

Several samples of baked white cake containing SMS were analyzed. The results obtained are listed in Table II.

Tables III and IV list the results obtained by the analysis of yellow and chocolate cake mixes, respectively.

The analysis of blank white cake and blank yellow and chocolate cake mixes showed the absence of apparent SMS, indicating that there was no interference by the other constituents in the cake.

The time required for the preparation of the polyol solution is approx 3-4 days. Assay by gas chromatography requires only a few hours additional time. Paper chromatography, on the other hand, requires 2-3 additional days of elapsed time for completion.

In addition, while the paper chromatographic technique appears straight forward, one has to develop the skill and patience to perform the analyses. After this phase of familiarization is passed, an operator may perform satisfactory analyses using the paper chromatographic technique.

#### ACKNOWLEDGMENT

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## A Study of Methods for Evaluation of the Stability of Fats and Shortenings

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### Abstract

The data from several laboratory tests [Active Oxygen Method (AOM), modified ASTM bomb, modified ASTM bomb with catalyst added to the fat, a manometric method and an accelerated oven test] were compared with data from a storage test at 85F evaluated organoleptically at selected intervals. Tests were carried out on lard, hydrogenated vegetable oil and tallow, with and without added monoglyceride, and with and without selected antioxidants. A comparison of the data from the laboratory tests with that from the storage tests indicated: 1) that the different types of fats behave differently, 2) the laboratory tests cannot be used as an index of shelf stability except for a given type of formulation of fat for which the relationship between the laboratory test and the shelf stability is known, 3) the modified ASTM bomb method is the most reliable for estimating shelf life, and 4) the modified ASTM bomb method with catalyst is not as precise as the bomb method, but may be used for control purposes where some accuracy may be sacrificed in the interest of shorter time.

### Introduction

ALMOST ALL SHORTENINGS are tested for keeping qualities as a part of quality control programs, or to be certain they meet a specification under which they are sold. The purpose of these tests is to assure the producer and the customer that the product has a satisfactory shelf life and will not develop an off flavor during the period that it will remain in the plant, store or home before it is ultimately used. The relationship between the shelf life and the laboratory keeping tests is often questionable, but little or no sound data have been available to throw light on this situation.

The purpose of this investigation was to obtain more

detailed information on the relationship between shelf stability of various fats and the laboratory tests customarily used to predict stability. The time a product can remain in storage, in a store or a home, without the development of objectionable flavors is the property that storage tests attempt to duplicate and laboratory tests to predict. The time required for a sample in storage to develop a selected degree of off flavor will be taken as an index of the stability of the sample. This will be the value with which the results by other methods will be compared in order to select the laboratory test or tests that most accurately indicate the stability of fat or shortening.

### Experiment and Results

The samples prepared for this investigation were made using three types of fat, (lard, hydrogenated vegetable oil and tallow) with and without added monoglyceride and various antioxidants as indicated below:

Code	Type	Mono-glycerides	0.01% BHA, 0.01% BIT	0.01% BHA, 0.01% propyl gallate	0.01% BHA, 0.0025% NDGA	0.01% BHA
L-O-O	Lard	None				
L-O-A	Lard	None	X			
L-O-B	Lard	None		X		
L-O-C	Lard	None			X	
L-O-D	Lard	None				X
L-M-O	Lard	5%				
L-M-A	Lard	5%	X			
L-M-B	Lard	5%		X		
L-M-C	Lard	5%			X	
L-M-D	Lard	5%				X

Samples with the same combinations of monoglycerides and antioxidants were prepared using hydrogenated vegetable oil (V-O-O) and tallow (T-O-O).

These samples were subjected to the following laboratory tests for evaluating stability: active oxygen method (1), a manometric method (2), modified ASTM bomb (3), a modified ASTM bomb with catalyst added to the fat (4) and an oven test at 140F in which the 8-oz samples were held at 140F in a jar

<sup>1</sup> Presented at the AOCS Meeting in Minneapolis, 1963.

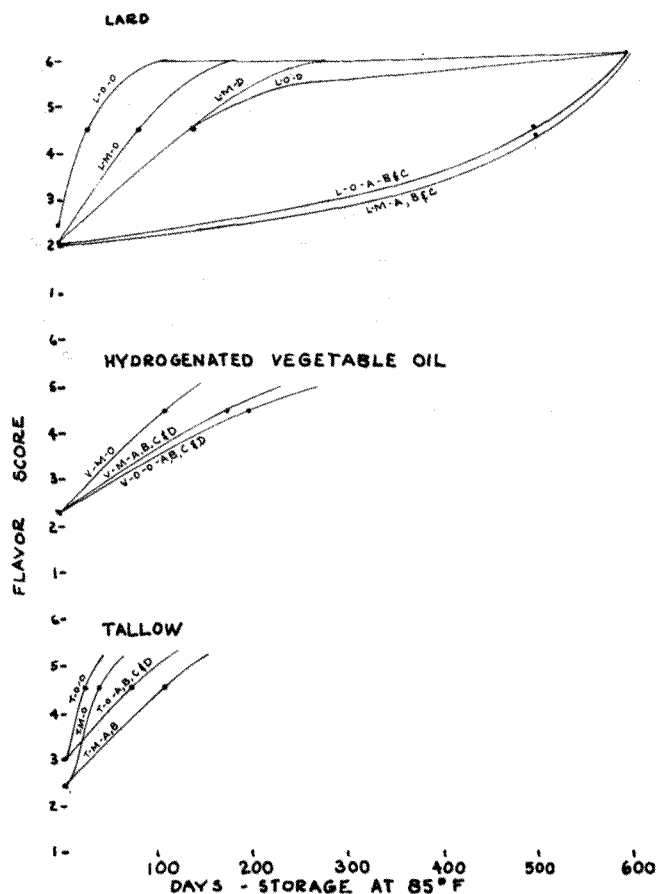


FIG. 1. Relationship between flavor score and days in storage for different types of fat and formulations.

with the lid on loosely and then evaluated for flavor by a panel of 6-8 judges at selected intervals. The flavor was scored as given below:

Score	Off flavor
1.....	None
2.....	Questionable
3.....	Barely detectable
4.....	Slight
5.....	Moderate
6.....	Strong
7.....	Extra strong

The test against which the above tests were compared to determine which was the better for evaluating fat stability was the storage test carried out at 85F. The time that elapsed before the samples developed a flavor score of 4.5 was taken as an index of the shelf stability of the sample. The 95% confidence limits for these values are approx  $\pm 20\%$  of the value determined for values below 50 days and approx  $\pm 10\%$  of the values above 100 days when the samples are tested for flavor at sufficiently frequent intervals.

The changes in flavor with time in storage at 85F is shown graphically in Figure 1. These graphs indicate 1) a marked difference in the shelf stability of the several types of fats with and without antioxidant, 2) the addition of antioxidant may have a slight effect on improving shelf stability (hydrogenated vegetable oil) or a marked effect (lard), 3) the cause for the development of off flavor is not the same with all types of fats and 4) investigations involving blends of different types of fats would result in a very complex situation.

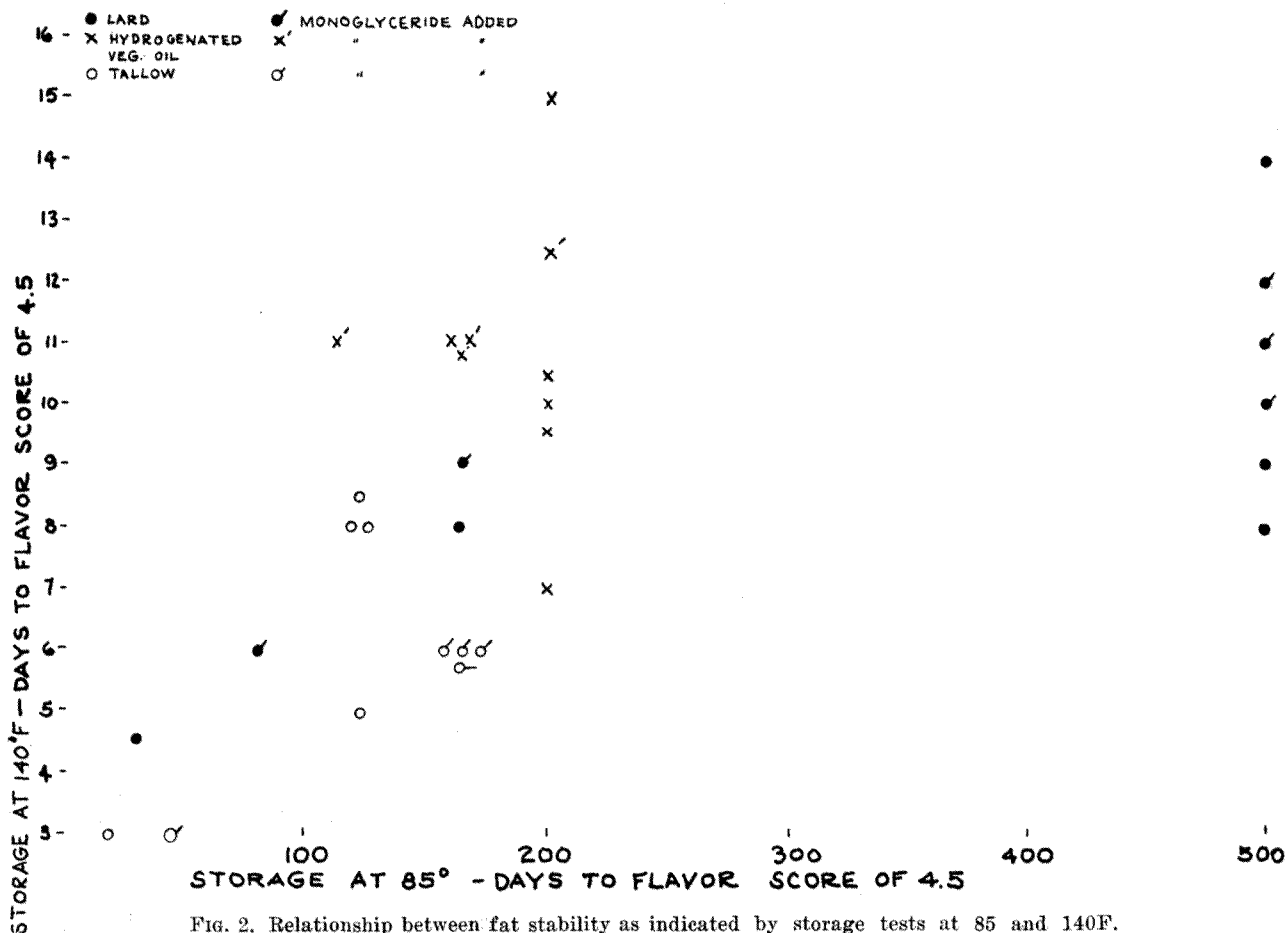


FIG. 2. Relationship between fat stability as indicated by storage tests at 85 and 140F.

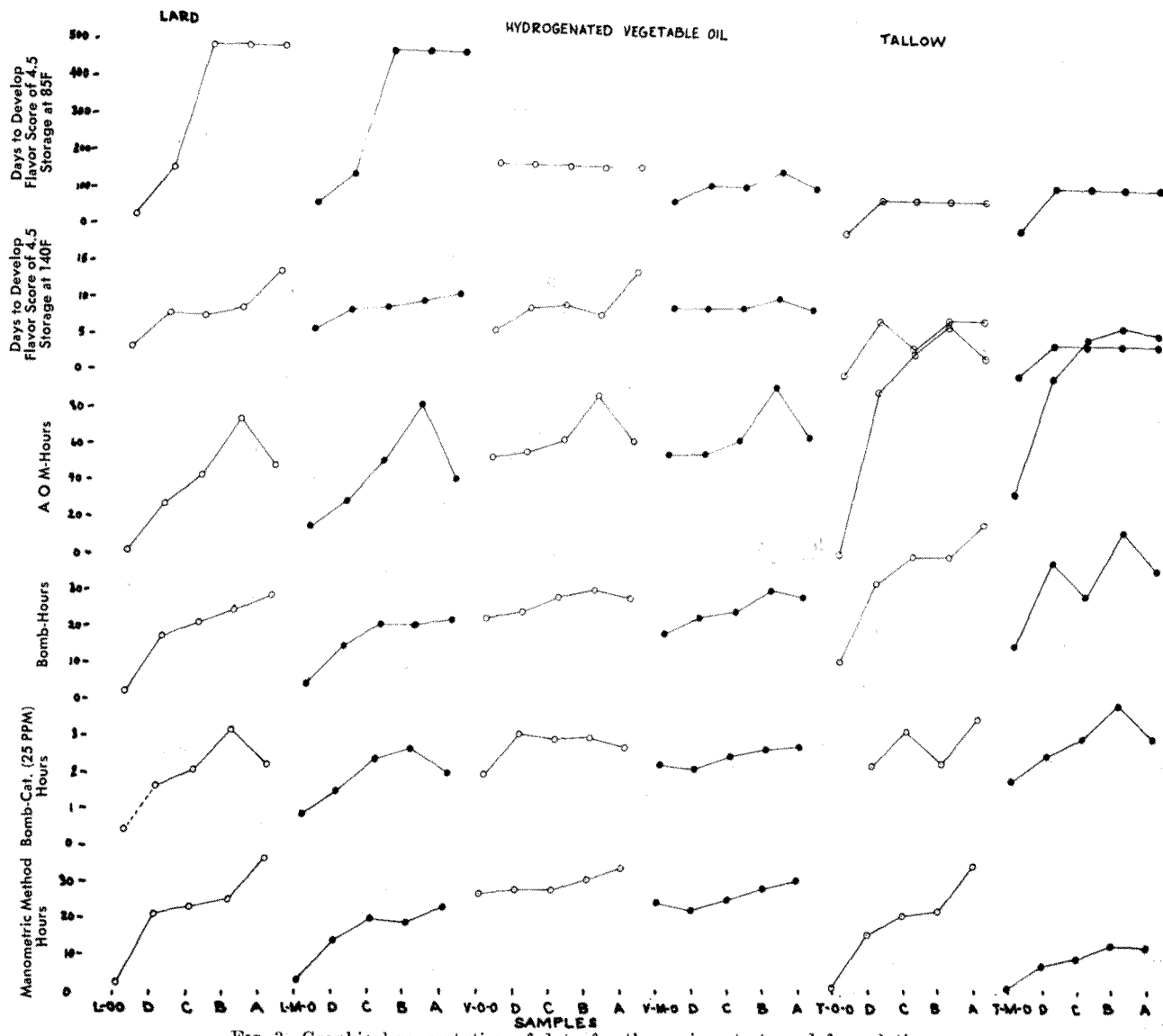


Fig. 3. Graphical presentation of data for the various tests and formulations.

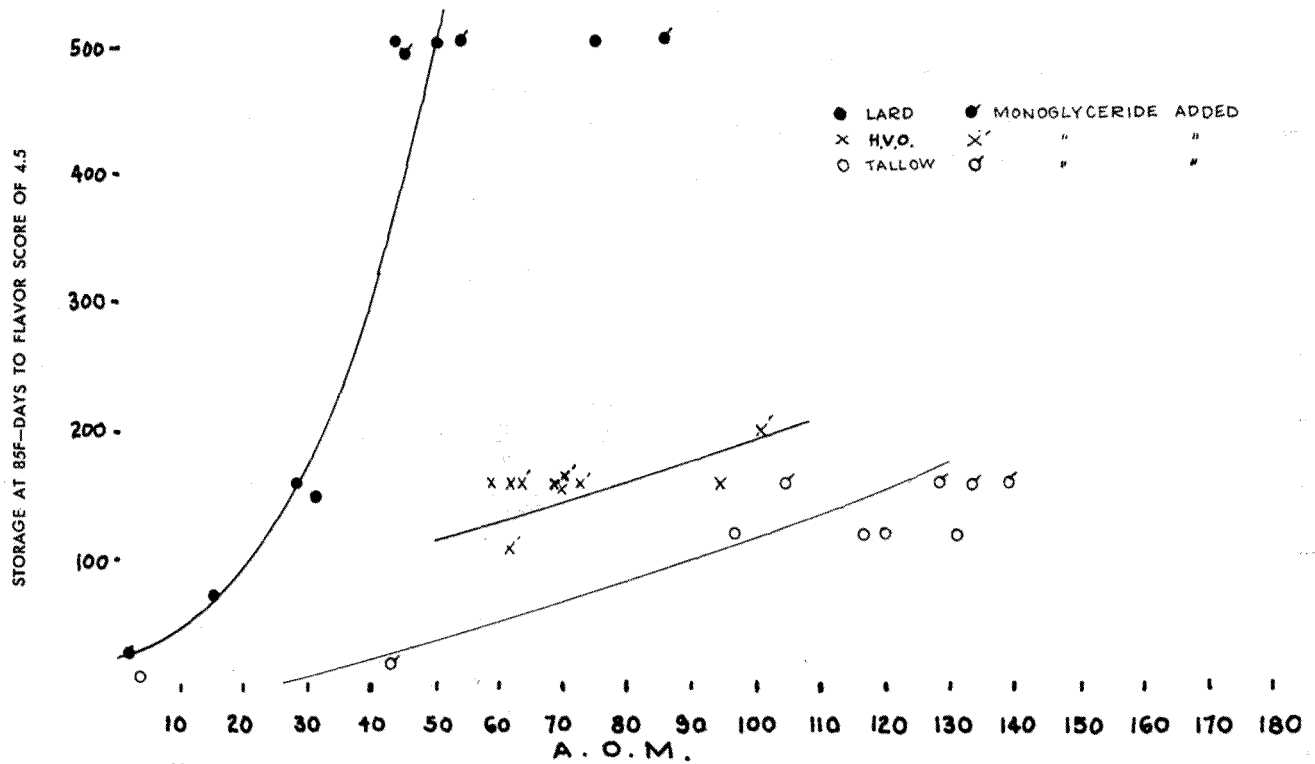


Fig. 4. Relationship between fat stability as indicated by storage tests at 85F and AOM in hr.

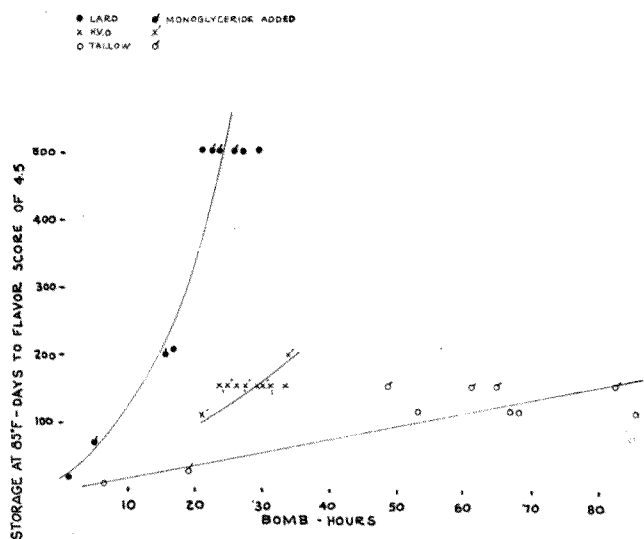


FIG. 5. Relationship between fat stability as indicated by storage tests at 85F and the modified ASTM bomb method in hr.

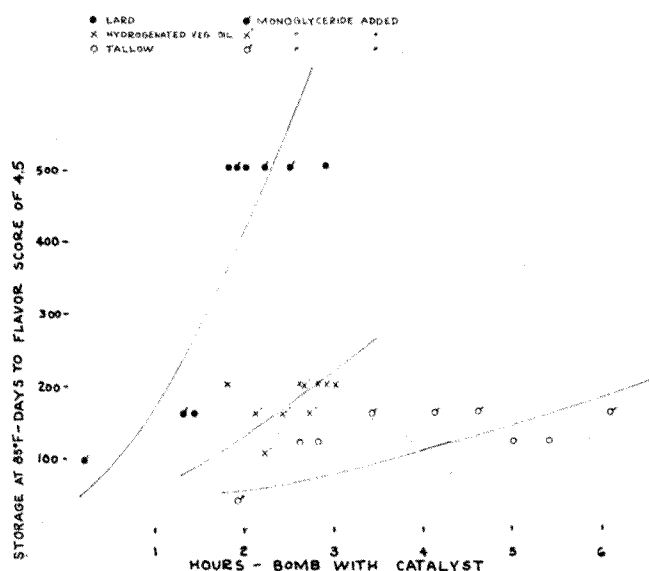


FIG. 6. Relationship between fat stability as indicated by storage tests at 85F and modified ASTM bomb in which catalyst was added to the fat to shorten test period.

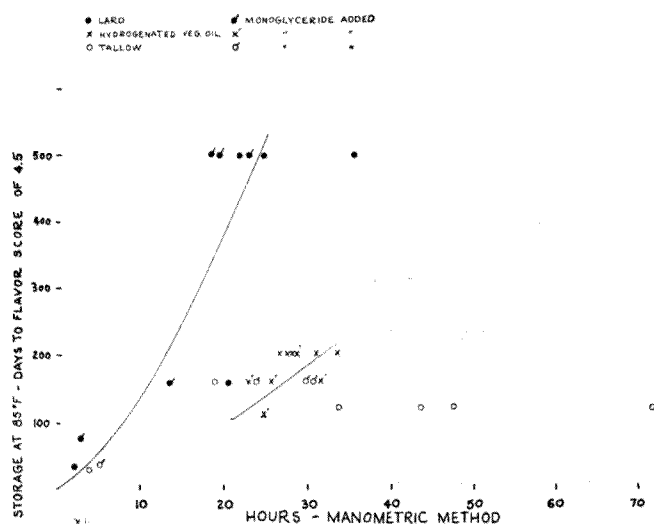


FIG. 7. Relationship between fat stability as indicated by storage tests at 85F and the manometric method in hr.

The storage test at 140F when used as an accelerated shelf life test is usually assumed to evaluate shelf stability more accurately than the laboratory methods such as AOM or bomb techniques. In Figure 2 the number of days required to develop a flavor score of 4.5 when held at 140F are plotted against the number of days in storage at 85F required to develop the same flavor score. From this data only a very rough estimate of shelf life can be made from the storage test made at 140F.

The data from all of the tests is summarized graphically in Figure 3. In this form, changes that take place under one method can be compared easily with those that occurred in another method. While this provides a good general means for comparison, a comparison of the respective laboratory methods with the stability as indicated by the storage test is evaluated more easily and accurately by a separate graph for each laboratory method used for estimating the stability of the sample. Figures 4-7 show the relationship between the stability of the samples established by storage at 85F to the stability as indicated by the respective laboratory tests employed for this purpose, AOM, modified ASTM bomb, modified ASTM bomb with catalyst added to the fat and the manometric method.

Examination of the graphs indicates that the modified ASTM bomb technique (Fig. 5), would be a more appropriate test for estimating the shelf life of a given type of fat or formulation than the other methods studied. Precision estimates for the various methods are given in a previous paper (4). The data shown graphically in Figure 6 indicates that the modified ASTM bomb method with catalyst added to the fat, in which the time required for completing a test is reduced to ca. one-tenth that of the modified ASTM bomb method, is not as precise as the modified ASTM bomb method but more precise than the AOM method. This is to be expected as one must usually sacrifice some precision when reducing the time required for a test.

## Discussion

This research has demonstrated that the several types of fats with the varying treatment factors studied result in differing stability relationships. There is no overall correlation that can be applied in all situations. None of the laboratory methods investigated can be used as an index of shelf stability except when it is applied to a given type of fat formulation for which a specific relationship has been established. The most reliable of the laboratory tests from the standpoint of precision and correlation with product stability in a given series or on a given type of formulation appeared to be the modified ASTM oxygen bomb method. The effect of antioxidants on shelf stability of the various types of fat varied from only a slight improvement on hydrogenated vegetable oils and tallows to a marked extension of stability on lard. The apparent effect of added monoglyceride varied depending upon the system studied.

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