WHIPPED BUTTER: MANUFACTURE, CHARACTERISTICS AND GAS CHROMATOGRAPHIC ANALYSIS OF OFF-FLAVOR COMPOUNDS

BY

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AN ABSTRACT

Submitted to the College of Agriculture
Michigan State University of Agriculture and
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ABSTRACT SHANKERLAL H. VYAS

Preliminary trials on a procedure for whipping butter by the batch method using a Hobart machine model A120 (beater r.p.m. was 90) indicated that a butter temperature of 70° F. was satisfactory. Butter at 60° or 65° F. whipped adequately to overruns of 50, 75, 100 and 125 percent but required a longer time. The color became lighter as the overrun increased and concomitantly the firmness decreased as measured by a Precision penetrometer. In comparison with standards for butter, whipped butter decreased in score of body and texture as the percent overrun increased from 50 to 125.

Whipped butter (salted) deteriorated in storage at 40° F. when held for 1 month and longer. The rate was accelerated as the overrun increased. Similar results were observed in trials with storage at 34° F. although the rate of flavor change was significantly slower. Duplicate samples that were held at -10° F. did not show any flavor deterioration until after 32 weeks. Whipped unsalted butter had similar keeping qualities.

The addition of 0.05 percent antioxidants, BHT or BHA, in general exerted a slight beneficial effect on the flavor stability during storage at 40° F. Combination of BHT and BHA and/or with NDGA was slightly more effective in delaying a decrease in flavor score.

Chromatographic analysis failed to reveal compound or compounds responsible for the off-flavor of fishy, rancid or oxidized butters.

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INTRODUCTION

A present trend in the dairy industry in the United States is the decreasing consumption of butterfat. The per capita consumption of butterfat in 1909 was 17.8 pounds and in 1940 it was 17.0 pounds, showing practically negligible decrease during these 30 years, but when the figure of 1940 is compared with 1959 it is approximately less than half or 8.0 pounds. This decrease presents a serious problem for dairy industry in general and for the butter industry in particular.

The reasons for lower consumption have been attributed to the availability and cheapness of other fats e.g. lard, margarine and shortening. The consumption of margarine in 1940 was 2.4 pounds while it had increased to 8.2 pounds in 1959. The decrease of butter use per capita is also attributed to increased availability and improvement of other spreads such as peanut butter, mayonnaise and cheese. The limited use of fat by many diet-conscious consumers is also important.

The use of butter depends upon various physical properties e.g. flavor, general appearance, body and texture, color and hardness as well as cost. Since butter is one of the most costly of spreads, desirable physical properties such as flavor, texture and spreadability are highly significant.

Whipped butter offers the possibility of retaining the desirable characteristics of butter and reducing the disadvantages of spreading qualities and calorie intake. While whipped butter sales are small compared to butter, the potential is great if the problems of manufacture, packaging and keeping quality can be resolved.

Particularly important to some consumers are the prospects of better

spreadability at cool temperatures and the resulting easier control of calorie content with the use of whipped butter.

Unfortunately whipped butter, as well as butter, looses its pleasing flavor and odor during storage, especially under unfavorable conditions. Both products are readily susceptible to chemical and other deteriorations. The common flavor and odor defects that develop are fishiness, tallowy, rancid, storage, oily and others.

In the past the detection and evaluation of these off flavors has been by organoleptic means. This method requires much training and inherent skill and even with the best of these qualities the limitations are serious. The relatively new method of gas chromatography has certain features that warrant its study for application to the compounds responsible for the common off-flavors of butterfat.

For use in the study of other applications gas chromatography has been shown to have the advantages of simplicity, low cost, easy data interpretation, high degree of sensitivity and usually little interference among components. Gas chromatography is proving to be valuable tool for uniform flavor development of food products in a few industries.

Since the potential for whipped butter is large but the study of this product has been extremely limited, as well as the application of gas chromatography to the volatile off-flavors, the objectives were:

(1) To develop a satisfactory batch procedure for the processing of whipped butter.

(2) To study the influence of various amounts of air in whipped butter on keeping quality and spreadability, and the affect of the addition of antioxidants.

(3) To investigate the components of common off-flavor by gas

chromatography.

REVIEW OF LITERATURE

Rogers, et al. (1913) made the following statement; "The most serious difficulty in experimental work on butter is in controlling the conditions under which butter is made. So many apparently unimportant factors have an influence on the flavor that it is nearly impossible to make butter with a normal flavor and have only one varying factor. The work is further complicated by the sequence of flavors that frequently occurs in butter held in storage. It is evident that the usual off-flavors are in many cases a combination of flavors and these flavors are caused by a combination of circumstances and not by a single cause. It is probable that identical flavors may be caused by different compounds."

Barnicoat (1937) emphasized that the market value of well manufactured butter is determined more by its flavor than by any other characteristic and the knowledge of desirable and undesirable flavoring substances is therefore of considerable importance.

Van Niel (1927) was the first to show that the aroma of butter made from ripened cream is related to its diacetyl content. A considerable amount of work has since been done, notably by Hammer, et al. (1935), Hedrick and Hammer (1942) on the development of diacetyl and its precursor acetylmethylcarbinol in starter cultures or in manufacture of starter butter. Diacetyl and acetylmethylcarbinol were retained in butter with the buttermilk fraction. Diacetyl content remained constant during 3.5 months storage at 14° F. and did not affect the keeping quality of butter in the usual concentration. Babel and Hammer (1944) found a slow diffusion of diacetyl between the fat and serum phases of butter during storage.

Pette (1949) studied the formation of diacetyl as a flavor component in butter and concluded that citric acid was the source of diacetyl. Ling (1956) observed that both the fat and serum of butter contribute to the flavor. Diacetyl was one of the compounds involved in flavor, but there were undoubtedly others that have not been identified. He further reports that there is a very great difference of opinion regarding the origin and mechanism of formation of diacetyl in butter.

Some of the most serious flavor defects in butter are: fishy or oily, rancid, tallowy or oxidized, etc. The presence of any of these flavors causes a reduction in commercial value of the butter. Considerable efforts have been made to determine their causes and prevent their development.

Fishy butter resembles the odor and taste of fish. The flavor usually resembles that of mackeral, salmon or herring. Sommer and Smit (1923) reported that fishy flavor in butter was first referred to by Storch in 1890.

Sommerfeld (1909) observed that many of the butter defects have their origin in the souring of cream. The oily taste of butter was often accompanied by a fishy taste which apparently was due to the formation of trimethylamine.

Rogers (1909) did not believe that trimethylamine was the immediate cause of fishiness, but high acid cream and overworking encouraged the defect by a slow chemical change in which acid was essential. Rogers, et al. (1913) showed that iron and copper salts have a strong effect in causing the development of fishiness. Rogers (1909) stated that fishiness rarely occurred in unsalted butter and that the salt possibly furnishes certain conditions which were essential to the development of the

flavor.

Dyer (1916) concluded that fishiness was caused by the slow oxidation of one or more of the nonfatty constituents of butter.

Washburn and Dahlberg (1917) also reported that salt hastened the deterioration of butter in storage; salted butter was more likely to develop fishiness during storage than unsalted butter. There appeared to be a tendency toward a progressive development of the off-flavor of metallic to oily, then to fishy butter.

Supplee (1919) was able to extract trimethylamine from fishy butter and by working trimethylamine salts of fatty acids into normal butter in small amounts was able to produce the fishy flavor; he concluded that lecithin, which was isolated from butter, was the source of the trimethylamine.

Sommer and Smit (1923) stated that the conditions for the development of fishiness by oxidation in storage butter were high acid, high salt, iron or copper compounds, overworking and a storage temperature between 30° and 40° F. They further showed that lecithin will undergo a purely chemical decomposition yielding trimethylamine. They also gave the following reasons for salted butter showing more fishiness than unsalted butter:

(1) Salt in butter intensified the flavors. This also is true for fishiness; when trimethylamine was incorporated into unsalted butter and then part of this butter was salted, it was found that the salted portion had a more intense fishy flavor. Supplee (1919) found that the addition of sodium chloride to trimethylamine salts of fatty acids caused the precipitation of sodium soap and the formation of trimethylamine hydrochloride.

This reaction may explain in part the intensifying effect of salt on the fishy flavor.

(2) Lecithin is soluble in common salt solutions. By dissolving the lecithin in the brine of the butter, an acceleration of the hydrolysis and oxidation of the lecithin into trimethylamine occurred.

(3) Salt solution of butter lowered the freezing point and thus caused a physical condition that was more favorable for chemical reactions.

The theory suggested by an early worker, Sommerfeld (1909) that trimethylamine was the cause of fishy butter was questioned by Van der Waarden (1947) and also by Storgards and Hietaranta (1949). They obtained results that do not verify the quantity of trimethylamine and its oxide was related to the intensity of the fishy flavor in stored butter. Furthermore, they thought that trimethylamine could not come from lecithin through an oxidation process, but more likely as a result of a reduction process. Van der Waarden (1947) has shown that the fishy flavor remained in the fat after the basic volatile constituents in stored butter have been removed, indicating that trimethylamine has little influence on these flavor defects.

Storgards and Hietaranta (1949) added trimethylamine to butter and produced fishiness even after artificially complete oxidation of the trimethylamine. There were many factors in favor of this theory; fishy flavors originate in the decomposition products that result from autoxidation of butterfat. During oxidation of melted butterfat, that was free of lecithin, a distinct pronounced odor of fish was observed before the fat became tallowy. On adding more oleic or linoleic acid the smell of

fish became more conspicious. The addition of lecithin or trimethylamine to butterfat prior to oxidation caused a preventive effect on the formation of fishy odor and in no case did it promote these flavor defects. Storgards and Hietaranta (1949) concluded that fishy flavors in butter were formed by certain decomposition products resulting from oxidation of butterfat. The oxidation products of linoleic acid, in particular, were of decisive importance.

Van Haeften and Pette (1953) of the Netherlands observed that the fishy off-flavor in summer butter stored at 0° F. developed more rapidly in salted than unsalted samples. It did not appear in winter butter and was not encouraged by added lecithin.

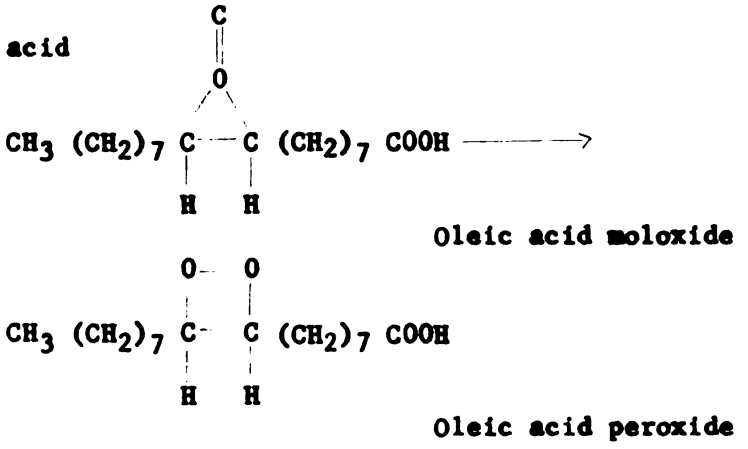
Pont, et al. (1960) stated that the development of fishiness in butterfat was due to the presence of dissolved acid, an antioxidant, a restricted air supply, and to, a lesser extent a moderate holding temperature. Copper was not an essential factor although it intensified the defect after its development. The flavors of oxidized fat were due to saturated and unsaturated carbonyl compounds of intermediate carbon chain length. Little is known about the chemical reactions originating from the initial hydroperoxidation with the formation of particular carbonyl compounds. Each of the major types of off-flavors resulting from oxidation of fat was associated with particular compounds or group of compounds. The relative proportions of such groups were influenced by various factors, such as temperature, catalysts, acidity and the particular chemical environment in which the decomposition of peroxides takes place. The factors promoting the development of fishy defect in oxidizing fat would be expected to influence strongly the chemical reactions following peroxide decomposition.

The term, rancidity, is used in the dairy field to indicate hydrolytic oxidation. Rancid flavor in butter resembles the pungent, rasping taste and odor of such volatile fatty acids as butyric, caproic and caprylic acids. Holman, et al. (1954) observed that prolonged exposure of edible fats and oils to air, heat and light eventually resulted in the development of this unpleasant odor and flavor, rendering the fat unpalatable and even toxic.

Rancidity in commercial butter is generally due to the enzyme lipase which splits the butter into free fatty acids and glycerol. Holman, et al. (1954) further stated that autoxidational rancidity of fats was caused primarily by the action of oxygen upon unsaturated constituents. The rate of oxygen reaction by a fatty substance increased with the degree of unsaturation of the substance. An intermediate moloxide was first formed which was changed into a stable peroxide.

$CH_3 (CH_2)_7 CH{:}CH (CH_2)_7 COOH \longrightarrow$

Oleic acid

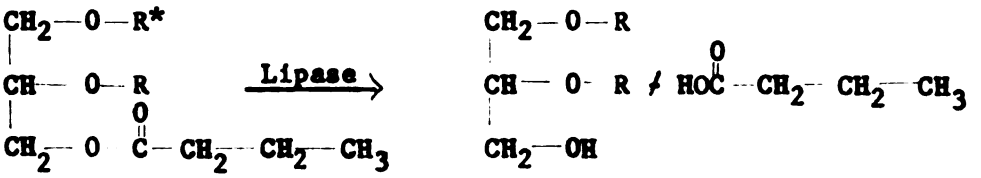


Oleic acid moloxide

Oleic acid peroxide

This peroxide can act as an oxygen carrier and increase the oxidation of unsaturated acids. Linoleic acid was more readily attacked than oleic acid while linolenic acid has a still greater susceptibility to oxidation.

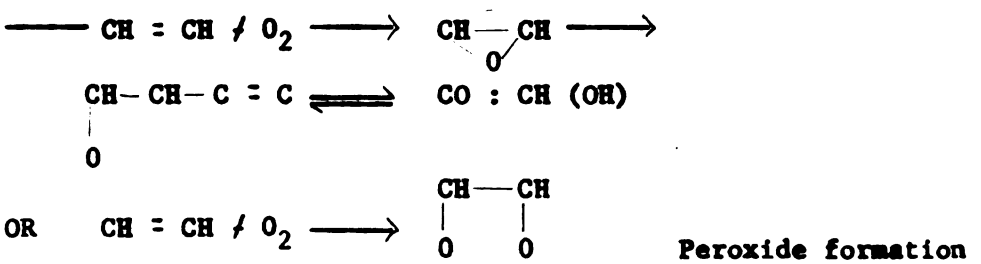
Jenness and Patton (1959) observed that butyric acid was the most volatile acid, a very limited degree of lipolysis of butyrin was required before the formation of rancid flavor can be detected.



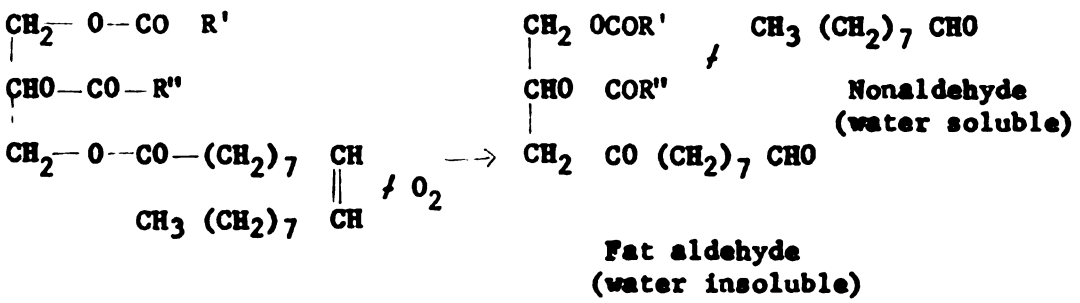
*R = An acyl Radical

Hunziker (1940) described the tallowy flavor of butter as resembling mutton tallow. In severe cases of tallowiness the color of the butter was bleached white. While rancidity was due to fat hydrolysis initiated by the action of lipolytic bacteria and enzymes, tallowiness was caused by oxidation of the fat involving unsaturated fatty acids, such as the oleic acid, in the butter.

Holman, et al. (1954) stated that tallowiness was the result of fat oxidation and that a product was needed to link up with the fatty acids, in order to cause tallowiness. The product was found to be glycollic acid ester of oleic acid. Only a very small amount (0.25 percent) of this ester added to butterfat produced a distinct tallowy flavor and odor. The chemical reaction was a simple oxidation:



Complex reaction showing oxidation of glyceride to talloviness is:



Water insoluble aldehydes are probably flavorless, but the water soluble aldehydes which are found upon oxidation of the etheroid linkages of the fatty acid radicals cause an oxidized flavor.

Jenness and Patton (1959) emphasized the importance of oxidized flavor in milk and milk products. Such terms as cardboard, metallic, oily and tallowy also were used to describe off-flavors. These off-flavors have the oxidation of lipid materials in common. They further observed that the phospholipides of milk serve as the precursor for oxidized flavor. One-third of the phospholipides were found in skim milk; the other two-thirds were associated with the material of fat globule membrane. Sweet cream buttermilk which was particularly high in phospholipides, was very susceptible to oxidized flavor development. Moreover, butter oil free of phospholipides can be prepared and stored for months under suitable conditions without development of oxidized flavor. It was very difficult to prepare phospholipides without immediate appearance of this defect. Phospholipid oxidation was accompanied by a flavor termed cardboard or cappy. Milk fat, undergoing oxidative deterioration shows an oily flavor and aroma resembling that of linseed oil.

A number of factors were responsible for the development of oxidized flavor. Light, air and heat as well as metallic ions of copper and iron were some important ones.

Greenbank (1949) observed that glycerides were more resistant to oxidation than the phosphatides. He reported that the oxidation of the glycerides may be divided into two phases. The first phase or induction period was a slow oxidation with little absorption of oxygen, a slight increase in peroxide value and little or no change in flavor. Keeping quality of butter was proportional to the length of induction period. The second phase was marked by a rapid absorption of oxygen and increase in the peroxide value. Off-flavors and aromas developed during this period.

According to Holm, et al. (1938) if the moisture was too low the oxidation was predominantly to aldehydes which have more objectionable flavors than fatty acids.

According to Stebnitz and Sommer (1937) and Mattsson (1949) linoleic acid is of great importance in the oxidation of butterfat. They have shown that methyl esters of linoleic and linolenic acids oxidize 12 to 15 times more rapidly at 20° C. (68° F.) than the methyl ester of the oleic acid. In addition, the hydroperoxide formed on oxidation of the first named esters appeared to catalyze the oxidation of the methyl oleate.

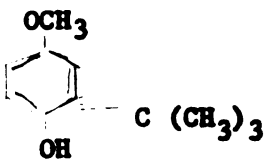
Tollenaar (1949) of Holland observed that oxidative change in lipids can be of at least three different kinds, resulting in ketone formation, peroxide formation and flavor reversion.

Ketone rancidity is limited to fats containing fatty acids with six to 12 carbon atoms, i.e. caproic acid to lauric acid. Ketone rancidity is an oxidation reaction; it cannot be prevented by antioxidants.

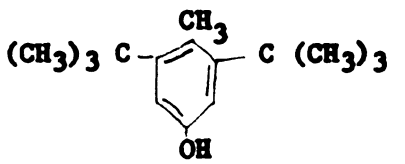
Holman, et al. (1954) defined antioxidants as substances that were able to prevent or delay the oxidation of the oil. They were present in most natural fats and oils and contributed to the natural stability of

raw oils often removed by purification. The inhibitor terminated carbon chains by interaction with peroxide radicals.

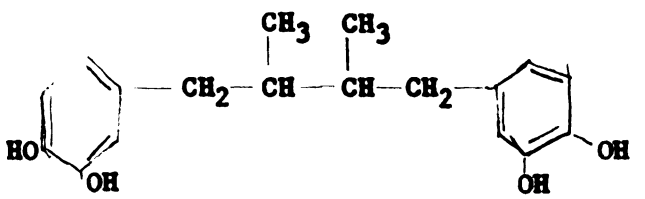
Koch (1956) discussed phenolic antioxidants which were approved for food use. Phenolic antioxidants possess a ring structure and each has OH groups attached to the rings. BHA (Butylated hydroxyanisole) and BHT (Butylated hydroxy toluene) differ from NDGA (Nordihydrogaiaretic acid) in the number of OH groups attached to the ring structure. BHA and BHT are insoluble in water, while NDGA is slightly soluble due to the presence of extra OH groups.



BHA



BHT



NDGA

(Phenolic type antioxidants)

Swartling (1949) studied the following, aromatic hydroxy compounds in butter with varying concentrations:

Antioxidants tested	Percent concentration in butter
Ethyl gallate	0.05—0.00125
Propyl gallate	0.05—0.005
Propenyl-methyl-guaethol	0.01—0.0025
P. Methoxy-phenol	0.01—0.0025
Ethyl-hydro-caffeate	0.01—0.0025

NDGA	0.01—0.0025
Conidendrin	0.05—0.0050
Alpha-tocopherol	0.01—0.0025
Alpha-tocopherol acetate	0.01—0.005

The highest antioxidative effect in these tests was shown by the esters of gallic acid and NDGA. A concentration of 0.005 percent NDGA inhibited oxidation to a certain degree and the butter possessed no oily flavor. The grade of butter, however, was not high enough to encourage further experiments. This was due to a slight, but distinct and objectionable flavor given to the butter by NDGA. The off-flavor was more noticeable when larger amounts of NDGA was used.

Gould (1946), studying the effect of antioxidants in preventing hydrolytic rancidity, observed that the addition of 0.75 and 1.5 percent Avenex concentrate to raw cream retarded lipolysis and rancidity in the resulting raw cream butter. Stull, et al. (1949) studied the effects of NDGA in dairy products and concluded that:

(1) Concentrations of 0.00125 to 0.005 percent (weight of butterfat) of NDGA were found to retard the development of oxidized flavor in sweetened frozen cream during storage for 12 months.

(2) During storage for 1 week at 40° F. an oxidized flavor developed frequently in the control samples that did not have the off-flavor when first were taken out of storage at sub-zero temperatures. This development of oxidized flavor occurred only in one trial in the cream which contained NDGA.

(3) During storage for 1 week at 40° F. the intensity of oxidized flavor usually increased in the control samples, which had the off-flavor, when they were removed from storage at sub-zero

temperatures. This did not occur in the oxidized sample which contained NDGA.

Gelpi, et al. (1952) studied the effect of BHA (Tennox), with and without a synergist, an oxidized flavor in frozen cream stored at -10° F. They found that the antioxidants were effective in preventing the development of oxidized flavor for at least 6 months or longer in the absence of added copper. None of the other antioxidants used were effective beyond 5 months of storage. Pedersen, et al. (1955) (Denmark) studied the testing of antioxidants, glycine, quinol, ascorbic acid and preparations containing gallic esters NDGA, and found them unsuitable for use in butter, due to their production of off-flavors in low concentration and/or their low antioxidant capacity. Tollenaar and Vos (1959) of Germany studied BHT as an antioxidant in foods and reported that when used, alone or in combination with other antioxidants, good results for butter, margarine, and dried milk were obtained.

Zalashko (1959) of Russia stated that the effect of synthetic antioxidants on the stability of butterfat and butter showed that propyl gallate and dodecyl gallate at 0.01 percent concentrations were most effective. Negoumy and Hammond (1960) used .01 percent BHT, NDGA and BHT plus .01 percent Isopropyl citrate on butterfat basis and stored the butter at -18° and 38° F. They concluded that these antioxidants did not improve the flavor of butter significantly over the control butter. NDGA gave best protection, although, butter containing it had an objectionable bitter flavor.

Scheib, et al. (1942) studied the effect of natural milk enzymes, acid, and salt upon the keeping quality of butter stored at 0° and -10° F. for 6 years and gave 92.3 as the score for butter which was free from

flavor defect. He concluded that the presence of salt in butter was harmful to its keeping quality, but less harmful than acid, or acid and salt. The scores of 90.8, 87.5 and 85.4 indicated the deteriorating effect of salt, acid and salt, and acid on the keeping quality of butter stored at 0° to -10° F. for 6 years. In the absence of harmful natural enzymes of milk or spoilage bacteria, the score of the sweet cream, salted butter was 1.5 points less than the sweet cream, unsalted butter; the score of the sour cream, unsalted butter, was 4.8 less than the sweet cream, unsalted butter; and the score of the sour cream, salted butter, was 6.9 points less than the sweet cream, unsalted butter, indicating the relative value of salt and acid as spoilage factors in storage butter.

McDowell (1955) showed that a stale, storage defect occurred in both salted and unsalted butter stored for 8 months at 14° F. Oxidation was much lower in both unsalted and highly salted butter than in butter of normal salt content. Salted butter treated with antioxidants showed retarded oxidation, but unsalted butter, and salted butter treated with antioxidants, all developed storage flavor during 8-month hold.

A report from Denmark (1949) showed that the higher percentage of salt and the longer the butter was in cold storage, the greater was the tendency for the development of oily flavor.

Khotsko of Russia (1957) stored butter for 12 months at -5° to -12° C. (23° to 10° F.). Salted ripened-cream butter deteriorated most rapidly and unsalted sweet cream butter least rapidly; unsalted, ripened cream and salted, sweet cream butter deteriorated at an intermediate rate. At temperatures of 5° to 7° C. (41° to 44° F.) salted ripened-cream butter kept the longest and unsalted sweet cream butter for the shortest time.

Meyknecht and Dam (1959) of Netherlands observed that the quality

of unsalted butter stored at -15° C. (5° F.) for 5.5 or 7.5 months was significantly higher than that of the same butter stored at -10° C. (14° F.).

ScottBlair (1938) defined spreadability as a psychological component in which a number of separate physical properties are involved. Two principal factors, brittleness and hardness, were important.

Many attempts have been made to apply mechanical or instrumental methods to measure the physical properties of butter. The different systems used for measuring the physical properties of butter were discussed in detail by McDowell (1953). They were listed as follows:

(1) Penetrometer methods

(2) Crushing or compression methods

(3) Sectility methods

(4) Extrusion methods

(5) Sagging beam methods

(6) Spreadability methods

(7) Oil drainage methods

(8) Tendency to become greasy

(9) Measurement of dielectric capacity

Huebner and Thomsen (1957a) stated that the best known methods of measuring hardness are the penetrometer, sectility, extrusion, and crushing-strength methods.

Coulter and Combs (1938) studied the spreadability of the butter on a glass plate by means of a knife fixed at a 45-degree angle. The butter was held at 40° F. for 1 week after manufacture before examining for spreading quality.

Reid and Arbuckle (1939) studied the effect of tempering butter

samples at 40°, 50°, 60° and 70° F. for 4 hours, and then spreading each on bread. The butter was very spreadable at 60° F., while the higher temperatures caused salviness, and the lower temperatures caused hardness and crumbliness. Dolby (1941a) showed an almost direct relationship between temperature and hardness within the range of 43° to 64° F. McDowell (1953) stated that the butter should be spreadable at a temperature 55° to 60° F. McDowell described penetrometer methods as follows: "Measurements are taken of the depth of penetration of a needle or of a ring, placed on the butter under load, or of the width or depth of depression in the surface of the butter when a metal ball is dropped onto it from a measured height or of the volume of butter displaced. When a metal cone is dropped onto the butter from a given height or measurements are taken of the velocity with which a cylinder sinks into the butter under fixed load."

The penetrometer method is used in Holland as an auxiliary test in the grading of butter. The butter is left in the store for a week at 13° C. (55° F.) before the test is applied.

Thomsen (1955) studied a number of factors in butter manufacturing and their effect on spreadability. The important variables were:

(1) The treatment of cream, such as pasteurizing, cooling and holding (includes ripening),

(2) Treatment of butter in churn, e.g. temperature and washing of granules,

(3) Treatment of butter when it is removed from churn, e.g. prompt cooling to below 15° F. This was the most important variable, and

(4) Market and consumer handling of butter.

Fairly important variables were:

(1) Composition of fat,

(2) Size and physical makeup of fat globules,

(3) Method and amount of working.

(4) Alternate method of making butter,

(5) Additives in butter, and

(6) Churning temperature.

Minor important variables were:

(1) Richness of cream,

(2) Size of butter granules,

(3) Salting of butter, and

(4) Composition of finished product.

In an effort to study the effect of additives to cream or butter, Thomsen observed that "Tween 40" (polyoxyethylene sorbitan monopalmitate) when added to cream at the rate of 0.07 to 1.17 percent of the weight of the cream caused no improvement in spreadability, but if it was added to the butter at the rate of .16 to 0.4 percent of the weight of the butter, there was a slight but significant improvement in the spreadability.

While studying the storage temperature, Wilster (1948) found that butter which was stored at 0° F., and then tempered for packaging, was always softer than butter which has been initially stored at 60° to 65° F. Coulter and Comb (1938) observed that butter stored for 7 days at 32° F. was softer than that stored at 60° to 65° F.

While Dolby (1941b) observed no significant improvement in spreadability of butter stored at freezing temperatures, Huebner and Thomsen (1957b) stored butter at 36° and -6° F. and found after 16 weeks that the hardness and spreading resistence values of butter stored at 36° F. were

only slightly greater than those of the butter which had been stored at -6° F.

DeMan and Wood (1958) suggested that hardness of butter was usually measured within the range of 12° to 17° C. (53° to 61° F.) according to the type of instrument used for testing. For the penetrometer method they suggested a temperature of 17° C. (61° F.) while for sectility they suggested 12° C. (53° F.). They did not observe any correlation between the increase of hardness and a storage temperature of -20° F. This was contrary to reports by Huebner and Thompsen (1957b). DeMan and Wood (1959) studied the influence of hardness of butter made by conventional and continuous methods. Hardness was less in continuously made butter than in churned butter.

Haigton (1959) found in studying the hardness of margarine and fats with cone penetrometers that the consistency ranged between very soft to very hard. Samples had values of approximately 25 to 2000 g/cm^2. Reil (1960) of Canada concluded after an organoleptic study on butter, that desirable spreadability ranged between 150 and 400 g. of resistance; acceptable spreadability ranged between 100 and 800 g. Betscher, et al. (1959) obtained improvement in spreadability and hardness of butter by adding various additives to cream at 2.3 percent levels, for example, distilled monoglycerides, vegetable lecithin, a mixture of mono-, di- and triglycerides and vegetable gum. He also observed appreciable improvement in spreadability by the addition of 2.3 percent Tween 40 (polyoxyethylene sorbitan mono-palmitate), Span 80 (Sorbitan mono-oleate), skim milk solids, buttermilk solids, and vegetable lecithin. He noticed, however, a wide variation in the results of several trials using the same additive, indicating the action of unexplained factors.

Kapsalis, et al. (1960) used consistometer to study spreadability and hardness of butter. In analyzing 109 commercial samples of butter from 14 different states, they could not find positive correlation between consistency of the butter and the type of cream, temperature of wash water, length of storage time before printing, printing temperature, and the geographic area of production. An experiment was conducted by Mickle (1960) to change the molecular sturcture of milk fat as a means of improving the spreadability of butter. He observed that molecular rearrangement by directed interesterification reduced the hardness of butter on an average by 50 percent. He stated that this fat had a rancid flavor which could be removed by refining processes which consisted of neutralizing and steaming.

Gas chromatography has been defined by Lederer and Lederer (1957) as an analytical method for the purification and separation of organic and inorganic substances, etc. It is particularly useful for the fractionation of complex mixtures, the isolation of unstable substances, and the separation of closely related compounds.

Forss, et al. (1955) studied the volatile compounds associated with oxidized flavor in skim milk by using chromatographic techniques and isolated acetone, acetaldehyde, n-hexanal, crotonaldehyde and the C_5 to C_{11} 2-unsaturated aldehydes. Several 2, 4 di-unsaturated aldehydes of medium chain length were obtained. Acetaldehyde and acetone were also isolated from normal skim milk; these two compounds do not contribute to the oxidized defect. The principal flavoring compounds were 2 enals particularly the C_8 and C_9 carbon compounds.

It was further concluded that cardboard flavor in milk was caused by the preferential oxidation of di- and polyunsaturated fatty acids.

The larger quantities of 2-octenal, 2-nonenal, 2, 4-heptadienal and 2, 4-nonadienal were found. The main constituents of the oxidized flavor originated in the oxidation of the more highly unsaturated fatty acids in milk lipids. Tamsa (1955) in his study of heat and light induced oxidized flavor in butterfat found the flavor was partly due to carbonyl compounds which showed no absorption in the ultra-violet region. They could be separated on celite chromatographic columns in light petroleum and were recovered in the first fractions.

Keeney and Patton (1956a) studied a coconut-like flavor defect of butter oil from unsalted butter held in storage. The off flavor was identified as delta-decalactone (the lactone of 5-hydroxy decanoic acid). The compound was confirmed with various tests, e.g. by its odor, infrared spectral properties and two different chromatographic procedures. In studying the coconut-like flavor defects in dried cream, dry whole milk and evaporated milk these same authors (1956b) in a later article revealed the presence of two other compounds (by chromatographic analysis) which gave a colored derivative when sprayed with $FeCl_3$ solution. Only one component was present in the coconut-like extract obtained from butter oil. Mattick, et al. (1959) studied a coconut-like defect in fat containing dairy products to determine the origin of delta-decalactone and postulated a possible mechanism for the formation of this lactone. The information obtained indicates that delta-decalactone is derived by a hydrolysis which liberates 5-hydroxydecanoic acid.

Tharp and Patton (1960) studied coconut-like flavor defect in the steam distillate from milk fat by gas chromatography employing a Barber-Coleman model 10 instrument. They used an 8-ft. glass column having a 6-mm. inside diameter, packed with 100 to 140 mesh celite and coated with

20 percent (w/w) Apiezon L.; carrier gas (argon) at a pressure of 30 p.s.i. and an approximate flow rate of 150 ml./min. The column was maintained at 220° C. (428° F.), detector at 265° C. (509° F.) and sample heater at 300° C. (572° F.). The sample injected into the machine was 1 microliter of a 0.1 percent solution of delta-dodecalactone in hexane. The experiment showed the presence of delta-dodecalactone, a compound structurally analogous to the C_{10} lactone except that it contains 12 carbons.

Wong, et al. (1958) studied the flavor compounds in evaporated milk and found actone, pentanone-2, and heptanone-2 as the main volatile carbonyl compounds in low-temperature, reduced-pressure distillates from commercial evaporated milk. Heptanone-2 is considered to be a possible flavor significance because heptanone-2 and pentanone-2 were found in heated whole milk but were undetectable in raw milk.

Patton and Tharp (1959) studied odor caused by heat treatment on milk fat and found an odor indicative of heptanone-2 which was in agreement with Wong, et al. (1958). Hawke (1957) who analysed the fatty acids of New Zealand butterfat and the volatile acids formed on oxidation, found formic acid as a large portion of these acids with smaller proportions of propionic, valeric and nonanoic acids. Madsen, et al. (1958) studied malty flavor in Danish butter. A chromatographic study of steam distillates of malty cultures revealed the presence of 3 methyl-butanol, a compound not found in steam distillates from nondefective starters. Forss, et al. (1960) studied the volatile compounds causing fishiness in butterfat with the help of gas chromatography on silicone oil and "carbowax 400" columns. Two fractions with distinct flavors were obtained, the one of oily flavor containing n-hexanal, n-heptanal, hex-2-enal and heptan-2-one.

The other, a metallic flavor, contained a single carbonyl compound in a relatively small amount. They considered these compounds with the exception of heptan-2-one are mainly responsible for the characteristic fish-oil flavor.

Results of the gas chromatographic fractionation of the off-flavor compounds on a silicone oil column:

Fraction	Compounds isolated in fraction	Flavor of fraction in butterfat
(1)	n-Pentanal, pentan-2-one, pent-2-enal	--
(2)	n-Hexanal, hex-2-enal, n-heptanal, heptan-2-one	strong oily
(3)	Hept-2-enal, "mushroom compound"	mushroom
(4)	Metallic compound	strong metallic
(5)	Trace "metallic compound" n-octanal, hepta-2, 4-dienal	slight metallic cardboard
(6)	Oct-2-enal, n-nonanal, nonan-2-one, octa-2, 4-dienal like compound	slight tallowy
(7)	Non-2-enal	cucumber
(8)	n-Decanal, undecan-2-one	--
	Fishy flavor chromatographed on carbowax 400	
(1)	Propanal	--
(2)	n-Pentanal, pentan-2-one	--
(3)	n-Hexanal, pent-2-enal, n-heptanal, heptan-2-one, hex-2-enal	strong oily
(4)	n-Octanal, "mushroom compound", hept-2-enal	mushroom
(5)	"Metallic compound" n-nonanal nonan-2-one	strong metallic
(6)	Oct-2-enal, hepta-2, 4-dienal, n-decanal, non-2-enal, undecan-2-one	cucumber tallowy

EXPERIMENTAL PROCEDURE

Salted butter from Michigan State University Dairy Plant and unsalted butter of McDonald Dairy were obtained for the experiments. Hobart machine, model A120 of ½ H.P. capacity, and a stainless steel bowl having 10-quart capacity, was used for whipping the butter. An A120B beater was used at a speed of 90 r.p.m. The butter to be whipped was held at 70° F. for 48 hours to temper it prior to whipping. The equipment in contact with butter was standardized to 70° F. also. To study the influence of temperature on whipping, the butter and equipment was stabilized at 60°, 65°, 70°, 75° and 80° F. For each trial 6 pounds of butter was whipped to obtain samples for 50, 75, 100 and 125 percent overruns.

An aluminum cup having a 1-pound butter holding capacity and a balance with a 500-gram capacity with a scale of 2 grams per division were used to obtain weights for calculation of the extent of the whipping of air into the sample. During the whipping process samples were weighed at regular intervals to check the amount of overrun. The sides of the bowl were scrapped regularly for a uniform whipping action on all the butter. The amount of overrun was calculated by the following formula:

$$\text{Percent overrun} = \frac{\text{Weight of butter--weight of equal volume of whipped butter}}{\text{Weight of equal volume of whipped butter}}$$

The butter was whipped first for 50 percent overrun and samples were taken in 1-pound wax-coated cartons for keeping quality and hardness test. Butter from same lot was also whipped to 75, 100 and 125 percent overrun and the samples were taken in the same manner. The whipped butter samples for keeping quality were held 52 weeks at 40° and 34° F. and for 64 weeks

at -10° F. The same procedure was followed for the trials on unsalted butter.

In an effort to prolong the keeping quality of whipped butter, antioxidants were added. The following antioxidants at the rate of 0.05 percent on butterfat basis were used:

	Antioxidants	Butterfat (percent)
(1)	NDGA	0.05
(2)	BHA	0.05
(3)	BHT	0.05
(4)	NDGA / BHA (0.025 / 0.025)	0.05
(5)	BHA / BHT (0.025 / 0.025)	0.05
(6)	NDGA / BHT (0.025 / 0.025)	0.05
(7)	NDGA / BHA / BHT (0.016 / 0.017 / 0.017)	0.05

Monoglyceride (Myverol 1800) in granular form was also added at the rate of 0.5 percent. NDGA was in a powder form; BHA and BHT were small crystals.

Approximately 100 grams of butter from the 6 pounds of butter for whipping were placed in a beaker and the antioxidant was added. It was dispersed while warming the mixture in a waterbath with constant stirring. When adequately dispersed the mixture of butter and antioxidant was added to the butter in Hobart bowl and the whipping was begun immediately. Samples were taken, weighed and returned to the bowl during whipping in order to terminate whipping when the correct amount of air had been incorporated.

Samples of whipped butter held at 34° and 40° F. were scored by a skilled judge 24 hours after whipping and thereafter at 4-week intervals for 52 weeks.

Samples held at -10° F. were scored at 8-week intervals for 64 weeks. The scoring for flavor, body and texture and color was patterned after the American Dairy Science Association butter score card which designates a maximum of 45 points for flavor, 30 for body and texture and 5 for color.

As an index of spreadability the hardness of whipped butter was measured. Samples with different overruns and additives that had been held at various storage temperatures were tested with a Penetrometer.* The readings represented the depth in millimeter of a cone weighing 102.5 grams penetrated into the sample. Samples were held at 55° F. for 48 hours prior to testing (Huebner and Thomsen 1957a) and tests were conducted in a room at the same temperature. Three samples of each treatment were tested with the penetrometer by allowing the penetrometer cone to penetrate for 5 seconds into butter samples. The readings recorded were the averages of three observations for each treatment and were expressed in one-tenth of a millimeter.

The whipped butter samples which after storage showed pronounced off flavors were saved for subsequent gas chromatographic studies. A Perkin Elmer model 154-B chromatograph was used for attempting to fractionate the collected volatiles. Figure 1 shows a schematic flow diagram of the apparatus. A continuous flow of carrier gas (helium) was supplied to the system from gas cylinder. It was maintained at a constant rate by a dual-stage pressure regulator. The helium was passed through a flowmeter and the reference side of the detector to the sample injection location. Gases were metered directly into the stream without heating. Liquids

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*Manufactured by Precision Scientific Company, Chicago, Illinois.

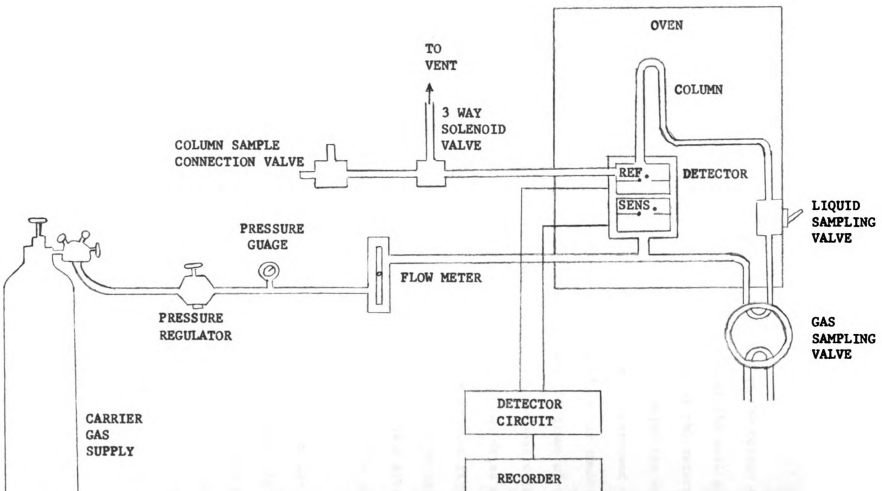

Fig. 1. Flow schematic of the vapor fractometer.

samples were injected into a heated chamber which served to vaporize the sample rapidly, whereupon the carrier-gas stream moved the vaporized material onto the column.

The components of the mixture were carried through the column at different speeds. They ultimately arrived separately at the exit of the column which is directly connected to the sensing side of the detector. The two detector sides were part of a bridge circuit of which the unbalance was constantly measured by a standard recorder. "Carbowax 400" (a polyethylene glycol, molecular weight 400) was used as the immobile phase. The column was prepared in the following way: 35 grams of crushed C23 firebrick, 30 to 60 mesh, were heated to 572° F., washed with concentrated hydrochloric acid, then washed free of acid and dried at 302° F.; 15 grams of "Carbowax 400" were mixed with the crushed firebrick and ethyl ether was added to insure that every particle would be uniformly coated with "Carbowax 400"; the ethyl ether was removed on a steam-bath, leaving an almost dry mixture which was dried further in an oven at 446° F.

Twenty grams of the packing material were poured into a 6-foot, ¼ inch (O.D.) copper tubing and packed while constantly vibrating the tubing with an electric vibrator. After plugging the ends with glass wool the column was bent into a W shape. As a precautionary measure the column was flushed with helium for several hours at a temperature of 266° F. to remove any contamination. The conditions that were established to test the samples were:

(a) temperature of column, 130° F.;

(b) voltage, 8;

(c) sensitivity, 4;

(d) flow of helium, 90 ml./min.;

(e) sample injected when liquid, 1 to 5 microliter; and

(f) gas sample metered through gas sampling valve, 10 seconds.

Steam distillation of approximately a 1000-gram butter sample was used to collect the off-flavor volatile compounds. Steam was bubbled into the sample for about 1.5 hours to 2.0 hours while keeping the flask in a constant warm waterbath. The steam distillate sample was passed through a fractionating column and through a condenser and was collected in 100 ml. of petroleum ether kept in an iced waterbath. The sample collected with petroleum ether was dried over sodium sulphate for 12 to 15 hours. The sample was separated from salt solution by distillation. The distillate sample was concentrated to small quantity by vacuum at room temperature. The sample of 1 to 5 microliter was injected in gas chromatogram. As no fractionation was obtained in first procedure, ethyl ether was used in place of petroleum ether and the other conditions were kept the same.

Nawar and Fagerson (1960) observed that conventional distillation and extraction with immiscible solvents have not proved universally satisfactory because of loss of sample during solvent removal, contamination introduced by solvents, difficulty in transferring small samples for injection without dilution and unavailability of a single immiscible solvent suitable for the wide range of composition of volatiles generally encountered. Sweeping the sample with a gas or vapor stream and condensing the volatiles from the stream in a cold trap offer advantages but required several traps for efficient collection. Use of multiple traps was a serious disadvantage in subsequent sample injection.

The sample collection system advocated by Nawar and Fagerson (1960)

was tried; 2000 grams of butter having the off flavor was placed in a flask and was uniformly heated at 140° F. for 2 hours. Nitrogen was passed through the sample of off-flavored butter and it was recirculated by a sigmamotor connected to a stainless steel tube kept in liquid nitrogen, in which a sample of volatiles was collected. Anhydrous potassium carbonate was used for absorption of moisture from the volatiles going to liquid nitrogen trap. The sample of volatiles in a gaseous form was injected for 10 seconds at intervals of 20 minutes for 5 times. Each trial for each off flavor was repeated six times.

RESULTS

The importance of the temperature of butter during whipping time is shown by the data on Table 1. The average times required to incorporate sufficient air for 50 percent overrun were 6.45, 8.75 and 16.16 minutes when the butter temperatures were 70° , 65° and 60° F., respectively. These same temperatures of butter corresponded with 43.30, 19.85 and 16.35 minutes of whipping to get 125 percent overrun, respectively.

If the butter was tempered to 75° F. difficulty was experienced in obtaining 100 percent overrun. At 80° F. the butter was much too soft to whip satisfactorily to 50 percent overrun with the Hobart equipment using a batch method.

The average body and texture score of salted butter having 50 percent overrun held 24 hours after whipping was 24.50. When the incorporated air was increased to 75, 100 and 125 percent, the average scores were 24.40, 24.0 and 23.75, respectively. The difference in score was 0.75 between the samples of 50 and 125 percent overrun. The main criticism of the body and texture of the different whipped butters was crumbliness which increased in magnitude as the air content increased from 50 to 125 percent in the samples. Score of the body and texture of unsalted butter 24 hours after whipping to 50 percent overrun was 24.5 and for 125 percent overrun was 24.0. After 52 weeks of holding at 40° F., the average score for body and texture of the salted whipped butter at 50 percent overrun was 24.30. The samples having 125 percent overrun had a score of 24.0. The samples (50 percent overrun) held at 34° F. for 52 weeks had an average score of 24.10 and 23.70 for samples with 125 percent overrun. The samples held at -10° F. for 64 weeks averaged 24.10, 24.0,

23.90 and 24.10. This included samples having 50, 75, 100 and 125 percent overrun, respectively. Many samples, especially those which scored 24 or less, were criticized as mealy. The samples which scored above 24.0 were criticized as slightly crumbly.

TABLE 1—Time required to obtain desired overrun at different whipping temperatures.

(Average of four trials)

Percent overrun	Butter tempered to: 60° F. Time (min.)	60° F. Temperature end of whipping (degrees F.)	65° F. Time (min.)	65° F. Temperature end of whipping (degrees F.)	70° F. Time (min.)	70° F. Temperature end of whipping (degrees F.)
50	16.16	60.0	8.75	65.0	6.45	70.5
75	21.30	61.0	11.65	65.5	9.15	70.5
100	29.38	61.0	17.00	66.0	11.35	71.0
125	43.30	61.0	19.85	66.0	16.35	70.0*

*When whipped for 20 minutes 125 percent overrun was not obtained. If cooled to 68° F. 125 percent overrun was obtained in 5 minutes.

The average body and texture score for the samples of unsalted, whipped butter held at 40° F. for 52 weeks was 24.50 for 50 percent overrun; for 125 percent overrun the average was 24.0 and had the same defects that were observed for salted butter. The average body and texture score for the unsalted butter held at 34° F. for 52 weeks ranged from 24.30 to 24.10 for the samples with 50 to 125 percent overrun. The samples held at -10° F. for 64 weeks showed the same score of 24.0 for body and texture for the 4 different overruns.

The color of whipped butter (salted or unsalted) with 50, 75, 100 and 125 overruns tended to get uniformly lighter as the overrun increased.

Samples with 125 percent overrun were very light yellow, even though the original butter was medium yellow. None of the samples with the four overruns showed a decrease in score of color during storage for 64 weeks at -10° F.

Butter whipped to 50, 75, 100 and 125 percent overruns and held at 34° or 40° F. for 52 weeks showed surface bleaching. The amount of bleaching from yellow to white increased with an increase in the percentage of overrun. First bleaching in the samples at 40° F. was evident upon examination after 12 weeks and at 34° after 16 weeks. Samples with NDGA antioxidants as additive showed a dull color.

Data in Table 2 show the average flavor score for the samples of salted whipped butter with 50, 75, 100 and 125 percent overruns held at 40° F. for 52 weeks. The samples evaluated 24 hours after whipping were given a score of 38 for the four overruns.

After 4 weeks at 40° F. the average flavor score for the samples having 50 percent overrun was 37.9. The scores for the samples which had 75, 100 and 125 percent overruns were 37.4, 36.6 and 36.0. The difference in the flavor score between those having 50 percent overrun and 125 percent overrun was 1.9. This difference decreased to 0.9 after 20 weeks. At the end of 52 weeks the average flavor scores were 33.2, 33.1, 32.5 and 31.8 for 50, 75, 100 and 125 percent overruns, respectively. The average difference between 50 and 75 percent overrun was negligible (0.1), between 50 and 100 percent was 0.6 and between 50 and 125 percent was 1.4. The flavor defect attributed to the samples having 50 percent overrun generally was rancid. Samples with 125 percent overrun were criticized for the same defect but was more intense.

The flavor evaluation results of whipped salted butter held at 34° F.

are presented in Table 3. The average flavor score for samples having 50 percent overrun remained at 38 for the 12 weeks, while a decrease of 0.3, 0.6 and 1.4 were observed for 75, 100 and 125 percent overrun samples. At the end of 52 weeks the average flavor scores were 34.5, 34.1, 33.8 and 32.5 for 50, 75, 100 and 125 percent overruns, respectively. The samples having 50 percent overrun generally showed storage off flavor while samples with 125 percent overrun usually were rancid. Some samples with 75 and 100 percent overruns had a storage defect and some were rancid.

TABLE 2—Keeping quality of salted butter whipped to various percentage of overrun and held at 40° F.

Storage (weeks)	Percent overrun			
	50	75	100	125
	Average flavor score of 6 trials			
0	38.0	38.0	38.0	38.0
4	37.9	37.4	36.6	36.0
8	36.5	36.5	36.1	35.3
12	35.6	36.0	35.5	35.0
16	35.0	34.8	34.6	34.0
20	34.1	34.5	33.6	33.0
36	34.6	35.0	33.6	33.1
52	33.2	33.1	32.5	31.8

Most samples from the same batchs retained a better flavor when stored at the 34° than at 40° F. Flavor scores for the samples held at 34° F. were 34.5, 34.1, 33.8 and 32.5 as compared to 33.2, 33.1, 32.5 and 31.8 for samples held at 40° F. for 50, 75, 100 and 125 percent overrun, respectively.

TABLE 3—Keeping quality of salted butter whipped to various percentage of overrun and held at 34° F.

Storage (weeks)	Percent overrun			
	50	75	100	125
	Average flavor score of 6 trials			
0	38.0	38.0	38.0	38.0
4	38.0	38.0	37.8	37.2
8	38.0	37.9	37.7	37.3
12	38.0	37.7	37.4	36.6
16	37.0	36.6	36.6	35.3
20	36.3	35.8	35.8	34.6
28	34.7	35.0	34.8	34.1
52	34.5	34.1	33.8	32.5

Data (Table 4) show the keeping quality of whipped salted butter held at -10° F. The average flavor score remained at 38 for all samples during 32 weeks. The decrease in score from 38 to 37.5 at the end of 40 weeks was uniform for samples having the 4 different percentages of overrun. At the end of 64 weeks the samples had an average score of 36.1, 35.9, 35.3 and 35.2 for 50, 75, 100 and 125 percent overruns, respectively. The keeping quality of the samples at -10° F. was much better than the similar samples held at 34° and 40° F. This is shown in Figure 2. Figures 3, 4, 5 and 6 illustrate the flavor score of salted butter that was whipped to 50, 75, 100 and 125 percent overruns, respectively, and held 52 weeks at various temperatures.

Observations on keeping quality of unsalted butter held at 40° F. are presented in Table 5. The average flavor score 24 hours after whipping was 36 with comment of coarse. The flavor score of butter with 50

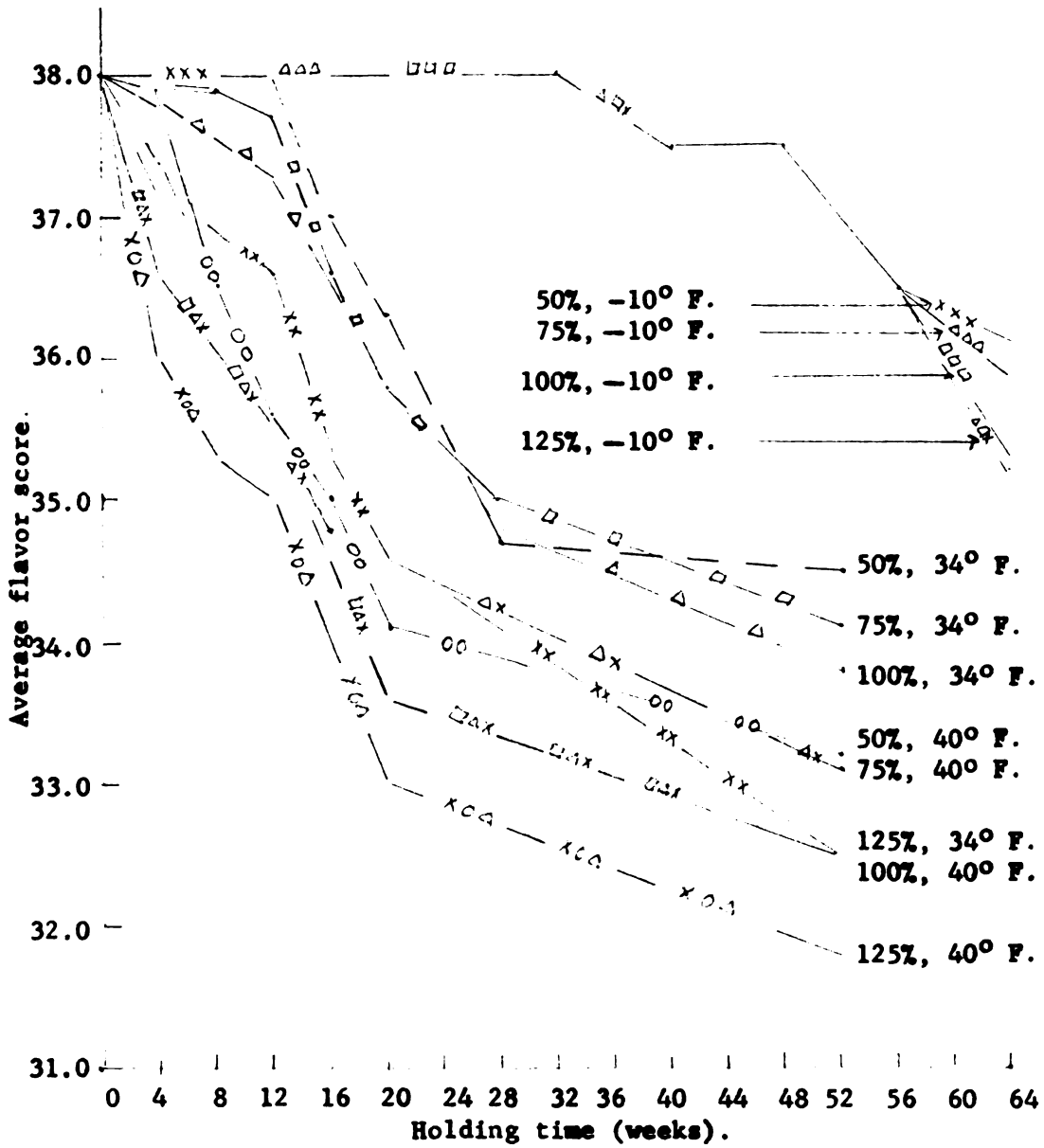

Fig. 2. Flavor score of whipped salted butter having 50, 75, 100 and 125 percent overrun and held at 40°, 34° and -10° F.

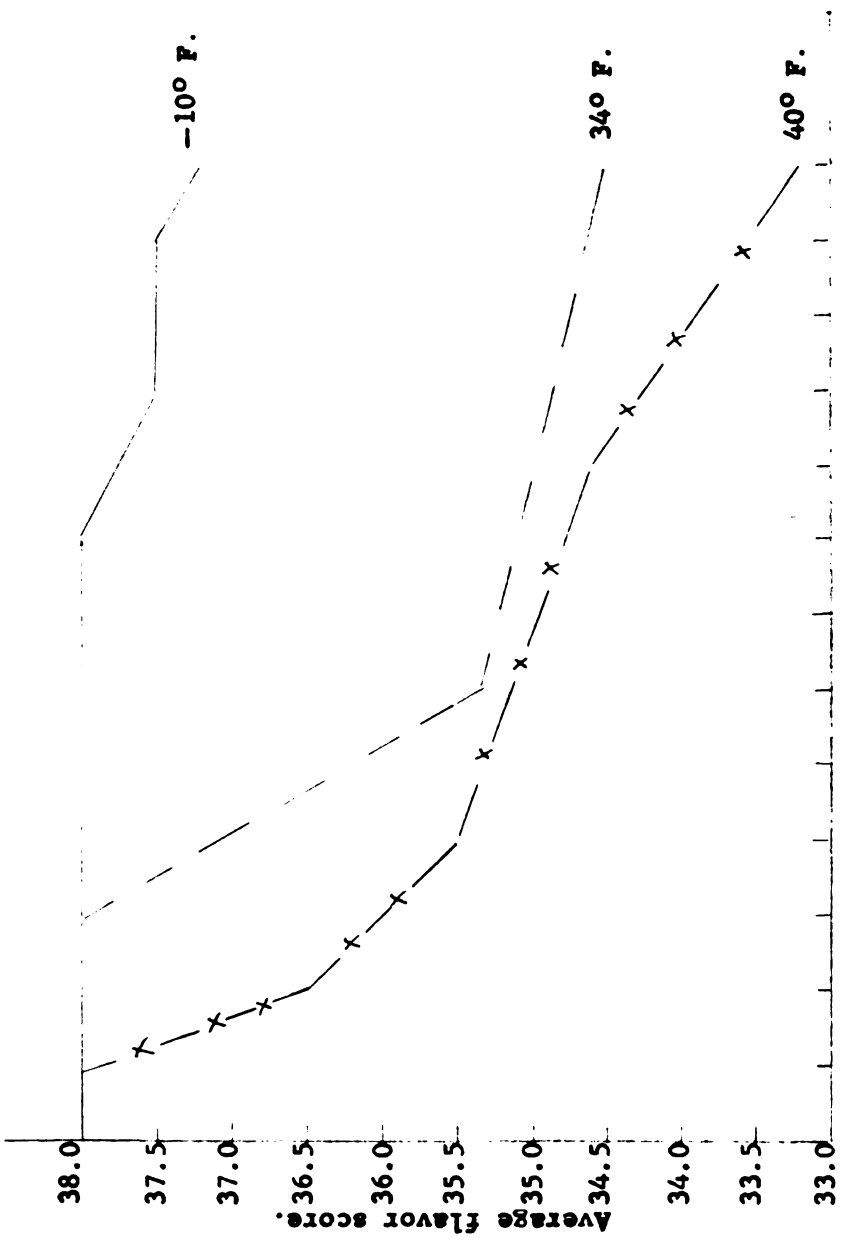

Fig. 3. Flavor score of whipped salted butter having 50 percent overrun and held at 40°, 34° and -10° F.

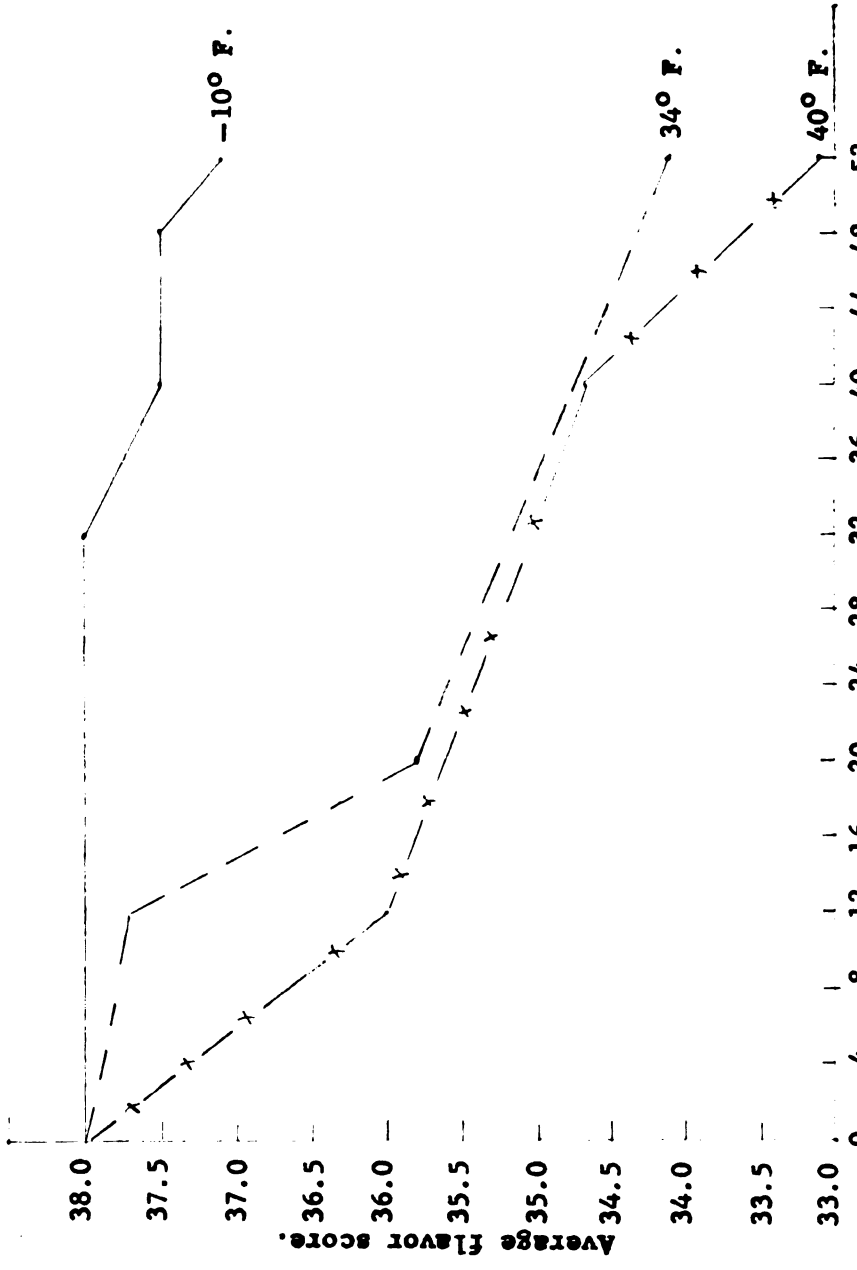

Fig. 4. Flavor score of whipped salted butter having 75 percent overrun and held at 40°, 34° and -10° F.

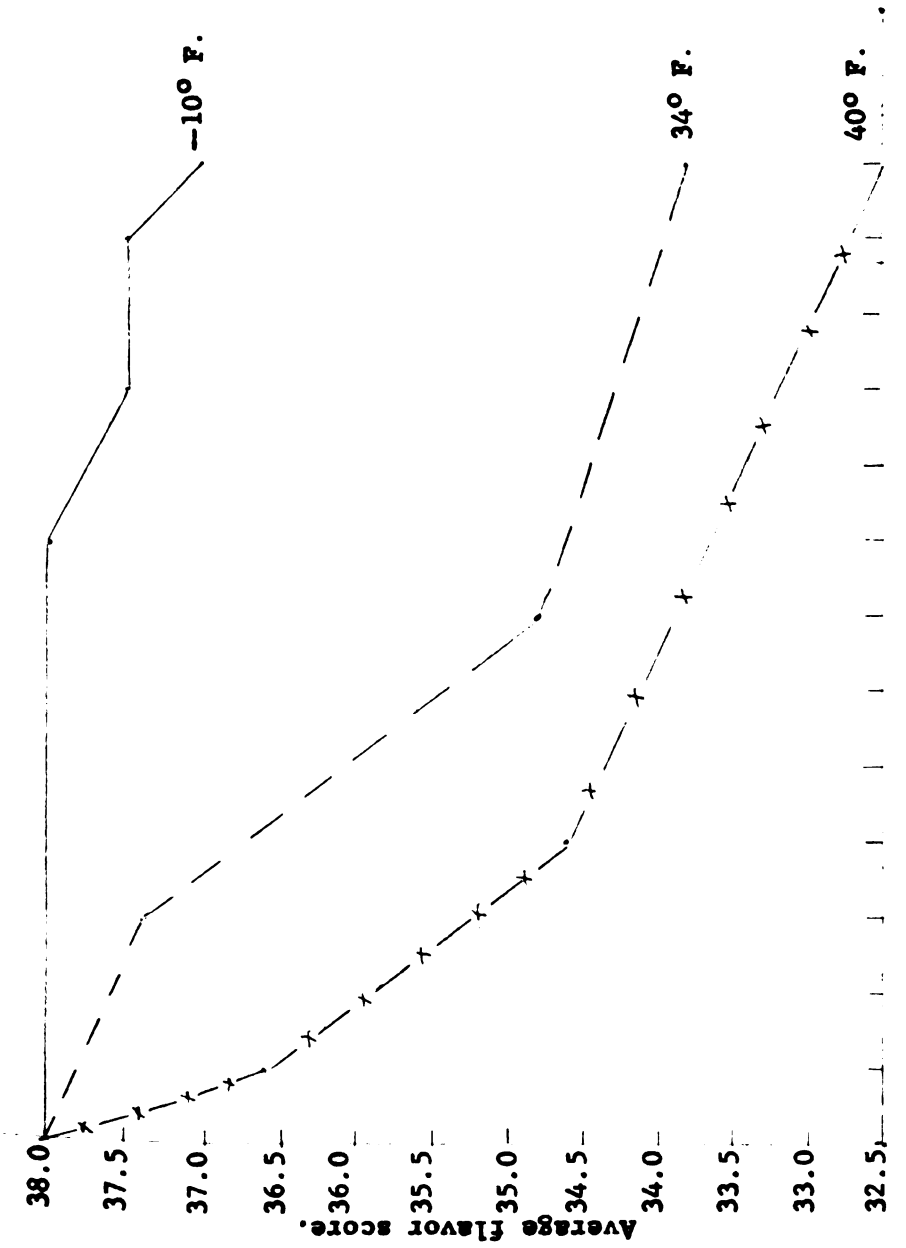

Fig. 5. Flavor score of whipped salted butter having 100 percent overrun and held at 40°, 34° and -10° F.

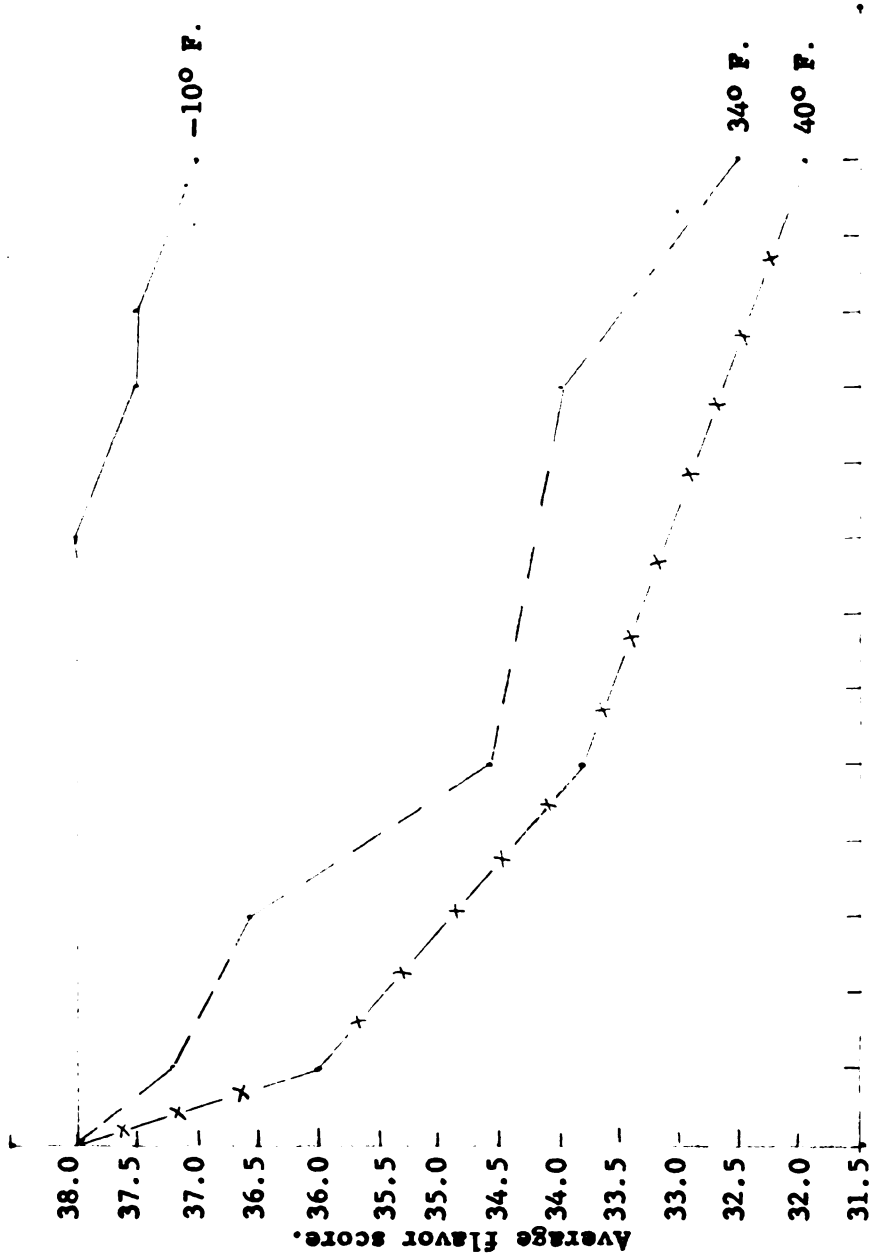

Fig. 6. Flavor score of whipped salted butter having 125 percent overrun and held at 40°, 34° and -10° F.

percent overrun remained 36.0, while the samples with 75, 100 and 125 percent overrun scored 35.5, 35.1 and 35.4 after 8 weeks. The average score decreased to 34.1 for 50, to 30.5 for 75, to 33 for 100 and to 32.5 for 125 percent overruns of samples by the end of 28 weeks. At the end of 44 weeks all the samples showed pronounced rancid flavor. The average scores ranged from 31.0 for samples of 50 percent overrun to 30.0 (lowest score assigned) for the samples with 75, 100 and 125 percent overruns.

TABLE 4—Keeping quality of salted butter whipped to various percentage of overrun and held at -10° F.

Storage (weeks)	Percent overrun			
	50	75	100	125
	Average flavor score of 6 trials			
0	38.0	38.0	38.0	38.0
4	38.0	38.0	38.0	38.0
8	38.0	38.0	38.0	38.0
16	38.0	38.0	38.0	38.0
24	38.0	38.0	38.0	38.0
32	38.0	38.0	38.0	38.0
40	37.5	37.5	37.5	37.5
48	37.5	37.5	37.5	37.5
56	36.5	36.5	36.5	36.5
64	36.1	35.9	35.3	35.2

Results in Table 6 show the keeping quality of unsalted butter with a different percentage of overrun held at 34° F. The initial average score of 36.0 remained unchanged for 12 weeks for samples having 50 and 75 percent overrun, but decreased to 35.0 and 34.8 for 100 and 125 percent overrun samples. At the end of 52 weeks, the flavor score was 33.9

31.0, 30.0 and 30.0 for 50, 75, 100 and 125 percent overruns, respectively. The flavor defect was rancid.

TABLE 5—Keeping quality of unsalted butter whipped to various percentage of overrun and held at 40° F.

Storage (weeks)	Percent overrun			
	50	75	100	125
	Average flavor score of 6 trials			
0	36.0	36.0	36.0	36.0
4	36.0	36.0	36.0	36.0
8	36.0	35.5	35.1	35.4
12	35.5	34.9	34.8	34.8
16	34.8	33.2	34.0	34.1
20	34.7	30.7	33.0	33.8
24	34.6	30.5	33.0	33.0
28	34.1	30.5	33.0	32.5
44	31.0	30.3	30.6	30.0
52	31.0	30.0	30.0	30.0

Flavor scores for unsalted whipped butter held at -10° F. are presented in Table 7. A flavor score of 36 was given to samples of all four overruns initially after whipping. The score of the samples with 50 percent overrun remained unchanged for 64 weeks at -10° F. Average flavor scores of 35.6, 35.5 and 35.5 were given to samples having 75, 100 and 125 percent overruns at the end of 64 weeks. Figures 7 and 8 illustrate graphically the comparison of flavor scores of unsalted butter whipped to 50 and 75 or to 100 and 125 overruns and held in storage at 40°, 32° and -10° F.

TABLE 6—Keeping quality of unsalted butter whipped to various percentage of overrun and held at 34° F.

Storage (weeks)	Percent overrun			
	50	75	100	125
	Average flavor score of 6 trials			
0	36.0	36.0	36.0	36.0
4	36.0	36.0	36.0	36.0
8	36.0	36.0	35.4	36.0
12	36.0	36.0	35.0	34.8
16	34.9	35.3	34.3	34.3
20	34.4	34.9	34.1	34.1
24	34.4	34.6	34.0	33.9
28	34.2	33.8	33.3	33.6
36	34.2	33.8	33.3	33.8
52	33.9	31.0	30.0	30.0

TABLE 7—Keeping quality of unsalted butter whipped to various percentage of overrun and held at -10° F.

Storage (weeks)	Percent overrun			
	50	75	100	125
	Average flavor score of 6 trials			
0	36.0	36.0	36.0	36.0
4	36.0	36.0	36.0	36.0
8	36.0	36.0	36.0	36.0
16	36.0	36.0	36.0	36.0
24	36.0	36.0	36.0	36.0
32	36.0	36.0	36.0	36.0
40	36.0	36.0	36.0	36.0
48	36.0	36.0	36.0	36.0
56	36.0	36.0	36.0	35.9
64	36.0	35.6	35.5	35.5

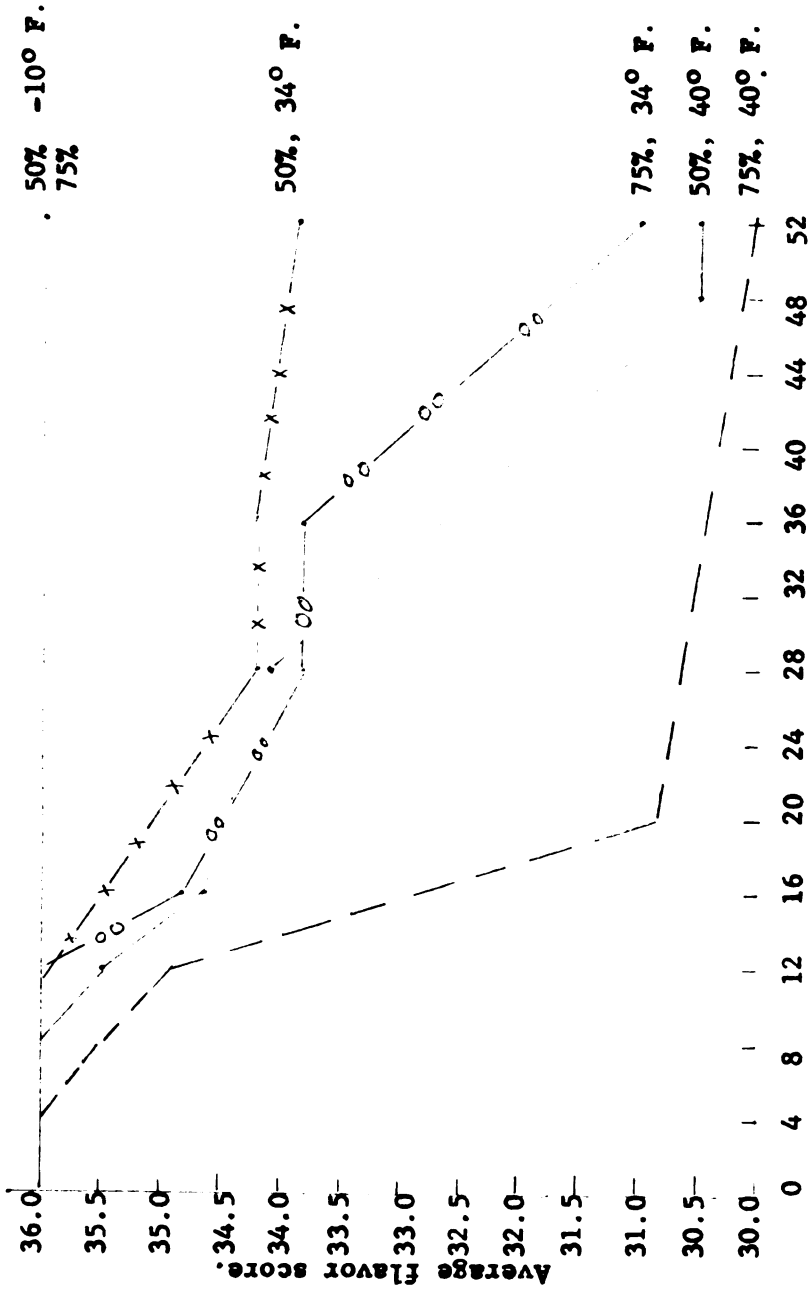

Fig. 7. Flavor score of whipped unsalted butter having 50 and 75 percent overrun and held at 40°, 34° and -10° F.

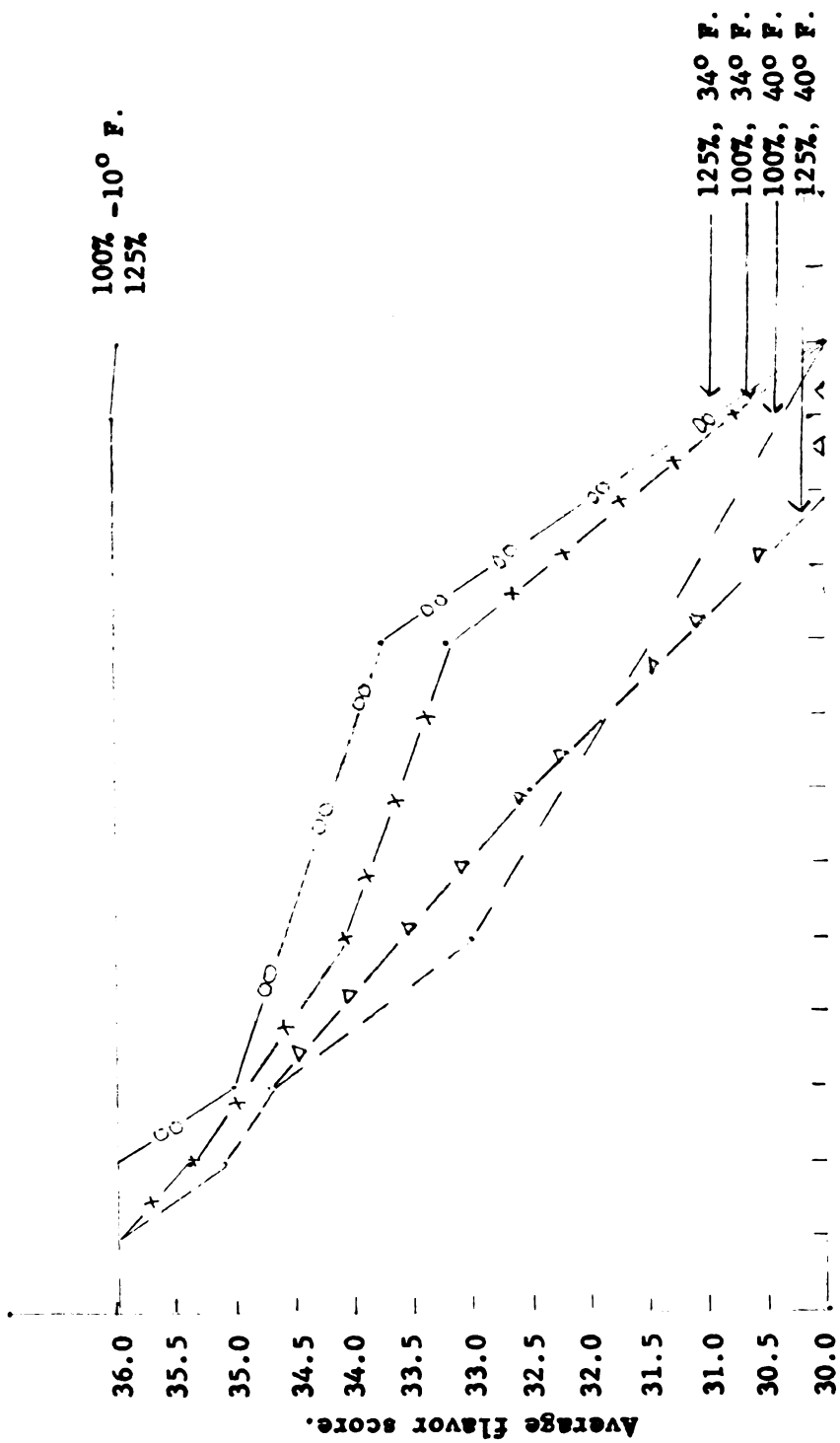

Fig. 8. Flavor score of whipped unsalted butter having 100 and 125 percent overrun and held at 40°, 34° and -10° F.

Average flavor scores for samples containing 0.05 percent addition of various antioxidants have been recorded in Table 8. The score after whipping to 50 percent overrun, varied from a low of 36.6 for samples with NDGA, to a high of 37.9 for those with BHA, NDGA and BHA and a mixture of equal amounts of NDGA, BHT and BHA. NDGA caused a medicinal (or foreign) off flavor in whipped butter. After 16 weeks at 40° F. the average flavor score for the samples having monoglyceride was 34.6, while the range for the others was from 35.3 (NDGA) to those with BHA / BHT, which scored 36.7. At the end of 52 weeks the highest average flavor score was 34.9 for the samples having NDGA / BHT, while the lowest was the 33.4 average given to the samples having NDGA. Flavor comment was "storage" for the control samples, "rancid" for samples containing monoglyceride and "medicinal" for samples containing 0.05 percent NDGA. Figure 9 presents a graphic comparison of the influence of the various antioxidants in storage during 52 weeks.

The data showing the penetrometer readings expressed in one-tenth mm. are presented in Table 9. A soft whipped butter gives a high reading. The control samples of salted butter (not whipped) held at 40°, 34° and -10° F. for 20 weeks then tempered to 55° F. showed average penetrometer readings of 40, 36 and 34, respectively. Samples having 50, 75, 100 and 125 percent overruns and held at 40° F. for 20 weeks showed averages of 68.6, 83.6, 92.0 and 95.6, respectively. Samples with the same overruns held at 34° F. showed averages of 70.3, 88.6, 97.3 and 99.0, respectively. When samples stored at -10° F. were examined after tempering to 55° F. the results were 74.0, 89.0, 103.0 and 104.0 (average penetrometer readings) for 50, 75, 100 and 125 percent overruns, respectively.

Control unsalted samples showed 35.0, 35.0 and 38.0 penetrometer

readings for the samples held at -10°, 34° and 40° F., respectively, for 20 weeks. The samples held at 40° F. having 50, 75, 100 and 125 percent overruns showed 88.0, 94.0, 101.0 and 113.0 penetrometer reading for respective overruns as compared to 92.0, 100.6, 101.3 and 116.3 averages for the similar overruns held at 34° F. Samples held at -10° F. of 50, 75, 100 and 125 percent overruns showed 93.6, 98.3, 102.6 and 123.0 penetrometer readings, respectively (Figure 10).

TABLE 8—Keeping quality of salted butter whipped to 50 percent overrun with various antioxidants and held at 40° F.

Storage (weeks)	Control	NDGA	BHA	BHT	NDGA BHA BHT	NDGA BHA	NDGA BHT	BHA BHT	Mono-glyceride
	Average flavor score of 6 trials								
0	37.7	36.6	37.9	37.7	37.9	37.8	37.9	37.6	37.8
4	37.6	35.9	37.5	37.5	37.7	37.6	37.8	37.6	37.5
8	36.6	35.6	37.2	37.1	37.4	37.4	37.6	37.5	36.5
12	36.0	35.3	36.4	36.5	36.6	36.3	37.1	37.0	35.1
16	35.6	35.3	36.1	35.8	36.3	36.5	36.6	36.7	34.6
20	34.8	34.8	35.4	35.5	35.6	35.8	35.9	36.0	34.1
32	34.8	33.3	34.5	34.8	34.4	34.7	34.9	35.0	32.1
52	34.1	33.4	34.7	34.6	34.4	34.6	34.9	34.6	34.0

The effect of various additives on hardness of butter is shown in Table 10. The control sample (not whipped) for these trials showed an average penetrometer reading of 40.6 while butter having 50 percent overrun but no additive showed 70.6 penetrometer reading. The samples having 50 percent overrun and additives of NDGA / BHA or NDGA alone showed 64.0 and 74.6 penetrometer readings. The other results of samples with various additives were within the range of these readings.

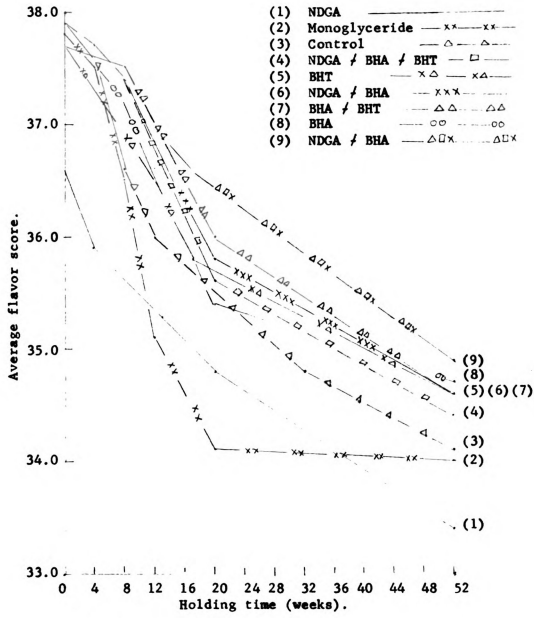

Fig. 9. Keeping quality of salted butter whipped to 50 percent overrun with various antioxidants and held at 40° F.

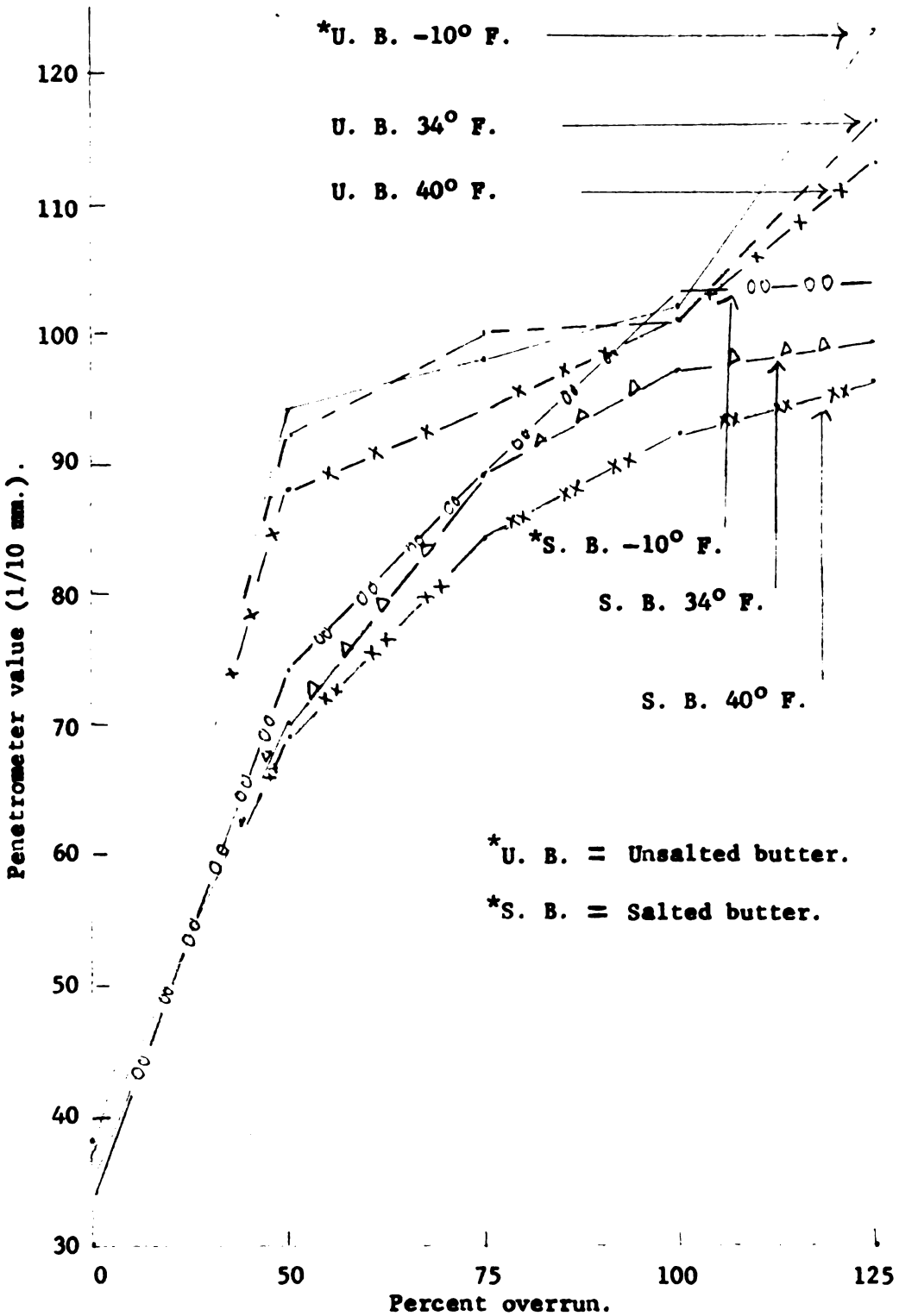

Fig. 10. Hardness of whipped butter having various percentage of overrun and held at different storage temperatures.

TABLE 9—Hardness of whipped butter having various percentages of overrun and different storage temperatures.

Overrun (percent)	Average penetrometer value of 3 trials 1/10 mm.[1]					
	Salted butter			Unsalted butter		
	-10° F.	34° F.	40° F.	-10° F.	34° F.	40° F.
0	34.0	36.0	40.0	35.0	35.0	38.0
50	74.0	70.3	68.6	93.6	92.0	88.0
75	89.0	88.6	83.6	98.3	100.6	94.0
100	103.0	97.3	92.0	102.6	101.3	101.0
125	104.0	99.0	95.6	123.0	116.3	113.0

[1]Tested in triplicate after 20 weeks of storage.

TABLE 10—Effect of different additives on hardness of butter whipped to 50 percent overrun and held at 40° F.

Average penetrometer value of 3 trials 1/10 mm.									
Control	No additive	Mono-glyceride	NDGA	BHA	BHT	NDGA BHA BHT	NDGA BHA	NDGA BHT	BHA BHT
40.6	70.6	68.0	74.6	72.0	72.0	72.0	64.0	64.0	65.0

A sample was collected from 1000 grams of fishy butter into 100 ml. of petroleum ether and was further concentrated to 5 ml.; a 5-microliter sample was analyzed but no peaks were obtained. No peaks resulted from a similar procedure using pronounced oxidized butter or pronounced rancid butter. The same negative results were observed when ethyl ether was substituted for petroleum ether. Negative results were obtained for each of six repeated trials.

Trials involving rancid, oxidized or fish butter investigated by the method of Nawar and Fagerson (1960) also failed to show any peak on the Chromatogram for six trials of each.

DISCUSSION

The temperature of the butter is important in obtaining overrun and materially affects the time required to whip by the Batch Method using a standard Hobart beater and bowl. The optimum for the butter in these trials was approximately 70° F. Butter with a cooler temperature of 65° or 60° F. can be whipped up to at least 125 percent overrun but the whipping time was increased substantially. Apparently the butter must be softened to a certain degree for physical retention of the small air cells.

Butter tempered to 75° F. was slow in attaining the overrun and the maximum attainable was reduced in comparison with butter whipped at 70° F. Too soft a butter looses its ability to physically retain air cells due to more liquification of the fat.

There was a tendency for the whipped butter with the high overruns to develop mealiness during prolonged storage at above freezing point temperatures of 32° and 40° F. The cause was not apparent and should be worthy of investigation.

The effect of temperature of storage on keeping quality of whipped butter correlated well with the experience in handling regular butter, both salted and unsalted. However, deterioration of whipped butter was more rapid, especially at 40° and 32° F. than the control. The amount of air obviously influenced the change, but the rate of chemical and enzymatic reaction was decreased with the decreasing temperature from 40°, to 32° to -10° F. Meyknecht and Dam (1959) observed similar results in trials with regular butter.

The amount of air as indicated by 50, 75, 100 and 125 percent overruns materially influenced flavor score of whipped butter in storage.

The rate increased with an increase in air content. The greater availability of oxygen (more air cells) and the greater surface exposure of the fat to air was thought to be responsible for the effect of the amount of air. Holman, et al. (1954) reported that air, as well as temperature and light, caused the development of rancidity in edible fats by the action of lipase.

Although the influence of salt was not studied directly, the type of chemical reaction did not appear to be affected substantially by the salt which was not above 1.5 percent in the butter. A small effect would not be noticeable in the limited number of trials without using identical butter for salted and unsalted trials.

Conflicting claims on the value of antioxidants can be found in literature. The explainations are varied such as type of reaction involved, dispersibility or solubility of antioxidant and flavor effect of the antioxidant per se, etc.

NDGA was not satisfactory for whipped butter because it contributed an off flavor of the medicinal type. Pederson, et al. (1955) noticed the same effect in regular butter. The mechanism by which the beneficial effects of BHT, BHA and various combinations with small amounts of NDGA has been explained by Tollenaar and Vos (1959) and Gelpi, et al. (1952). These authors have reported on the retarding effects of oxidative changes on these compounds in milk fat containing products such as cream, butter and dry whole milk.

Specific tests for spreadability have not been devised with much accuracy. Other tests have been used as hardness measured by the penetrometer to indicate spreadability. Whipped butter penetrometer values increase directly with the percentage of overrun. This phenomenon is

anticipated on the basis of the decreased density of whipped butter with higher overruns. Temperature changes would undoubtedly be directly proportional to the overrun, a fact that would favor better spreadability on short holds at room temperature after removal of whipped butter from the refrigerator.

Whipped butter held at -10° F. showed less hardness than duplicate samples held at 40° F. before tempering both to 55° F. A reason for this may be due to the difference in crystallization of the fat at the two holding temperatures. Holding at 40° F. would allow large crystals to form which may have great structure strength. A hold at -10° F. may inhibit crystalization or encourage formation of a small size resulting in less structure strength or hardness.

SUMMARY AND CONCLUSIONS

Butter can be whipped by a batch procedure to an overrun of at least 125 percent. Optimum butter temperature for whipping was found to be approximately 70° F. Lower temperatures increased total whipping time and higher temperatures reduced the maximum overrun obtainable.

The body and texture score if rated by butter standards decreased as the overrun increased from 50 to 125 percent. The color became lighter, but was uniform.

The flavor deterioration of whipped butter was directly related to the percent of overrun and the increase in storage temperature. At 40° F. the flavor score of whipped butter decreased rapidly, especially with overruns of 100 and 125 percent. Changes at -10° F. were first observed upon completion of 40-week hold.

Keeping quality of whipped butter was slightly improved by the addition of 0.05 percent of BHA, BHT and/or combinations of these with NDGA.

As the overrun increased from 50 to 125 percent the hardness decreased. Storage of whipped butter at -10° resulted in slightly less hardness than storage at 40° F. for 20 weeks when both were tempered to 55° F.

Chromatographic analysis revealed no peaks for compounds responsible for oxidized, rancid or fishy flavors of butter.

In conclusion, whipped butter can be processed without difficulty by the batch method. Storage deterioration was rapid at temperatures above freezing.

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