

action of some of the detergent solutions has also been determined.

The results of these detergent tests on rosin, fatty acid and fatty acid-rosin soaps indicated the following: (1) Rosin soaps made from different gum rosins, produced from longleaf and slash pine gums, have equal detergent action; (2) The presence of soaps of oxidized rosin acids has no effect on detergent action of the rosin soap; (3) The detergent action of soaps made from pyroabietic acid, abietic acid and hydrogenated rosin parallels their ability to lower the surface tension of water and the amount of hydrogen present in the rosin acids; (4) The addition of builders that increase the

alkalinity of the rosin soap solution improves the detergent action of the solution; (5) The blending of rosin soap with tallow soap improves the detergent action of the tallow soap in solutions having a soap concentration of 0.25 percent or more; (6) Temperature affects the detergent action of rosin and coconut oil soaps more than tallow soap.

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## Estimation of Saturated Glycerides in Shortenings and Margarines

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### INTRODUCTION

Component glyceride data for many fats have been obtained by utilizing oxidation, crystallization and hydrogenation methods. Outstanding in this phase of fat study has been the work of Hilditch and co-workers. Their concept divides fats into two classes.

The first includes seed fats and marine animal fats whose glycerides are characterized by "even distribution" of fat acid radicals within the glyceride molecules.

Further, this concept applies to the hydrogenation of these oils; the saturation of glyceride molecules having unsaturated linkages to fully saturated follows in fairly well defined progressive stages.

The second class of fats, which includes the larger land animal fats, are characterized by glycerides of "uneven distribution" of the fat acid radicals. The amount of fully saturated glycerides present is much greater than in the seed fats with the same ratio of saturated to unsaturated fat acids.

Component fat acid data has not proved to be an enlightening manner by which to demonstrate the quantity and type of saturated glyceride present in a shortening. In this study, an attempt has been made to devise a method which will identify and quantitatively express predominantly saturated glyceride content. The study has included fat mixtures containing vegetable stearin (i.e. hydrogenated soybean oils) of two different degrees of saturation and mixtures containing animal stearin. In brief, the method employs the principle involved in the Bomer Number Test. Gravimetric and iodine number data are obtained for the solid fat fractionated from acetone-fat solution at 30°C.

### METHOD

In a graduated centrifuge tube, 20 gms. of melted fat (40-45°C.) is dissolved in a quantity of acetone sufficient to make a total volume of 100 cc. at 30°C.

The fat-acetone mixture is held in a 30° ± 0.2°C. water bath for 18 hours. This period is sufficient to permit the crystallizing of the predominantly saturated glycerides.

The precipitated glycerides are filtered into a tared Gooch crucible. The fat crystals are washed with small portions of acetone, 30°C. until 100 cc. of the solvent has been used in this manner. The tared crucible is

held in a vacuum dessicator until a constant weight is obtained.

The iodine number determinations, closed tube and Wiley melting point determinations were made in accordance with the Fat Analysis Committee Methods. The Bomer Numbers were calculated in the usual manner.

### RESULTS AND DISCUSSION

The data given in Table No. 1 demonstrated that the predominantly saturated glycerides of cottonseed oil, coconut oil and partially hydrogenated cottonseed oil are not precipitated from acetone solution, 30°C.

The absence of precipitated crystals as indicated above led to investigating the solubility of the three stearins in acetone, 30°C. Table No. 2 shows that predominantly saturated glycerides are soluble in acetone, 30°C.; the extent of solubility is dependent upon the degree of saturation of the glyceride.

From Table No. 3 it is seen that the predominantly saturated glycerides are crystallized from fat-acetone solutions at 30°C. The weight of solid fat recovered for fat mixtures containing 2.19 Iodine number soya stearin is greater than theoretical value for the stearin content of the fat mixture. This is believed to be due to occluding action which may occur during the crystallization period. This, and the low solubility in acetone of the 2.19 Iodine number stearin favor a greater crystallization of predominantly saturated glycerides. Undoubtedly the solubility of the other stearins studied greatly affect the quantity of solid fat which is crystallized from acetone solutions.

Further, the data indicate that oleo stearin has appreciably less percentage of saturated glycerides than either of the two soya stearins.

The closed melting point tube data for fat mixtures with 8% soya stearin Iodine number 20.94 and those with 8% oleo stearin when compared with the data for the corresponding stearins show a preferential solubility of the more unsaturated glycerides in acetone; this separation taking place during the crystallization period.

### SUMMARY

A method has been used which has been successful in estimating the quantity of predominantly saturated glyceride present in a fat mixture such as a shortening or margarine oil base.

TABLE No. I.

Crystallization of Saturated Glycerides from Acetone at 30°C.

Fat	I <sub>2</sub> #	% Crystallization
Cottonseed Oil	.....	0.0
Coconut Oil	.....	0.0
Partially Hydrogenated Cottonseed Oil	66.7	0.0

TABLE No. II.

Solubility of Stearin in Acetone at 30° C.

Stearin	I <sub>2</sub> #	Gms Added to 100 cc	Gms Soluble in 100 cc	% Solubility
Soya	2.19	1.5334 Gm	0.2310 Gm	15.05
Soya	20.94	1.6276	0.6822	42.00
Oleo	23.33	1.6257	1.1282	69.30

TABLE No. III.—Data for Fat Crystallized from Acetone at 30° C.

Fat Mixture PH C/S*	Stearin	Wgt. Stearin in 20 gms Fat	I <sub>2</sub> #	% Solid Fat Recovered	Wgt. Solid Fat Recovered	I <sub>2</sub> # Solid Fat Recovered	M.Pt. Glycerides	M.Pt. Fat Acids	Bomer Number**	Wiley M.Pt. Solid Fat Recovered
100.0%	0.0%	0.0000 gm	0.00	0.00%	0.0000 gm	0.0	.....	.....	.....	.....
99.0% Soya	1.0%	0.2056 gm	.....	1.82%	0.3654 gm	11.04	.....	.....	.....	.....
97.0	3.0	0.6009	.....	5.37	1.0754	9.80	.....	.....	.....	.....
95.0	5.0	1.0048	.....	7.21	1.4435	7.59	67.0	63.7	73.6	66.2
92.0	8.0	1.6070	.....	10.74	2.1492	6.03	68.0	64.3	75.4	66.9
0.0	100.0	.....	2.19	.....	.....	.....	68.9	.....	.....	67.8
99.0% Soya	1.0%	0.2047 gm	.....	0.58%	0.1166 gm	11.60	.....	.....	.....	.....
97.0	3.0	0.6067	.....	3.13	0.6264	10.38	.....	.....	.....	.....
95.0	5.0	1.0087	.....	5.17	1.0352	9.98	66.0	62.4	73.2	.....
92.0	8.0	1.6041	.....	6.82	1.3643	9.00	66.9	63.0	74.7	.....
0.0	100.0	.....	20.94	.....	.....	.....	64.5	.....	.....	62.9
99.0% Oleo	1.0%	0.2063 gm	.....	0.01%	0.0034 gm	.....	.....	.....	.....	.....
97.0	3.0	0.6291	.....	0.16	0.0320	10.07	.....	.....	.....	.....
95.0	5.0	1.0102	.....	0.38	0.0777	9.16	.....	.....	.....	.....
92.0	8.0	1.6119	.....	2.26	0.4538	8.90	62.0	58.5	69.0	.....
0.0	100.0	.....	23.33	.....	.....	.....	56.0	.....	.....	55.9

\*Partially Hydrogenated Cottonseed Oil, I<sub>2</sub># 66.7.

\*\*Bomer No. = M.Pt. Glycerides + 2 (M.Pt. Glycerides - M.Pt. Fat Acids)

# Continuous Deodorization—Two Years Later

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## 1. INTRODUCTION

Two years ago we had the pleasure of presenting to the American Oil Chemists' Society a description of the process of continuous deodorization of edible oils, which was developed over a period of several years, and was represented at that time (in 1938) by the successful operation of one 5000 lb. per hour capacity unit and one 1200 lb. per hour unit shown in Figure No. 1.

Developments since that time have confirmed the economies and practicability of the continuous deodorization process, and the very marked saving in steam, fuel and condensing water requirements reflected in lower operating cost as compared to the usual batch method of handling.

## 2. OPERATION CYCLE OF CONTINUOUS DEODORIZATION VERSUS BATCH DEODORIZATION

Primarily, the process of deodorization consists of distilling out of the oil various volatile constituents which are undesirable, including residual free-fatty acids.

The release of volatile matter from the non-volatile glycerides is accomplished in continuous deodorization, just as in batch deodorization, by the combined application of heat and vacuum together with the introduction of injection steam, which, as in other distilling operations, reduces the partial pressure of the volatile materials, allowing them to vaporize more freely.

**BATCH OPERATION**—In batch deodorization this injection steam also serves as a method of agitation of the oil. The undeodorized oil is placed in a vessel under vacuum and is heated by steam, Dowtherm vapor, or other means, through closed coils in the deodorizer, and when a certain minimum temperature is reached injection steam is introduced over the remaining period

of the deodorizing cycle to agitate and help distill out the volatile material. The finished deodorized oil is then cooled before allowing it to come in contact with air.

**CONTINUOUS OPERATION**—Continuous deodorization involves in general the same cycle of operations. In the first continuous installation the oil was partially heated before being subjected to the high vacuum, and although the stripping of volatile materials was satisfactorily accomplished and a good commercial product obtained, certain advantages were indicated in the process of placing the oil under high vacuum before and during the heating operation. This arrangement is incorporated in the latest installations.

Deodorization is accomplished in the continuous unit with only a portion of the injection steam required for batch operation because of the progressive and counter-current contacting of the oil with the stripping steam. The heated oil passes successively downward over a series of contacting trays. The stripping steam enters at the bottom tray, so that the pure deodorized oil is contacted only by the incoming pure injection steam, free of volatiles and fatty acids.

The contact tray consists of a series of nozzles through which the steam flows upward; each nozzle being fitted with a cap slotted at the lower edge, which directs the steam downward inside the cap and sideways through it, where it then bubbles through the relatively shallow layer of oil. These caps and trays are illustrated in Figure No. 2.

This method of steam and oil distribution gives very intimate contact and is much more efficient than steam blowing upward through a 7 or 8 foot depth of oil,—as is customary under batch operation.

The oil on the first or top tray flows across the tray