoylglycerol	0.3	1.31 ± 0.03
1-propionyl-2-palmitoyl-3-butyrylglycerol	0.3	
1-acetyl-2-palmitoyl-3-propionylglycerol	0.28	0.72 ± 0.01
1-acetyl-2-propionyl-3-palmitoylglycerol	0.28	
2-acetyl-1-propionyl-3-palmitoylglycerol	0.1	
1,2-dipropionyl-3-palmitoylglycerol	0.29	
1-acetyl-2-butyryl-3-palmitoylglycerol	0.29	1.64 ± 0.08
2-acetyl-1-butyryl-3-palmitoylglycerol	0.29	
1,3-dipropionyl-2-palmitoylglycerol	0.14	
tristearoylglycerol	0.31	0.37 ± 0.03
1,2-dibutyryl-3-palmitoylglycerol	0.31	0.79 ± 0.04
1-propionyl-2-palmitoyl-3-butyrylglycerol	0.3	0.20 ± 0.00
1-acetyl-2-palmitoyl-3-butyrylglycerol (1)	0.29	0.33 ± 0.03
1,2-diacetyl-3-palmitoylglycerol	0.27	0.2 ± 0.00
1,3-dibutyryl-2-palmitoylglycerol	0.15	0.38 ± 0.01
1-acetyl-2-palmitoyl-3-stearoylglycerol	0.1	0.18 ± 0.01
2-propionyl-1-palmitoyl-3-stearoylglycerol	0.1	0.19 ± 0.00
1-propionyl-2-palmitoyl-3-stearoylglycerol	0.1	0.19 ± 0.005
1-butyryl-2-stearoyl-3-palmitoylglycerol	0.1	0.21 ± 0.001
total diacylglycerols	NP	2.08 ± 0.50
total acylglycerols	96.22	88.35 ± 0.72

below our arbitrary cutoff of 0.1%. The amount of triacylglycerols predicted below the 0.1% cutoff increased with the complexity of the mixture. For example, the model predicts that 4CA would contain a total of 0.82% triacylglycerols below 0.17% in concentrate, while the 234CS would contain a total of 2.97% triacylglycerols below the 0.17% threshold. Comparing the data in Tables 4–7, one sees that when more short-chain fatty acids are included, the reaction mixtures become progressively more complicated and chromatographic resolution of some peaks becomes increasingly difficult. This was particularly true in 234CA and 234CS, where multiple compounds with similar molecular weight appeared in various regions of the chromotogram. Qualitative identification of the mixtures under the unresolved peaks was conducted by HTGC/MS as described by Huang et al. (1994a,b). For the purpose of regression analysis the totals for the observed unresolved peaks were compared to the total values predicted for the particular triacylglycerols. The response of the chromatographic system was nonlinear below 0.1%. The total fatty acid data in Table 3 also help confirm the observed triacylglycerol data. Estimation of fatty acid residues for the data in Tables 4–7 correlated with fatty acid data in Table 3 at $R^2 = 0.95$ or greater for all SALATRIM preparations.

Klemann et al. (1994) predict that a random distribution of fatty acids results in a 2:1 ratio of 1,2- to 1,3-isomers of triacylglycerols such as diacetylstearoylglycerol. The results in Tables 4–7 confirm the predictions. For instance, in Table 4 (SALATRIM 4CA) the ratio of 1,2-dibutyryl-3-stearoylglycerol (BBS) and 1,3-dibutyryl-2-stearoylglycerol (BSB) is 2.09:1. Similarly, in Table 6 (SALATRIM 234CA) the identical compounds occur at a ratio (BBS/BSB) of 2:1. In Tables 4–7, in addition to the predicted triacylglycerols, small amounts of diacylglycerols such as acetylstearoylglycerol (Tables 5–7) were observed. The model does not predict that a diacylglycerol would be formed when simple interesterification chemistry is assumed. In the deodorization process SALATRIM is treated with steam at high temperature and, although this is done under vacuum, it is possible for limited hydrolysis

Table 7. Predicted vs Actual Triacylglycerols in SALATRIM 234CS

acylglycerols	pred	wt %
1,2-dibutyryl-3-stearoylglycerol	5.15	5.61 ± 0.07
1,3-dibutyryl-2-stearoylglycerol	2.57	2.25 ± 0.07
2-propionyl-1-butyryl-3-stearoylglycerol	5.00	
1-propionyl-2-butyryl-3-stearoylglycerol	5.00	13.87 ± 0.23
1-propionyl-2-stearoyl-3-butyrylglycerol	5.00	
2-acetyl-1-butyryl-3-stearoylglycerol	4.86	
1-acetyl-2-butyryl-3-stearoylglycerol	4.86	20.40 ± 0.11
1,2-dipropionyl-3-stearoylglycerol	4.86	
1-acetyl-2-stearoyl-3-butyrylglycerol	4.86	
1,3-dipropionyl-2-stearoylglycerol	2.43	
2-acetyl-1-propionyl-3-stearoylglycerol	4.71	
1-acetyl-2-stearoyl-3-propionylglycerol	4.71	12.62 ± 0.09
1-acetyl-2-propionyl-3-stearoylglycerol	4.71	
1,2-diacetyl-3-stearoylglycerol	4.57	3.79 ± 0.08
1,3-diacetyl-2-stearoylglycerol	2.28	1.89 ± 0.03
1,2-dibutyryl-3-palmitoylglycerol	1.68	1.66 ± 0.02
1-propionyl-2-palmitoyl-3-butyrylglycerol	1.63	
1-propionyl-2-butyryl-3-palmitoylglycerol	1.63	4.47 ± 0.05
2-propionyl-1-butyryl-3-palmitoylglycerol	1.63	
1-acetyl-2-butyryl-3-palmitoylglycerol	1.58	
1-acetyl-2-palmitoyl-3-butyrylglycerol	1.58	
1,2-dipropionyl-3-palmitoylglycerol	1.58	6.20 ± 0.01
1,3-dipropionyl-2-palmitoylglycerol	0.79	
2-acetyl-1-butyryl-3-palmitoylglycerol	1.58	
2-acetyl-1-propionyl-3-palmitoylglycerol	1.53	
1-acetyl-2-palmitoyl-3-propionylglycerol	1.53	3.56 ± 0.06
1-acetyl-2-propionyl-3-palmitoylglycerol	1.53	
1,2-diacetyl-3-palmitoylglycerol	1.48	1.07 ± 0.17
1-butyryl-2,3-distearoylglycerol	1.30	1.28 ± 0.02
1-propionyl-2,3-distearoylglycerol	1.27	1.21 ± 0.02
1-acetyl-2,3-distearoylglycerol	1.25	1.17 ± 0.02
1,3-dibutyryl-2-palmitoylglycerol	0.84	0.80 ± 0.01
1,3-diacetyl-2-palmitoylglycerol	0.74	1.07 ± 0.02
2-butyryl-1,3-distearoylglycerol	0.65	0.67 ± 0.02
2-propionyl-1,3-distearoylglycerol	0.64	0.61 ± 0.02
2-acetyl-1,3-distearoylglycerol	0.62	0.54 ± 0.01
2-butyryl-1-palmitoyl-3-stearoylglycerol	0.43	0.45 ± 0.02
2-propionyl-1-palmitoyl-3-stearoylglycerol	0.42	0.41 ± 0.01
2-acetyl-1-palmitoyl-3-stearoylglycerol	0.41	0.35 ± 0.01
1-acetyl-2-stearoyl-3-palmitoylglycerol	0.41	0.75 ± 0.01
1-acetyl-2-palmitoyl-3-stearoylglycerol	0.41	
1-propionyl-2-stearoyl-3-palmitoylglycerol	0.42	1.21 ± 0.01
1-propionyl-2-palmitoyl-3-stearoylglycerol	0.42	
1-palmitoyl-2-propionyl-3-stearoylglycerol	0.42	
1-palmitoyl-2-butyroyl-3-stearoylglycerol	0.43	
1-butyryl-2-stearoyl-3-palmitoylglycerol	0.43	1.31 ± 0.02
1-butyryl-2-palmitoyl-3-stearoylglycerol	0.43	
tristearoylglycerol	0.15	0.15 ± 0.005
1-acetyl-2,3-dipalmitoylglycerol	0.14	
1-propionyl-2,3-dipalmitoylglycerol	0.14	0.15 ± 0.002
total diacylglycerols	NP	2.97 ± 0.05
total acylglycerols	97.69	92.50 ± 0.67

Table 8. Regression Analysis of Predicted vs Observed Triacylglycerols

	SALATRIM family member				
parameter	4CA	23CA	23SO	234CA	234CS
R^2	0.997	0.995	0.997	0.994	0.997
no. of observations	18	18	17	23	28
degrees of freedom	16	16	15	21	26

Table 9. Analysis of Metals in the SALATRIM Family of Compounds

	starting materials				SALATRIM compounds				
metal (ppm)	CA[a]	CS[b]	SO[c]	C[d]	23SO	23CA	4CA	234CS	234CA
Al	<0.308	<0.308	<0.308		<0.308	0.40	<0.308	<0.308	<0.308
As	<0.25	<0.25	<0.25		<0.25	<0.25	<0.25	<0.25	<0.25
Cd	<1.25	<0.088	<1.25	<1.25	<0.088	<0.088	<0.088	<1.25	<1.25
Cu	<0.5	<0.088	<0.5	<0.5	<0.05	0.70	<0.088	<0.30	<0.5
Co	<0.088	<0.088	<0.088		<0.088	<0.088	<0.088	<0.088	<0.088
Ca	0.85	<0.698	<0.50	<0.25	<0.05	1.10	0.21	<0.25	0.26
Cr	<0.05	<0.08	<1.25	<0.5	<0.08	0.10	<0.08	<0.5	<0.5
Fe	<1.25	<0.092	<1.25	<1.25	<0.092	0.80	0.50	<1.25	<1.25
Mg	<1.25	<0.555	<1.25	<1.25	<0.555	<0.555	<0.555	<1.25	<1.25
Mn	<0.5	<0.090	<0.50	<0.5	<0.090	<0.090	<0.090	<0.5	<0.5
Ni	<2.5	<0.092	<2.5	<2.5	<0.092	<0.092	<0.092	<2.5	<2.5
K	<12.5	<5.07	<12.5	<12.5	<5.07	<5.07	<5.07	<12.5	<12.5
Na	<1.25	<0.655	<1.25	<1.25	<0.655	4.70	1.40	<1.25	<1.25
Zn	<1.25	<0.082	<1.25	<1.25	0.11	1.00	<0.082	<1.25	<1.25
Pb (ppb)	<25	<72	<30	0.06	<72	<72	<72	<0.05	0.09
P	<0.622	<0.622	<0.622		<0.622	1.71	<0.622	0.70	<0.622

[a] CA, hydrogenated canola oil. [b] SO, hydrogenated soy oil. [c] CS, hydrogenated cottonseed oil. [d] C, corn oil.

Table 10. Phytosterol Composition (Milligrams per 100 g) of SALATRIM Preparations and Starting Materials

phytosterol	corn oil[a]	soy oil[a]	canola oil[a]	cottonseed oil[a]	4CA[b]	23CA[c]	23SO[a]	234CA[b]	234CS[a]
cholesterol	0.00	1.27	3.15	3.24	1.54	0.36	0.00	8.20	0.00
brassicasterol	0.00	6.28	3.89	1.03	5.07	3.29	2.96	5.07	1.57
campesterol	123.07	8.28	185.17	8.36	90.22	84.37	10.40	109.41	30.03
campestanol	8.17	40.80	90.41	76.50	47.11	69.44	35.70	36.85	95.53
Δ^2-campestene	0.00	14.78	11.88	8.13	19.91	19.80	4.76	14.89	0.00
campestane	0.00	2.34	39.89	0.00	1.30	11.48	1.31	2.30	4.97
stigmasterol	39.91	10.39	3.17	0.00	0.00	1.43	4.31	0.62	0.00
stigmastanol	23.24	23.54	57.89	52.91	35.54	50.40	16.24	28.03	67.50
Δ^6-stigmastene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.89	1.64
stigmastane	0.00	17.01	46.04	7.87	19.33	17.40	3.12	5.09	2.90
isostigmastane	0.00	6.74	46.84	0.00	6.90	16.52	10.98	5.56	3.02
β-sitosterol	424.34	64.34	246.92	238.68	123.19	106.92	38.08	152.08	106.94
$\Delta^{8(14)}$-ergostene	0.00	3.47	37.24	0.89	2.36	11.65	1.40	4.13	0.00
ergostane	0.00	5.04	7.92	3.61	26.77	23.30	2.51	0.00	0.00
total	618.73	204.26	780.42	401.23	379.25	416.37	131.76	373.11	314.10

[a] Single determination. [b] Mean of duplicate determinations. [c] Mean of triplicate determinations.

to take place. In SALATRIM 23CA the two diacetylstearoylglycerol isomers are the most prevalant species. Upon hydrolysis both isomers yield acetylstearoylglycerol. Acetylstearoylglycerol was found at low levels as might be expected with limited hydrolysis. The low levels of diacylglycerols do not appear to have any impact on the function of SALATRIM preparations or on the overall fatty acid distribution in the products. The total of all diacylglycerols for each preparation of SALATRIM are reported at the bottom of Tables 4–7.

It is important that no heavy metals to be concentrated in the SALATRIM oils as a result of the manufacturing and refining process. In Table 9 we report the results of the inorganic analysis of the starting oils and the SALATRIM preparations. The results are compared to the analysis of a typical food grade corn oil. The values for heavy metals in SALATRIM are consistent with those found in corn oil and show no concentration of metals from the hydrogenated starting oils. The results provide assurance that the process of manufacture does not result in concentration of any metals.

Phytosterols are present in most vegetable oil preparations. In Table 10 the results of phytosterol analysis of SALATRIM preparations are reported. Sonntag (1979) reports the total phytosterol concentration of soybean oil, cottonseed oil, and rapeseed oil to be 0.15–0.38%, 0.26–0.31%, and 0.35–0.50%, respectively. It is possible that significant portions of the phytosterols are removed during the bleaching step. The phytosterol distributions within the various SALATRIM fats are similar to those reported by Padley et al. (1978). Our results reflect the effect of hydrogenation on the phytosterols. The hydrogenated starting oils and resulting SALATRIM preparations contain reduced derivatives of the normal phytosterols. Although some variability exists, it appears from the data in Table 10 that there is no significant concentration of any individual phytosterol. In all cases it can be seen that the SALATRIM preparations contain lower levels of total phytosterols than the parent oils. Reductions in total phytosterols are up to 40% of that present in the starting material.

Tocopherols contribute to the oxidative stability of vegetable oils and, like phytosterols, are present in most vegetable oil preparations. Tocopherols for the SALATRIM fats are shown in Table 11. The levels of tocopherols in SALATRIM preparations derived from soy and canola oils were essentially the same as levels found in the parent oils. This suggests that the refining process does not remove tocopherols from the oils.

A mass balance study was conducted to account for as much of the SALATRIM preparation products as possible. Table 12 contains the summary of the results from the analyses conducted on the SALATRIM samples in this study. The results generally account for over 90% of themass in all of the materials tested. Considering the relative standard deviation of all of the tests, this would seem to be excellent recovery. No unusual or unexpected materials were found as a result of the interesterification and refining of the material.

The SALATRIM family encompasses a broad spectrum of triacylglycerol mixtures. Mixtures are controlled by blending different short-chain fatty acid sources at various

Table 11. Tocopherols in SALATRIM Preparations (Milligrams per Kilogram)

SALATRIM/tocopherol	α	β	γ	δ	total
hydrogenated rapeseed oil	37.6 ± 0.6	<5	191.7 ± 3.3	9.8 ± 0.57	239.10
hydrogenated soy oil	109.3 ± 1.9	11.63 ± 0.2	572.0 ± 4.3	109.3 ± 2.4	883.23
hydrogenated cottonseed oil	294.7 ± 7.0	<5	292.0 ± 5.7	9.8 ± 0.5	596.50
4CA	43.4 ± 8.0	<5	153.0 ± 2.2	37.2 ± 0.4	233.60
23CA	42.1 ± 8.6	<5	162.5 ± 5.4	44.0 ± 1.4	248.60
23SO	101.2 ± 1.8	17.8 ± 1.5	572.7 ± 11.7	186.0 ± 6.4	877.70
234CA	33.1 ± 1.5	<5	153.0 ± 2.2	39.0 ± 5.0	225.10
234CS	128.7 ± 1.9	5.7 ± 4.0	268.3 ± 3.8	43.9 ± 5.0	451.60

[a] Data from Sonntag (1979).

Table 12. Summary of the Total Mass for Selected Members of the SALATRIM Family

SALATRIM	23CA	23SO	4CA	234CA	234CS
acylglycerols	90.44 ± 0.37	97.49 ± 0.32	96.85 ± 0.72	86.66 ± 0.45	88.27 ± 0.30
unsaponifiables	0.81 ± 0.16	0.20 ± 0.01	0.85 ± 0.05	0.43 ± 0.03	0.28 ± 0.03
free fatty acids	0.82 ± 0.03	0.36	0.22 ± 0.01	0.07 ± 0.01	0.19 ± 0.03
phytosterols	0.04	0.01	0.04	0.04	0.03
tocopherols	0.025	0.088	0.023	0.023	0.045
ash	0.004	0.002	0.002	0.002	0.003
total[a]	92.13	98.14	97.98	87.22	88.81

[a] Total of acylglycerols observed, free fatty acids, phytosterols, tocopherols.

Table 13. Composition (Weight Percent, Mean ± Standard Deviation) of Five Lots of SALATRIM before and after Distillation

	composition of undistilled SALATRIM in					
acylglycerol	lot A	lot B	lot C	lot D	lot E	overall mean ± SD
			Before Distillation			
acylpalmitoylglycerol	0.59 ± 0.03	0.53 ± 0.02	0.60 ± 0.01	0.67 ± 0.03	0.75 ± 0.02	0.63 ± 0.01
diacetylpalmitoylglycerol	6.92 ± 0.07	5.73 ± 0.03	6.46 ± 0.08	6.82 ± 0.03	6.96 ± 0.11	6.58 ± 0.03
acetylpropionylpalmitoylglycerol	1.56 ± 0.04	1.34 ± 0.02	1.45 ± 0.03	1.49 ± 0.02	1.51 ± 0.02	1.47 ± 0.01
acetylstearoylglycerol	1.70 ± 0.22	1.83 ± 0.08	2.02 ± 0.05	2.27 ± 0.08	2.61 ± 0.07	2.09 ± 0.07
diacetylstearoylglycerol	58.90 ± 1.14	57.46 ± 0.50	59.07 ± 0.67	59.68 ± 0.30	59.42 ± 0.10	58.91 ± 0.40
acetylpropionylglycerol	11.39 ± 0.07	11.40 ± 0.12	11.41 ± 0.15	11.31 ± 0.11	11.23 ± 0.08	11.35 ± 0.03
dipropionylglycerol	0.47 ± 0.02	0.47 ± 0.01	0.44 ± 0.02	0.45 ± 0.01	0.44 ± 0.03	0.45 ± 0.01
diacetylarachidoylglycerol	0.31 ± 0.01	0.33 ± 0.00	0.31 ± 0.01	0.32 ± 0.01	0.32 ± 0.01	0.32 ± 0.00
diacetylbehenoylglycerol	0.26 ± 0.02	0.30 ± 0.01	0.28 ± 0.01	0.28 ± 0.01	0.27 ± 0.01	0.28 ± 0.00
acetyldipalmitoylglycerol	0.31 ± 0.03	0.33 ± 0.03	0.31 ± 0.03	0.27 ± 0.03	0.27 ± 0.03	0.30 ± 0.00
acetylpalmitoylstearoylglycerol	2.26 ± 0.39	2.33 ± 0.03	2.10 ± 0.04	2.20 ± 0.39	2.66 ± 0.46	2.31 ± 0.21
propionylpalmitoylstearoylglycerol	0.48 ± 0.01	0.51 ± 0.01	0.48 ± 0.01	0.46 ± 0.01	0.46 ± 0.01	0.48 ± 0.00
acetyldistearoylglycerol	8.89 ± 1.12	10.41 ± 0.08	9.40 ± 0.09	9.15 ± 0.05	8.98 ± 0.04	9.37 ± 0.47
propionyldistearoylglycerol	1.40 ± 0.03	1.50 ± 0.03	1.40 ± 0	1.35 ± 0.03	1.34 ± 0.01	1.40 ± 0.01
acetylstearoylarachidoylglycerol	0.31 ± 0.05	0.38 ± 0.06	0.40 ± 0.10	0.32 ± 0.04	0.21 ± 0.16	0.32 ± 0.05
dipalmitoylstearoylglycerol	0.31 ± 0.15	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.06 ± 0.07
palmitoyldistearoylglycerol	0.77 ± 0.03	0.64 ± 0.03	0.53 ± 0.05	0.53 ± 0.02	0.55 ± 0.01	0.60 ± 0.01
tristearoylglycerol	1.31 ± 0.07	1.11 ± 0.06	0.87 ± 0.01	0.84 ± 0.03	0.87 ± 0.01	1.00 ± 0.03
total	98.12 ± 1.74	96.6 ± 0.84	97.55 ± 0.98	98.43 ± 0.67	98.86 ± 0.48	97.91 ± 0.48
total diglycerides	2.29 ± 0.23	2.36 ± 0.1	2.62 ± 0.06	2.95 ± 0.09	3.35 ± 0.08	2.71 ± 0.07
total triglycerides	95.84 ± 1.89	94.25 ± 0.81	94.92 ± 0.96	95.48 ± 0.6	95.5 ± 0.49	95.20 ± 0.56
			After Distillation			
acylpalmitoylglycerol	0.65 ± 0.01	0.58 ± 0.02	0.64 ± 0.02	0.69 ± 0.04	0.78 ± 0.02	0.67 ± 0.01
diacetylpalmitoylglycerol	7.57 ± 0.07	6.42 ± 0.08	7.12 ± 0.08	7.07 ± 0.14	7.48 ± 0.20	7.13 ± 0.06
acetylproypionylpalmitoylglycerol	1.69 ± 0.02	1.49 ± 0.03	1.61 ± 0.02	1.55 ± 0.03	1.63 ± 0.03	1.59 ± 0.01
acetylstearoylglycerol	2.01 ± 0.07	1.90 ± 0.30	2.28 ± 0.08	2.56 ± 0.11	2.82 ± 0.25	2.31 ± 0.11
diacetylstearoylglycerol	66.86 ± 0.54	66.26 ± 1.07	67.79 ± 0.81	67.10 ± 1.69	67.93 ± 2.13	67.19 ± 0.65
acetylpropionylglycerol	12.91 ± 0.10	12.95 ± 0.19	13.10 ± 0.14	12.81 ± 0.30	12.93 ± 0.40	12.94 ± 0.12
dipropionylglycerol	0.52 ± 0.01	0.52 ± 0.02	0.50 ± 0.02	0.48 ± 0.02	0.48 ± 0.03	0.50 ± 0.01
diacetylarachidoylglycerol	0.35 ± 0.01	0.38 ± 0.01	0.37 ± 0.01	0.36 ± 0.02	0.36 ± 0.01	0.36 ± 0.00
diacetylbehenoylglycerol	0.30 ± 0.01	0.35 ± 0.03	0.33 ± 0.01	0.32 ± 0.05	0.31 ± 0.02	0.32 ± 0.02
acetyldipalmitoylglycerol	0.28 ± 0.02	0.29 ± 0.04	0.29 ± 0.02	0.28 ± 0.03	0.26 ± 0.03	0.28 ± 0.01
acetylpalmitoylstearoylglycerol	1.87 ± 0.36	2.21 ± 0.45	1.57 ± 0.28	1.77 ± 0.32	1.71 ± 0.34	1.83 ± 0.06
propionylpalmitoylstearoylglycerol	0.36 ± 0.01	0.40 ± 0.02	0.35 ± 0.01	0.38 ± 0.01	0.34 ± 0.01	0.37 ± 0.00
acetyldiastearoylglycerol	4.62 ± 0.57	5.92 ± 0.82	5.02 ± 0.08	5.46 ± 0.61	4.57 ± 0.17	5.12 ± 0.31
propionyldistearoylglycerol	0.81 ± 0.01	0.94 ± 0.06	0.81 ± 0.04	0.90 ± 0.02	0.75 ± 0.03	0.84 ± 0.02
total	100.80 ± 1.44	100.61 ± 1.93	101.78 ± 1.10	101.72 ± 2.01	102.37 ± 3.01	101.46 ± 0.72
total diglycerides	2.65 ± 0.08	2.49 ± 0.31	2.91 ± 0.08	3.24 ± 0.13	3.60 ± 0.24	2.98 ± 0.10
total triglycerides	98.15 ± 1.45	98.12 ± 2.04	98.87 ± 1.07	98.47 ± 1.98	98.77 ± 3.12	98.48 ± 0.77

ratios for interesterification. Further refinement of the mixtures can be obtained by adding a step of distillation in a wiped film evaporator which is followed by an additional deodorization step. It is important to assess the amount of variability introduced by the additional step in the process. In Table 13 we report the compositional analysis of five lots of SALATRIM before distillation and in the composition after distillation. From the data it can be seen that the recoveries are 97.4% to nearly 100% as a result of the distillation process. It is also important

to note that in the process the triacylglycerol distribution is shifted. The tri-long triacylglycerols such as tristearin are eliminated from the distilled products and the di-long triacylglycerols are reduced. The data in the table support the reproducibility of the synthetic process (undistilled) and the reproducibility of the distillation step.

CONCLUSIONS

In SALATRIM, a low-calorie fat, over 96% of the measurable components are acylglycerols. The results confirm that the composition of SALATRIM materials can be accurately predicted by employing a randomly esterified reaction model. The triacylglycerol distribution in the SALATRIM mixtures reflects the predicted distribution based on the fatty acid profile of the starting materials. As a result, we have demonstrated that triacylglycerols constitute approximately 90% of SALATRIM preparations. All SALATRIM family members contain similar structures including SSL, SLS, LLS, and LSL, where S and L represent short- and long-chain fatty acids, respectively. The analysis of non-triacylglycerol components demonstrates that tocopherols survive the process, that phytosterols are reduced, and that there is no evidence for concentration of heavy metals. No unexpected or unusual organic compounds were found during the analysis. We therefore conclude that SALATRIM materials are similar to other edible oils, except for the unique fatty acid distribution which makes SALATRIM lower in calories.

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