THE OVEN TEST AS AN INDEX OF KEEPING QUALITY

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Abstract

The Schaal or Oven Test, originally developed by the biscuit and cracker industry to provide a relative rating for stability of various shortenings, has been generally adopted by shortening manufacturers. Advantages as well as limitations of the test are enumerated. It is possible to estimate the Oven Test by determining peroxide development in the test sample after two to four days in the oven. Oven Tests may also be conducted without having determined them organoleptically by observing the color change at the point of extreme rancidity. Peroxide formation occurring in a fat contained in biscuits or crackers when subjected to the Oven Test has been investigated and a simple and rapid method of extracting the fat, without changing its peroxide characteristics, has been devised.

THE oven test, also known as the cabinet or Schaal test, was introduced about fifteen years ago. It was developed by the biscuit and cracker industry to provide a relative rating for the stability of the various shortenings offered. Thus, it made possible the selection of those shortenings which indicated the greatest resistance to rancidity development in the baked products as they moved through the regular channels of distrbiution to the consumer.

As the test came into more general use it was adopted by shortening manufacturers, not so much as a control upon production quality, for because of its nature it is not particularly suitable for this purpose, but because it provided them with an index of keeping quality which rated their product on the same basis as those who purchased it. In this indirect manner the oven test has undoubtedly been one of the most potent factors promoting the improvement in keeping quality of shortenings which has been effected by manufacturers during the past decade.

Since its introduction other accelerated stability tests have been developed which are more rapid in their action upon the sample under examination. Most of these methods are based upon either oxygen absorption or peroxide formation. The equipment used and the evaluation of the data obtained are naturally more complicated. The simplicity of the oven test determination probably explains why it is today still one of the most widely used methods for evaluating the stability of oils and fats.

There are other reasons why it remains in favor. In addition to requiring a minimum of equipment, the test can be conducted with a reasonable degree of success by persons having a little or no technical knowledge. The result is expressed as "days required to develop rancidity" which is most significant to the biscuit and cracker manufacturer who must always think in similar terms of his products in the field before they reach the consumer. Technical data such as oxygen absorption or peroxide formation are difficult to convey to the layman, and at times tax the ingenuity of the chemist for adequate comprehension.

The oven test being conducted at a temperature not greatly above normal temperatures is useful for revealing odors and flavors other than rancidity, which may occur either in the shortening itself or in baked pieces containing it. Some fats develop characteristic odors and flavors due to their nature, while other odors may arise from faulty processing or contamination during distribution. The oven test will in many instances accelerate this condition if it is likely to develop as the shortening ages under normal usage. Such factors are not brought out by other accelerated stability tests.

Most of you are familiar with the procedure employed, or some variation of it. The oven should preferably be of the convection type capable of maintaining a uniform temperature of ± .5°C, and located in a room free from odors. The temperature most generally used is about 63.0°C. Ovens of the forced circulation type can be used without materially changing the results obtained, but tend to make the detection of the initial stages of rancidity development more difficult.

A sample weight of 50 grams is a convenient amount. An ordinary Griffin low form beaker of 250 ml. capacity, with or without lip, provided with a three-inch watch glass for a cover is a suitable container. The glassware must be scrupulously clean. The usual mixture of potassium dichromate and sulphuric acid may be used for cleaning, but it is not necessary. This mixture is not easily removed from glassware and even minute traces of it remaining in containers used for the oven test will certainly lead to erroneous evaluation of stability. Thorough washing with soap and water plus careful rinsing in distilled water, and drying, without wiping, in a heated cabinet is a much more dependable procedure.

The test samples should be smelled daily, preferably in the morning when the nose is keenest, for the development of a rancid odor. It is essential that the watch glass be kept on the beaker at all times except when actually smelling the sample, and the sample should be smelled immediately after removal from the oven. Usually as the end of the induction period approaches, the sample darkens in color, at which stage organoleptic rancidity soon becomes evident. The oven test is reported as the number of days required for the characteristic rancid odor to manifest itself.

Throughout the procedure the utmost care must be exercised not to change in some manner the inherent stability of the sample under test. This involves such well-known factors as freedom from any contamination, particularly metallic, avoiding overheating if the sample must be melted for preparation, and unnecessary exposure to light. With precautions taken for the elimination of any factors which tend to alter the normal keeping quality, then the stability of the shortening becomes a function of the oven temperature. For this reason the temperature must be carefully controlled within narrow limits. Also, to further the maintenance of a uniform temperature the oven should not remain open any longer than is necessary for inspection of the samples.

It is possible to estimate the oven test using peroxide formation as the index. To do this a curve must be plotted showing the course of peroxide formation during the oven test. Any one kind of fat which has been subjected to the same processing conditions will nearly always develop organoleptic rancidity at a fairly uniform peroxide concentration.

The length of time required to reach this concentration is determined by the rate of peroxide formation, which in turn is governed by the stability of the particular sample. Thus, if the peroxide content is known when the test is started, and is determined again after an interval of from two to four days, the increase during this period may be used as a basis for estimating the oven test, providing the peroxide content at the point of rancidity has previously been determined for the type and brand of shortening being examined. The rate of increase and the concentration of peroxides at the rancid point are influenced principally by the composition of the shortening, the amount and type of unsaturated glycerides present, and whether or not antioxidants have been used. Hence, any peroxide data for the purpose of estimating stability cannot be applied generally, but must be determined for each type and brand of shortening.

The principal limitation of the oven test is personal evaluation of the point at which organoleptic rancidity occurs. Fats with shorter induction periods are easier to judge because they become rancid so quickly at the end-point that there is a distinct change in odor between daily inspection. Those with longer oven tests are more difficult because they change much less from day to day as the development of rancidity is approached. Odors other than rancidity which may become evident, due to the type of shortening or to some variation in manufacture, sometimes mask rancidity or are confused with it. This further complicates the problem of arriving at uniform evaluations.

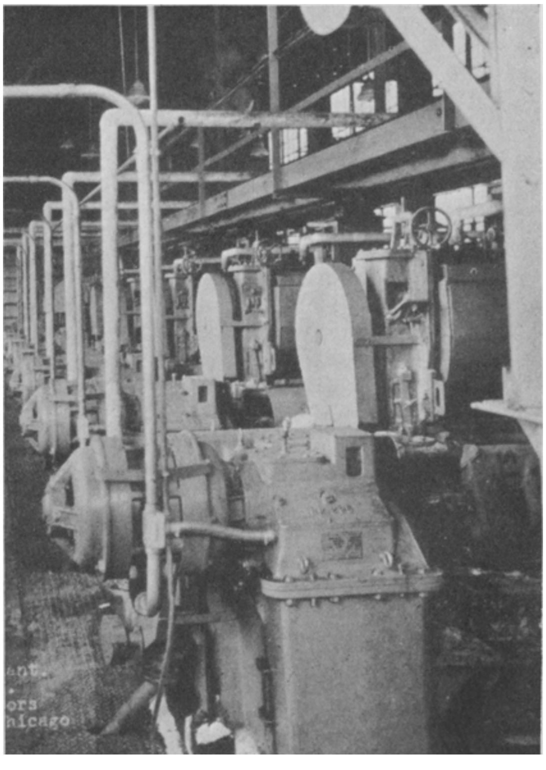
Changes in color can be used to advantage in determining the end-point. Essentially the same physical and chemical changes occur in a shortening during the oven test as take place under conditions employed in more rapidly accelerated methods.

As the end of the induction period is approached the color gradually darkens and rancidity soon becomes evident. If the test is carried beyond this stage to extreme rancidity the color will generally become lighter and this change occurs quickly, frequently overnight. Thus, the darkening in color may serve to indicate rancidity is approaching. A further reduction of personal variation can be effected on the basis of color change by carefully determining the end-point which would ordinarily be reported as the oven test, and then continuing the test until the sample becomes lighter in color again. The interval in days between the normal end-point and the point where the color lightens can be applied as a minus factor on future samples which are carried to this same color change. In this way it is possible to conduct oven tests without having actually determined them organoleptically once the preliminary data has been established. This procedure is helpful in reducing variations in judgment of the individual, but like using peroxide data for estimating the oven test must be used with care. The factors to apply will vary with the type and brand of shortening, and also with the length of the oven test. For example, a fat with an oven test of twenty days may run twenty-two days before the color lightens and require a factor of two, whereas a fat with an oven test of forty days may require a factor of four or five days.

One other limitation of the oven test is the length of time required to complete the determination. It has been discussed how peroxide formation during the early stages of the oven test can be utilized to obtain some idea of the indicated keeping quality.

There are several modifications of the Schaal Oven Test, all of which tend to accelerate the test and decrease the time of the determination. Some of these modifications employ the use of higher temperatures, others the use of absorbent materials to increase the surface of the fat exposed and still others in which a controlled amount of a pro-oxidative substance is added to the sample in order to increase the rate of oxidation.

Correlation of the keeping quality of a shortening, and biscuits or crackers containing it, by means of the oven test has been the occasion for much investigation. Baked pieces from commercial production or those prepared by the laboratory show a fair degree of correlation with the original fat, though the oven test of the baked pieces is as a rule of shorter duration. In some instances the relationship is very irregular. There are several explanations for these differences. Even with the best of handling in the laboratory or in the factory, variables are introduced which cannot be completely controlled. Pro-oxidative effects are incurred, such as metallic contamination in moving through production equipment. Also, the effect of the ingredients used is a variable. Some tend to exert a preservative action while others behave in the opposite manner. The greater surface area of the fat exposed to the air in the baked piece as compared with that of the fat when tested alone will contribute to differences in values for stability. One of the most important considerations is whether or not the shortening contains an antioxidant, and if it does, is its



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effectiveness carried through the mixing and baking operations and retained in the finished piece.

Peroxide formation occurring in a fat contained in a test piece such as a soda cracker, when subjected to the oven test offers an interesting field of investigation. Since the peroxide content cannot be determined directly on the piece, it is necessary to devise a simple and rapid method of extracting the fat without altering its peroxide characteristics. Standard extraction methods are time consuming and involve procedures which are apt to change the peroxide content of the fat as it existed in the baked piece. We have been working with the usual solvent mixture of acetic acid and chloroform used for the peroxide determination. Our procedure involves grinding the test piece finely, taking a sample weight dependent on the fat content, warming it gently and extracting with successive portions of the solvent mixture, which are decanted through a fast flowing filter. The peroxide number is determined directly on the combined extracts. By this method a determination can be carried to completion within a few minutes. The fat content of the test piece is accurately determined by the usual extraction procedure, and the proper correction applied to the peroxide figure obtained.

The weight of sample taken and the amount of the chloroform-acetic acid mixture used for extraction seem to exert some slight effect on the peroxide value found. Moisture in excess of that ordinarily present during the peroxide determination must also be avoided. However, these variables including any slight incompleteness of the extraction do not appear to be sufficiently great to render the method unsatisfactory for following peroxide formation of fats in baked pieces. If further work substantiates this indication, it should be helpful in studying the relative keeping quality of shortenings before and after the usual mixing and baking practices have been applied.

Aside from its limitations the oven test is subject to the same errors inherent in all accelerated stability procedures. These errors for the most part are largely a matter of technique and generally exert their influence as pro-oxidative factors tending to produce stability data below the true keeping quality of the product under examination. By adopting the simple practical procedure outlined in the foregoing remarks and careful attention to cleaning of the apparatus, reproduceable results and reasonable checks between laboratories are possible.

THE IODINE VALUE OF TUNG OIL

A Modified Wijs Method Giving Constant Values With Varying Excess, Time and Temperature.

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IN comparing the merits of analytical methods it is well to keep in mind just what we expect the method to do.

From the practical angle we expect such a method to be as simple as possible. At the same time it must give constant results. These results should be as independent as possible from secondary factors, such as temperature, time, etc.

From the scientific standpoint we expect it to give the true theoretical values. The Iodine Value of an oil, for instance, should indicate either the true degree of total unsaturation or with some oils, a partial unsaturation point. For instance, in the case of tung oil there is the true total Iodine Value of around 230 (3) (4) (5). Also, two true partial Iodine Values are possible, one around 90 and one around 160, corresponding to the points where one and two of the three double bonds of the elaeo stearati, respectively, have added iodine.

In many cases we will find that a method that gives a true theoretical value will also be the one that gives the most consistent results and is least influenced by secondary factors. The most accurate method from the scientific standpoint will therefore almost always be the most practical one—provided, of course, that it is simple to operate.

The Iodine values of many oils, as determined with the present methods, are such true constants, and are therefore not influenced by minor variations in temperature, time or excess reagent.

With tung oil the situation has unfortunately been different. The value found by the Wijs method in its various customary forms does not agree with either one of the theoretical true values—it is too high for the true partial ⅔ value of 160 and on the other hand, much lower than the true total value of around 230.

It should, therefore, not surprise us that this Iodine Value is a function of excess iodine, of time and of temperature. Instead of dealing with a true constant in this case, as we do, for instance, in the case of cottonseed oil, we are really dealing with an arbitrarily chosen section of a variable function.

This has been known for some time and the iodine value of tung oil has been the subject of a number of investigations. An admirably accurate and comprehensive study of the dependence of the iodine value of tung oil on the excess of reagent, on time, and on temperature was published in 1935 by the Chinese investigators, Ho, Wan and Wen (1). From the quantitative relationship which they derived it is apparent that with the ordinary method no constant value is obtained. In order to overcome the inconsistency of the results they suggest recalculating the determined value to certain standard conditions. However, such standard conditions are purely arbitrary, i.e., they do not correspond to any true constant of the sample.

The fact that tung oil has a true partial iodine value at around 160 has first been observed by van Loon (2) who noticed a break in a curve obtained by plotting a series of 2½ hour Wijs numbers against the weight of sample used in their determination.

A similar break at 160 is apparent in curves which were obtained by calculating back from some of the data given by Ho, Wan and Wen and similarly plotting the iodine numbers against the sample weights per 25 ml. of Wijs solution. Two such curves, corresponding to two different temperatures, are shown on Fig No. 1.

Translated into physical chemistry these curves show that two distinct reactions of different mechanisms are taking place. This agrees with the observation made about ten years ago by Gelber and Boeseken (3) that 2 out of the 3 double bonds in eleostearic acid will add iodine instantly, whereas the third double bond is attacked more slowly.