

quite significant at the 10% level. These findings indicate that supplies of Kimpak and preparations of catalyst need not be problems in the performance of the rapid method. The variation due to bombs represents a random variable and is included in the overall precision estimate for the method.

Since the effect of varying the amount of catalyst in the region of 25 ppm was small, possible errors associated with addition of catalyst will not be of serious magnitude in the routine performance of the method.

It should be noted that while the standard deviation for samples of longer stability is twice that of the shorter stability samples, the coefficient of variation is about half.

Results obtained by application of the AOM method, the bomb method without catalyst and the bomb method with catalyst to commercially available fats and shortenings are tabulated in Table II and shown graphically in Figure 2.

It is evident from these data that an overall general correlation exists between the AOM and bomb methods, but that it is not possible to relate bomb values directly to AOM. This is not surprising when one considers the complex nature of the makeup of shortenings, not only with respect to type and extent of modification of the fatty portions, but also because of the varying response of such formulations to the array of possible antioxidant combinations.

All of the methods for measuring stability are basically empirical, so that the ultimate selection is made in light of the specific application. As previously suggested, some of the distinction in stability normally required for research applications can logically be sacrificed in control situations where speed is a primary consideration.

The rapid oxygen bomb method has the following advantages for control purpose:

- 1) The time required for evaluating the stability of any shortening can be adjusted to between 1 and 6 hr by proper selection of the level of catalyst.
- 2) Samples can be tested at different accelerated rates in the same bath at the same time. This is not possible when acceleration of the test is accomplished by increasing the temperature.
- 3) Tests on samples of different stability can be completed in about the same time in the same equipment by proper selection of the amount of catalyst to be used with the different types.
- 4) The equipment has been standardized by the A.S.T.M. and can be purchased complete from supply houses and can be used without modification.

REFERENCES

1. Gearhart, W. M., B. N. Stuckey, and J. J. Austin, *JAACS*, **34** 427, (1957).
2. AOCS Official and Tentative Method, Cd 12-57.
3. Pohle, W. D., R. L. Gregory, and J. R. Taylor, *JAACS*, **39** 226, (1962).

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Report on Activities of Industrial Oils and Derivatives Committee, 1963

FOUR OF THE Industrial Oils & Derivatives Subcommittees which met during the Society meetings in Atlanta, April 22-24, 1963, have been very active since the last meeting of the Society. Below is a brief summary of these activities, by K. E. Holt, Chairman.

Epoxidized Oil Subcommittee, Dave Barlow, Chairman

Two modifications to AOCS Tentative Method Cd 9-57, Oxirane Oxygen, were submitted to the Epoxidized Oil Subcommittee for consideration. The first involved an alternate method for the preparation of the HBr acetic acid reagent; the second modification proposed the addition of an excess of HBr - acetic acid reagent to the sample and back titrating for the unreacted HBr.

A study was set up, with eight collaborators participating, to study the effect of these modifications to method Cd 9-57. Samples of epoxy soybean oil, 2-ethyl hexyl epoxytallate and glycidyl stearate were selected for this study. The AOCS statistical design as outlined in Method M 1-59 was followed. All of the collaborators completed their part of the program and the results were studied at the meeting of the Subcommittee in Atlanta. It was concluded that modifying the method of preparation of the HBr - acetic acid reagent did not affect the results obtained by the method. The addition of an excess of HBr - acetic acid reagent, allowing the reaction to stand for

30 min and back titrating the excess HBr, produced results that were slightly higher than those produced by the present method. The Subcommittee was in agreement that the higher results were due to hydrolysis or side reactions with the excess HBr and an accurate measurement of true oxirane not being made.

Some minor modifications were recommended for method Cd 9-57 which the Subcommittee believes will improve the precision, but not change the numerical values obtained by the method. These recommendations were passed on to the Uniform Methods Committee and will appear in the next issue of the method.

Requests have been received for a method that will measure hydroxyl value on epoxidized oils. The subcommittee will undertake to review the current methods now being used and solicit ideas or methods from AOCS members.

The Drying Oils Subcommittee, Don Bolley, Chairman

The Drying Oils Subcommittee completed a study of the Color and Viscosity Methods and are recommending revisions of these methods to the Uniform Methods Committee.

Method Ka 3-58, Gardner Color, is revised to make the primary standards from glass with liquids as secondary standards. Glass standards presented to the Subcommittee by two different manufacturers were checked out and found to be satisfactory. Glass standards have the advantage of permanency which

has been lacking in the liquid standards. It is believed that a large percentage of the liquid standards that have been in use for a few years will not meet the chromaticity coordinates set forth in Ka 3-58 and should be replaced if accurate color readings are required.

Revision of Method Ka 6-59, Viscosity by Bubble Time, to use numerically marked comparator tubes as standards replacing the Gardner-Holdt letter series has been completed and sent on to the Uniform Methods Committee for final approval and inclusion in the Methods Book.

The Subcommittee is attempting to write a method on "Appearance." Ed Handschumaker presented a method on "Determination of Clarity by Total Nephelos," which employs a nephelometer and would give a quantitative measurement. The Subcommittee decided a qualitative method also is desirable and solicits suggestions.

Polymerized Fatty Acids, Gerald Wilson, Chairman

The Subcommittee has completed collaborative tests on polymerized fatty acids using the following AOCS Methods: L 2b-57, Moisture, Modified Karl Fisher Reagent; L 3a-57, Acid Value; L 4a-57, Unsaponifiable Matter; L 7a-57, Saponification Value; and Ka 3-58, Gardner Color. It was found that all of the methods were applicable to polymerized fatty acids and their adoption for use in the analysis of polymerized fatty acids is recommended to the Uniform Methods Committee.

The Subcommittee decided that none of the common methods of determining unsaturation such as halide addition, high pressure hydrogenation or ozone absorption will work on polymerized fatty acids. The Subcommittee is open to suggestions from members of AOCS and will consider any method proposed if it is accompanied by supporting data.

The following definition was proposed for polymerized fatty acids: "polycarboxylic polymerized fatty acids in essentially free acid form."

Fatty Nitrogen Subcommittee, Gerald Wilson, Chairman

Collaborative work on fatty amido amines showed the following methods in the "N" section of AOCS Methods to be applicable to fatty amido amines: Gardner Color, Moisture by Karl Fisher, Total Amine Value and Iodine Value. It was recommended that fatty amido amines be included in the definition and introduction to the "N" section and that the precisions obtained be included as a part of the methods. Collaborative work is continuing on other methods applicable to fatty amido amines and, also, methodology on dimethyl fatty amines, fatty diamine and fatty amine.

Consolidation of Methods

At the present time, each Subcommittee of the Industrial Oils and Derivatives Committee has its own section of methods: that is, Drying Oils—Section K, Fatty Nitrogen—Section N, Commercial Fatty Acids—Section L, etc. As new Subcommittees are added, such as Polymerized Fatty Acids, the establishment of a new section will be required if the same procedure is to be continued. The Industrial Oils & Derivatives Committee recommend to the Uniform Methods Committee that all of the methods falling under the jurisdiction of the Industrial Oils & Derivatives Committee be consolidated into one section. This will eliminate much duplication in methods that are common to many or all of these compounds such as Color, Iodine Value, Moisture, etc. It is, also, recommended that an introduction be written for each of the classes of compounds to be tested similar to that now appearing in Section N Fatty Nitrogen Compounds. This would give the analyst an idea regarding test methods to be used and assist him in interpreting results obtained.

The Uniform Methods Committee gave tentative approval to this recommendation and requested that a format of introductions and methods be written and submitted prior to the fall meeting of AOCS.

Continuous Deglycerination of Monoglycerides

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Abstract

A relationship was established between the smoke point of a superglycerinated shortening and free glycerol content. A maximum free glycerol content of 0.02% was established for smoke points of 340F or better.

Based on a 6% usage in emulsifier type shortening, commercial monoglycerides should have 0.30% glycerol or less to meet a desirable smoke point. A 2-stage extraction with 5% NaCl solution reduced free glycerol from 5.51% to 0.14%.

A continuous process was developed for removing glycerol from commercial monoglyceride preparations with a thin-film evaporator. Operation at 340F and 6 mm Hg absolute pressure with stripping steam reduced glycerol content to 0.3%. Analysis showed substantially no monoglyceride reversion under process conditions, and considerable improvement in flavor and odor was obtained.

Introduction

GENERAL PURPOSE SHORTENINGS have been and are used in large tonnage by institutions, bakeshops, and housewives for baking. One of the characteristics of good shortenings for this purpose has been a high smoke point. Within the past 30 years we have seen the considerable growth of special all-hydrogenated shortenings with superior emulsifying properties which allow the baker to use a higher ratio of sugar to flour and still obtain light and full-volumed cakes. These shortenings contain more combined glycerol than ordinary fats and oils as a result of adding 6-8% of mono- and diglyceride preparations, and are known in the trade as "superglycerinated," or "emulsifier type" or "high-ratio." They are very popular in cakes and pies and are used substantially by the average household as well as commercial bakeries.

Superglycerinated shortenings are approximately similar to ordinary all-hydrogenated shortenings in