

meats, and cottonseed meal have been studied in both vacuum and forced draft ovens at 101° C. and in forced draft ovens at 130° C. The values obtained have been compared with values for moisture obtained by the Karl Fischer method. The amount of moisture in the sample had very little or no influence on the length of time required to attain a constant dehydration level at the times and temperatures studied. The rate-of-loss curves indicated that it is not feasible to reduce the drying times required by the official methods for determination of moisture in cottonseed materials.

With whole cottonseed 12 to 16 hours' drying at 101° in the forced draft oven as required by the present official method was found satisfactory. With crimped seed, however, 8 hours' drying in the 101°-forced draft oven was required to attain an equivalent degree of dehydration. Values approximately 0.2% lower were obtained in the 5 hours specified in the official method. For fumed and ground cottonseed, whole or ground meats, and cottonseed meal, the oven loss-time curves indicated that 5 hours at 101° C. are necessary to reduce the rate of loss in weight to a minimum. This time interval, confirmed by the data obtained by the Karl Fischer method, is somewhat longer than the time specified in the official method, namely, 2 hours for fumed and ground cottonseed and 3 hours for the meal and meats.

On heating cottonseed materials in forced draft ovens, larger losses in weight were obtained at 130° than at 101° C.

The results obtained indicated that while at 101° C. both the vacuum and the forced draft ovens reduce cottonseed materials to equivalent degrees of dehydration, the times required are different.

Any changes which appear to be indicated in the official methods by the present work should be carefully studied and checked by collaborative investigations, taking into account the wide range of samples and the variations of individual workers in the application of recommended procedures.

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#### REFERENCES

1. American Oil Chemists' Society, Official and Tentative Methods—Chicago, The Society (1944).
2. Jamieson, G. S., and McKinney, R. S., *Oil and Fat Industries* 6 (7), 11 (1929); 7, 291 (1930).
3. Moisture Committee, American Oil Chemists' Society, *Oil and Fat Industries* 6 (5), 16 (1929); 7, 191 (1930); 8, 342 (1931). *Oil and Soap* 13, 206 (1936); 14, 211 (1937); 15, 211 (1938); 16, 142 (1939).
4. Hoffpauir, C. L., *Oil and Soap* 22, 283 (1945).
5. Mehlenbacher, V. C., Private Communication.
6. Krober, O. A., and Collins, F. I., *Oil and Soap* 21, 1 (1944).
7. Soybean Analysis Committee, American Oil Chemists' Society, *Oil and Soap* 18, 132 (1941).

## Report of the Soybean Analysis Committee 1945-46

**D**URING the past year no collaborative work on soybeans has been carried out by this committee; however R. T. Doughtie, Jr., agent of the Commodity Credit Corporation, has conducted an extensive test of the present official AOCS procedure for determining the oil content of soybeans. Results of this test, participated in by 38 of the leading soybean chemists of the country, have been made available through the courtesy of Mr. Doughtie. Two series of 20 samples were studied with results furnished by all 38 chemists on each of the 40 samples.

In the first series of 20 samples, standard deviation averaged 0.28, with the highest standard deviation 0.46. The sample giving this extremely high standard deviation contained an excessive amount of dockage (5.2%) as compared with all of the remaining samples. The difficulties found by the chemists on this sample were attributed to this cause, and the analysts were cautioned to obtain proper screens and follow the official procedure for dockage removal more closely.

On the second series of 20 samples the standard deviation averaged 0.22 (a distinct improvement) and the highest standard deviation was 0.29. This value of 0.22 may appear somewhat high but represents the agreement between results when a large group of chemists (some very experienced and some rather inexperienced) uses the present official AOCS method for this determination. It should be remembered that on any given sample two-thirds of the

results of individual chemists will lie within plus or minus one standard deviation of the general average. In an effort to estimate the precision of the method when used by the most experienced workers, the average standard deviation was calculated for the second series of 20 samples using the 19 best chemists (half of total). This average was found to be only 0.14, and this improvement shows that the method of analysis is still responsive to expert treatment.

During the year, in addition to the above series of tests, Mr. Doughtie and his collaborators also studied the effect of regrinding and the increase of sample size from 2 to 5 grams. All analysts would like, if possible, to eliminate the regrinding procedure, and many workers feel that increased accuracy would result from the use of 5-gram samples. The results obtained in Mr. Doughtie's study were too inconclusive to suggest changes at this time.

It should also be noted that during the past year there has been more general satisfaction with this method of analysis, and more confidence in it, than in the past. This is reflected by fewer variations in the check analyses performed by CCC and by fewer appeal analyses requested both by the processors and CCC.

At the Northern Regional Research Laboratory studies on the grinding of soybeans with a hammer-type of mill have been carried out. This mill shows sufficient promise so that it will be submitted to a

number of other laboratories for test under their operating conditions.

Since the official AOCS soybean analysis methods developed by this committee are now used satisfactorily in marketing the entire soybean crop for processing, this committee has completed its purpose. Any further modifications and study of the method

may well be undertaken by the recently organized Seed and Meal Analysis Committee as one of its functions.

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## Interesterification Reactions of Triglycerides<sup>1</sup>

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IT was suggested in a recent publication from this laboratory (10) that interesterification might possibly be one of the mechanisms involved in the digestion and metabolism of fats. Before an investigation of such a possibility it has been deemed essential that we investigate the mechanism of the interesterification reaction *in vitro*. This work has been incorporated into a general program of study of the glyceride structure of natural fats and their modification by chemical and physical means. In the present paper we shall attempt to show the probable glyceride structure produced in fats by interesterification.

Intesterification refers to the free exchange and redistribution of component fatty acids among the various triglycerides. Such an interchange has been known to occur under certain conditions of chemical and physical treatment. The process was first described in 1924 in a British patent by Van Loon (12), and since that time several patents have covered catalysts and conditions favoring the rearrangements (6, 7, 13).

It was deduced in our preliminary discussions that the end product of interesterification would probably be constituted according to the principles of random distribution. During the progress of this work a reference book by A. E. Bailey appeared, in which this same deduction was made without supporting experimental evidence being presented (1). It would be anticipated from chemical kinetics that when glycerol is esterified with a mixture of fatty acids of equal reactivity, the acids would distribute themselves in a random fashion among the glycerol molecules. Such an hypothesis is difficult to establish experimentally. Hilditch and his co-workers presented the first experimental evidence in support of random distribution when they showed that the structures of synthetic mixed glycerides corresponded to those of natural animal fats (3), and that in the latter the curve showing the relationship of trisaturated glycerides (Y) to mol percent of saturated acids (X) approximated that represented by the equation:

$$Y = K (X)^3 \quad (2)$$

This principle has been further elucidated by Dean (5) and more recently Longenecker and his co-workers (9), who evolved the following set of equations

(in which we shall use as an example a fat containing only palmitic, stearic, and oleic acids):

Percent tripalmitin =

$$\frac{P}{100} \times \frac{P}{100} \times \frac{P}{100} \times 100$$

Percent steardipalmitin =

$$\frac{P}{100} \times \frac{P}{100} \times \frac{S}{100} \times 3(100)$$

Percent oleostearopalmitin =

$$\frac{P}{100} \times \frac{S}{100} \times \frac{O}{100} \times 6(100)$$

P, S, and O represent the molar percentages of the respective fatty acids in the mixture. The equations may be applied to any combinations of acids in mixtures of all degrees of complexity.

The only readily available method for establishing in part the random distribution of a fat is an analysis for trisaturated glycerides. This method has been applied in our investigation of the end products of interesterification. Thus, if a mixture of known saturated acid content is interesterified, its anticipated trisaturated glyceride composition may be calculated according to the above equations. If the experimentally determined amounts of trisaturated glycerides are in agreement over a range of values, one has rather good circumstantial evidence in favor of random distribution. In order to have a more quantitative measurement we have used not only mixtures of natural and hydrogenated fats, but also mixtures of known proportions of a synthetic saturated triglyceride (tripalmitin) with a synthetic unsaturated triglyceride (triolein).

### Experimental

#### 1. Preparation of Synthetic Triglycerides.

Methyl palmitate was prepared in a high degree of purity by a fractional distillation of the methyl esters of a commercial palmitic acid<sup>2</sup> through a Podbielniak Hyper-Cal column at a reflux ratio of 2.5:1 (11). This ester after conversion to the free acid exhibited a melting point of 62.5-63.0° C.

Tripalmitin was prepared by heating glycerol with 7% excess palmitic acid in the presence of 0.3% SnCl<sub>2</sub>·2H<sub>2</sub>O catalyst at 200° C. and 20 mm. pressure for 3 hours. The amount of water which was produced indicated that the reaction was more than 99%

<sup>1</sup>Presented before the fall meeting of the American Oil Chemists' Society, Nov. 7-9, 1945, in Chicago.

<sup>2</sup>Neo-Fat 1-56.