ABSTRACT

RHEOLOGY AND STABILITY OF FREEZE-DRIED CULTURED CREAM

By

Morton Oswald Hamilton

Freeze-dried sour cream exhibiting highly desirable body and flavor characteristics following reconstitution was prepared from fresh cream (20% fat) fortified with non-fat dry milk (2%), caseinates (1%) and appropriate stabilizers (0.5%). By employing extended culturing with an active starter culture to a titratable acidity of 0.9-1.0% (as lactic) and a diacetyl content of 3-5 ppm, sufficient volatile flavor components were retained in the dehydrated product to yield excellent flavor. Losses of 50-60% of the original diacetyl content during drying and masking or dilution of the volatile aroma and flavor constituents required that higher final concentrations of these materials be present in the fresh cultured cream prior to freeze drying.

The cultured cream with added stabilizers and stabilizing agents was effectively freeze dried in ten hours at platen temperatures of 175F (80C). The "endpoint" of

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the freeze drying cycle was based on the rapid increase in the product temperature (at the bottom of the tray) to 75-80F (24C). The loss of diacetyl was therefore found to be a function of product, rather than platen temperature. Exposure of the dry product to high platen temperatures resulted in greater losses of diacetyl. The retention of diacetyl during the freeze drying process was consistently greater in the cultured cream containing added solids.

The Brabender visco/amylo/graph was adapted to evaluate differences in the rheological properties which accompanied change in formulation and processing conditions, because of its reproducibility and sensitivity to small changes.

The high free fat content of the freeze-dried cultured cream (95-99% of total fat) was not reduced either by high homogenization pressures (4000 psi) or by the use of high levels of emulsifiers (0.75%) encompassing the entire HLB range. The use of emulsifiers (0.2%) did improve the dispersibility of the powder.

The shelf life of the powder was greatly improved by:

1. Maintaining a reasonably low level of moisture (0.5-1.5%);
2. Use of appropriate antioxidants;
3. Packaging in an inert atmosphere; and
4. Storage at temperatures of 22-24C and lower.

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The loss of diacetyl from the stored powders was found to be minimal even under adverse conditions, provided the moisture content of the powder placed in storage did not exceed 2%. The viscosity and body of these reconstituted powders remained relatively unchanged in storage for up to 6 months.

Sensory evaluations showed the reconstituted freeze-dried cultured cream to be significantly better than the commercial spray dried preparations tested. There was however no significant difference between the reconstituted freeze-dried and the fresh cultured creams purchased in the supermarket.

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Morton Oswald Hamilton

A THESIS

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INTRODUCTION

Cultured cream generally referred to as sour cream by the consumer, is a smooth, viscous, creamy product resulting from the ripening of pasteurized sweet cream by a starter culture of desirable organisms, usually Streptococcous lactis, Leuconostoc citrovorum and Leuconostoc dextranium. Cultured cream is characterized by a clean acid flavor, but should also possess a desirable aroma of diacetyl and other flavor compounds. Fresh cultured cream, however, has a limited shelf life and may develop many defects, due to the use of poor raw materials, unsatisfactory culture, improper ripening or prolonged storage (Kosikowski, 1966).

The United States has seen a recent increase in the per capita consumption of cultured cream. This is due in part to increased use in cakes, cookies, salads, vegetables, and beef stroganoff. It is also a favorite item in the dairy industry because it is profitable and provides a good market for butterfat. In addition, the present trend toward "instant" or "convenience" type foods has encouraged the food industry to produce instant gourmet dishes for the working homemaker.

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During the past few decades interest in freeze-drying of foods has been increasing, due to the fact that freeze-drying usually produces a superior product from the standpoint of flavor, minimal protein denaturation, and excellent reconstitutability when compared to products dehydrated by other means.

The obvious advantages of freeze-drying have, therefore, stimulated this research project which was directed to the development of a dehydrated cultured cream of superior flavor, physical properties, and stability. Such a product would serve to overcome most of the shortcomings of the fresh cultured cream and would provide a convenient, uniform stable food, which could be packaged to meet the demands of institutions and the housewife.

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REVIEW OF LITERATURE

Freeze-drying, as the name implies, involves the two distinct operations of freezing and dehydration. To date many researchers have considered freezing only incidental to the process of dehydration (Burke and Decareau, 1964). On the other hand, biological research on the effects of freezing on cell viability has made increasingly apparent the fact that the freezing step is a particularly important phase of freeze-drying and may well be the most significant (Luyet, 1962). Burke and Decareau (1964) proposed two questions to be answered with regard to the freezing step:

1. What is the relation between freezing rate and the structure of the material to be freeze-dried?
2. What is the importance of the eutectic temperature in freezing prior to freeze-drying?

Freezing Rates

There is a difference of opinion as to the most desirable freezing rate for products that are to be freeze-dried. Evidence has been presented that supports both slow and rapid freezing. Rolfe (1958) stated that the fine pore

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structure obtained on quick freezing reduced the rate of drying. In addition, Rolfe pointed out that rehydration is more difficult after fast freezing because air trapped in the fine pores resists the penetration of water. Smithies (1962) claimed that rapid freezing, for example, in an acetone-dry ice mixture, resulted in poor rehydration. In meats, the slower rehydration has been attributed to a protein gel which forms in the external layer and resists penetration by water; consequently, total rehydration may require two or more hours.

McIlrath and Dekazos (1962) in studies with Swiss chard, obtained best rehydration of the freeze-dried product when freezing was rapid. Meryman (1962) summarized the effects of freezing and dehydration in the process of freeze-drying by suggesting that the main cause of tissue injury was the concentration of solutes rather than mechanical damage by ice crystals.

Eutectic Point

The eutectic temperature is that temperature at which crystallization or solidification of the aqueous solution is complete and further cooling does not introduce new structural changes (Rey, 1960). From this definition, it is apparent that it is only at the eutectic temperature or below that the chemical activity of salts or protein ceases. Rey (1960), therefore, recommended the following procedure for efficient freeze-drying:

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1. Determine the point of complete solidification.
2. Determine point at which incipient melting occurs.
3. Freeze to the temperature of complete solidification or below.
4. During the sublimation phase, dry at temperatures below the incipient melting point.

On the basis of this work, Rey and Bastien (1962) studied the danger of interstitial melting in the freeze-drying of dietary products, milk, and baby food. In some runs, interstitial melting caused hypertonic solutions which degraded the structure and the activity of sensitive nutrients. In this study, they also found that in spite of the high volatility of acetone, substantial amounts were strongly adsorbed by the dry material. Saravacos and Moyer (1968) also observed the strong adsorption of acetone to dry materials.

Harper and Tappel (1957) postulated that the chief advantage of freeze-drying would be in the low temperature of the process and its effect on the retention of flavor volatiles.

Changes in Milk and Milk Products Resulting from the Freezing Process

Since freeze-drying was initially applied mainly to biological material, comparatively little work has been published on freeze-dried dairy products. Recent research

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in the freezing and freeze-drying of foods has been directed to fruits, vegetables, and meats. The superiority of freezing as a means of preservation has been clearly shown with reference to these products (Tressler et al., 1968), but this is not necessarily true with dairy products. Whole milk consists essentially of a continuous phase, called serum, in which fat globules, calcium phosphocaseinate and other colloidal material are suspended. Several workers (Webb and Hall, 1935; Doan and Baldwin, 1936; and Dahle, 1942) have observed the disruption of the fat emulsion by freezing of milk systems which contain fat. According to Doan and Baldwin (1936) this disruption of the fat emulsion leads to the formation of a surface layer of fat aggregates in the thawed material which appears to be identical to fat aggregates brought about by churning. Webb and Hall (1935) found that freezing destabilized the fat emulsion even without storage. They also observed that the extent of aggregation and agglomeration was directly dependent on the fat content of the frozen milk product. Doan and Baldwin (1936) suggested that the emulsion breakdown during freezing was due to pressures within the frozen mass. Undoubtedly the disruption of lipid protein complexes in the fat globule membrane is involved in the mechanism of deemulsification.

Nickerson et al. (1952) also described the destabilization of the fat emulsion of fluid milk due to

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freeze-drying, resulting in a great increase in "free fat" and rather poor dispersibility properties. Although the initial flavor of the freeze-dried milk was better, no improvement in keeping quality was observed over spray-dried whole milk during storage. Lovelock (1957) and Powrie et al. (1963) have shown lipoproteins in other biological systems to be altered by freezing and thawing. The flocculation or precipitation of protein masses in thawed materials was evidence of the destabilization of proteins in frozen milk systems. Rutz et al. (1953), however, suggested that destabilization occurred during the drying process and not, as was generally believed, during the freezing cycle. Doan and Warren (1947) and Wildasin and Doan (1951) have shown the coagulated particles, or flocs, to be calcium caseinate which has properties similar to those salted-out from concentrated skimmilk.

Although the mechanism of flocculation has not been explored extensively, Doan and Warren (1947) have suggested that the flocculation of calcium caseinate micelles is due to the salting-out effect of the unfrozen liquid of the frozen product. Wildasin and Doan (1951) indicated that a 10% reduction in the total calcium content of milk can retard the flocculation of caseinate in concentrated milk, which suggested that soluble calcium was a requirement for caseinate flocculation.

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The possibility of pH change during freezing as a factor in protein destabilization was studied by Tessier and Rose (1956) and Van den Berg (1961). They found no relation between pH change and protein flocculation. Many factors have been investigated in regard to their possible influence on the destabilization of proteins in frozen milk and concentrates. Bell and Mucha (1952) showed that as the solids in milk are increased, the amount of precipitate increased in the thawed products. The effect of homogenization and preheat treatment on protein stability of frozen milk products has also been studied (Trout, 1950). Tumerman et al. (1954) reported that enzymatic hydrolysis of lactose in concentrated milk inhibited protein destabilization. Desai et al. (1961) showed that the loss of soluble lactose through crystallization during freezing and frozen storage of milk products enhanced destabilization. This observation had been made earlier by Wildasin and Doan (1951) who showed that the addition of sugar to milk prior to freezing inhibited protein destabilization. The addition of sugar was theorized to reduce deemulsification due to the smaller ice crystals produced, which form a less rigid frozen surface, and to a freezing point depression. The protective effect of sucrose has also been attributed to an increase in viscosity of the unfrozen protein.

Rutz et al. (1953) investigating emulsion stability of freeze-dried milk and cream showed the addition of sodium

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citrate and dipotassium phosphate to be ineffective in promoting emulsion stability. Several workers have investigated the effect of added emulsifiers and surfactants on the emulsion stability and dispersibility of whole milk. Nelson and Winder (1963) observed that such compounds greatly improved dispersibility but adversely affected the emulsion stability. Mickle (1965, 1966), on the other hand, found that the stability of the milk fat emulsions increased with increased concentration of emulsifier with an optimal HLB between 11 and 14.

Czulak et al. (1961) reported that attempts to dry cheddar cheese under partial vacuum were unsuccessful, primarily because of melting and exudation of fat. They concluded that cheese could be successfully dried if the cheese were mixed with 30% by weight dried skimmilk, dried buttermilk, or dried whey powder.

Freeze-Drying

Stein (1966) referred to freeze-drying as "a procedure uniquely capable of performing certain difficult tasks very well indeed, but not a contender for a leading place among food processing methods." This statement represents only one school of thought, since it has already been demonstrated that reconstituted freeze-dried foods are superior in flavor and quality to the same foods preserved by the more conventional drying methods. The relatively high cost of freeze-drying compared with other forms of

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drying has been the main deterrent to increased production of freeze-dried foods.

Mann (1966, 1967) reviewed freeze-drying of dairy products and concluded that, in view of the costs involved, freeze-drying was likely to be confined to special or luxury dairy products. Improved and more efficient installations have been lessening these costs. Continuous freeze dryers consisting essentially of a constantly evacuated tunnel, with a throughput of up to 30 tons of wet material per day have been developed (Anter and Hoffman, 1968). Milk and other products have been spray frozen and freeze-dried, producing water soluble powders (Mann, 1967). Freeze-dried ice cream has been produced commercially for use as centers for cookies and candy (Anon, 1964).

Freeze-drying has been utilized for many years in the commercial manufacture of high quality bacterial cultures. Recently, the merits of freeze-drying natural cheese have been investigated. Several workers, Evsrateva et al. (1960), Schultz (1966), Meyer and Jokay (1959), working with different cheese varieties found that freeze-drying partially destroyed the natural body properties of most cheeses. Finch (1966) demonstrated the utility of using freeze-dried cheddar cheese in dry mixes for process cheese production.

Desai (1966) studying the effects of different drying methods on some of the physical, chemical, and

organoleptic properties of cultured cream observed the complete loss of the typical body and texture during freeze-drying; however, the retention of volatile flavor compounds was better in the freeze-dried than in the foam-spray dried sour creams examined.

Body, Texture, and Flavor of Cultured Cream

Various factors influence the resulting overall acceptability of a cultured cream. These include raw materials, processing variables, the bacterial culture, and culturing techniques employed. Cultured (sour) cream is a smooth heavy-bodied dairy product which is made by pasteurizing, homogenizing, and ripening light cream (18-20% fat).

Guthrie (1952) determined the effects of various manufacturing procedures in making an acceptable product. From his study, rennet was shown to be an extremely effective stabilizer. Optimum body in the cultured cream was obtained if a pasteurization temperature of 165F (74C) for 30 minutes was employed and when the cream was double homogenized at 2500 psi pressure at a temperature of 165F (74C). This study also showed that the addition of milk solids not fat (MSNF) could increase the desirable consistency of the sour cream. Fresh cream was shown to be preferable to frozen cream in the preparation of cultured cream (Guthrie, 1952).

In further studies of the body of cultured cream, Guthrie (1963) confirmed many of his previous findings and,

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in addition, established that excessive heat treatment produced a weaker bodied cultured cream. The addition of MSNF was found only to improve the body of cream which had a low solids not fat, with excessive additions producing quite undesirable results. Other workers have confirmed Guthrie's observations. Savage et al. (1953) demonstrated that processing cream at 165F (74C) for 30 minutes had the least effect on changes in viscosity due to subsequent homogenization. Doan and Dahle (1928) recommended pasteurization at 180F (82C) for 10-30 minutes and homogenization at pressures above 2000psi. They also recommended the use of MSNF and stabilizers to improve the viscosity of the product. Little (1963) suggested that reduction in microbial load and preparing the cream for efficient homogenization were the principal goals of pasteurization. For chemically acidified creams, Litchfield (1964) suggested 170F (76.6C) to 180F (82.2C) for 20 minutes for the batch pasteurization method and 180F (82.2C) for 18 seconds for high temperature short time. Guthrie (1952) demonstrated that the temperature of the cream at the time of homogenization greatly affected the body of the final product. Aule and Storgards (1958) also reported that viscosity and stability of the cream increased directly with increasing homogenization temperatures.

Roberts et al. (1953) found that cream separated at 42F (5.6C) had a higher viscosity than cream separated at

90F (32.2C) irrespective of whether pasteurization was accomplished before or after separation. Glazier et al. (1954) on the other hand, found cream separated from cold pasteurized milk possessed a lower viscosity than cream separated from raw cold milk.

Guthrie (1963) also found that the body of the resulting cultured cream was, to a large extent, dependent on the quality of the culture. Several review articles have covered the topic of cultures, their propagation, and their requirements (Marth, 1962; Mizuno and Jezeski, 1959, 1961; Greene and Jezeski, 1957a,b). Considerable data have also been published on the factors influencing the production of diacetyl, volatile acids, and associated flavor compounds by lactic cultures. These factors include citrate content (Harmon, 1967), pH (Mather and Babel, 1959), aeration (Pack et al., 1968) and temperature of incubation (Marth, 1962). A suggested pathway for the production of diacetyl, acetic acid, and carbon dioxide from citric acid has been described in an article by Seitz et al. (1963a).

Storage Stability of Dehydrated Dairy Products

Since the nutritional value of a food can only be realized on the consumption of that food, the successful storage of foods implies acceptability at the time of consumption. Hearne (1964) adequately describes the many hazards to which stored foods might be exposed. Other

selected reviews on the deterioration of dried foods are those concerning lipid autoxidation (Lea, 1958, 1962), hydrolysis and reversion of lipids (Patton, 1962), browning reactions (Patton, 1955), change in structure of proteins (King, 1960, 1962, 1965), enzymatic activity and changes in nutritive value (Jensen, 1964). The physical, biochemical, and sensory properties of stored dry milks were reviewed by Hall and Hedrick (1966).

Rheology

The study and application of rheological measurements in food research was well reviewed by Scott Blair (1958). It was pointed out that in a certain sense, all rheological problems are the concern of the manufacturer or processor, since the consumer does little about rheology except to tend to reject the rheologically unsatisfactory.

Rheology is defined as "the science of the deformation and flow of matter" (Scott Blair, 1958). Rheology is therefore mainly concerned with forces, deformations, and time. The passage of time does not always of itself result in changes in materials; chemical changes in foodstuffs, however, often occur with time and they may be studied by rheological methods. Temperature is also important and often appears in rheological equations.

Scott Blair (1958) suggested three reasons why the consumer might be conscious of the rheological properties of foods:

1. normal response to the mechanical behavior of foodstuff;
2. unfounded prejudices; and
3. unrationalized preferences.

Several instruments have been developed to transform the subjective methods of the practical man to the objective methods of the scientist. Scott Blair (1958) classified these instruments into three main groups:

1. imitative tests, which tend to imitate the conditions to which the material will be subjected in practice;
2. indirect empirical tests--for example the penetrometer value, which measures: (a) the rigidity of gels, (b) the force required to penetrate the material, and (c) the consistency of the material as measured by resistance to further penetration;
3. fundamental tests, applied where foodstuffs have complex rheological properties.

Viscosity and Consistency of Dairy Products

Viscosity or consistency is an appearance property of great importance in dairy products. It is related both to the senses of feel and sight. Measurement of this

quality factor may be made not only to indicate the consistency of the finished products, but also as a quality control tool on the raw product or on the product at various stages of the process for predicting final consistency. Such a measurement may also be used as an index of composition, heat treatment, or disaggregation or depolymerization, such as occurs at the initial stages of hydrolysis of protein, starch, or pectin. Viscosity is defined as the friction resulting from the resistance to flow between the liquid layers or the resistance offered by a substance to deformation when subjected to a shearing force (Kramer and Twigg, 1966).

Viscosity, different types of flow, units of measurement, and different measuring systems have been reviewed by Kramer and Twigg (1966).

With particular reference to dairy products, various researchers have found the viscosity of dairy products to be influenced by many factors. Hunziker (1949) described the viscosity of milk and concentrated milk products as they are affected by concentration, composition, and the physical state of the colloidally dispersed constituents. The milk proteins, as influenced by season of the year, process of manufacture, and temperature and time of storage, are particularly involved in the colloidal phenomena which affect viscosity.

Trout (1950) reviewed the effect of homogenization on viscosity of whole milk. Prentice and Chapman (1969) showed homogenization as the process treatment producing the greatest effect on viscosity. Roberts et al. (1953) elucidated the effects of temperature of separation on the viscosity of the resulting cream. The interrelationship of viscosity, fat content, and temperature of cream between 104F (40C) and 176F (80C) was studied by Phipps (1969), the results of which are confirmed by the findings of Rothwell (1966).

The viscosity changes attributable to different heat treatments of milk or cream under varying processing conditions have been studied by several research workers (Douglas et al., 1968; Prentice and Chapman, 1969). Hunziker (1949) reviewed the heat coagulation of milk protein and the control of heat stability of evaporated milk by added chemicals or predetermined forewarming temperatures. For all practical purposes pasteurization does not appreciably affect the viscosity of milk. Davies (1939) stated that milk heated to any temperature below 140F (60C) showed a decrease in viscosity on cooling to 68F (20C), while milk heated to 158F (70C) increased in viscosity on cooling. The viscosity of milk is influenced largely by the colloidal constituents of milk (Jenness and Patton, 1959). These include calcium phospho-caseinate, albumen, globulin, and other whey proteins, dicalcium phosphate, and

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to a lesser extent, by the non-colloidal aspects of clumping by fat globules. The influence of heat on these constituents is reflected in a change of viscosity. An example of such changes would be the increase in viscosity due to colloidal precipitation of lactalbumin by heat (Douglas et al., 1968).

When homogenization accompanies heat treatment of the milk, further changes in viscosity are to be expected, depending upon the temperature and pressure of homogenization and the temperature of pasteurization (Guthrie, 1952, 1963). The effect of pH on the viscosity of cultured dairy products has also been extensively studied. Dairy fermentation processes have been reviewed by Chandan et al. (1969). Several patents have also been issued for the manufacture of sour cream by direct acidification (Edwards, 1969; Loter, 1967).

Sensory Evaluation

Sensory evaluation of food products is universally employed as an analytical tool in flavor research. To most individuals, the measurement of dairy product quality implies evaluation by taste, smell, sight, touch, and in rare cases, sound. In many respects, sensory evaluation is the most logical and useful approach in determining product quality, since this is the method by which the consumer spontaneously accepts, rejects, or classifies the product he eats. It is, however, a complex evaluation requiring

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the use of mouth, nose, eyes, ears, and brain in detecting flavor and evaluating food products. Sensory evaluation of dairy and other food products is limited by its subjective nature. Individual differences, training and experience, systematic biases, sensory interaction, group differences, and social influences represent only some of the factors which affect sensory evaluations (Harper, 1962). In addition, there is the problem of selecting words and terms which correspond to the same sensory impression received by several individuals and to the same chemical and physical situations in the product.

One of the shortcomings of sensory evaluation of dairy products involves the standards for various characteristics such as flavor, body, texture, and color, which tend to be imposed more by the preference of professional graders and judging experts than by preferences of consumers.

It must be remembered, however, that consumers expect to be pleased with the food they select and are displeased if the product does not measure up to their expectations. Adverse impressions created by displeasure have a greater influence on the consumer's reaction than do pleasant impressions. Liska (1965) stated that the consumer's viewpoint of flavor has changed in the past 10 to 20 years and will continue to change with the next, and succeeding generations. Drayton (1959) and Walsh (1959) pointed out that studies of consumer preferences were not

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the same as studies of consumer practices. For example, although a consumer prefers an item, he may choose to purchase a lower-cost competitive item. Cost, however, is only one of many factors which influence the final preference and acceptance of food products. Among these factors are various attributes of the food product such as sensory properties, nutritive value, uniformity of flavor, availability; and the attributes of the consumer, which include race, religion, socio-economics, psychological and psychological motivation, sex, age, and geographical location.

Methodology, experimental design, and statistical treatment of the data must be varied according to the food product being evaluated, the objective of the sensory evaluation test, and the panel size. To this end, the Institute of Food Technologists Committee on Sensory Evaluation (1964) has prepared an excellent reference for sensory evaluation studies. Amerine et al. (1965) also discusses the principles of sensory evaluation.

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METHODOLOGY

Preparation of Sour Cream

Fresh raw whole milk for this study was obtained from the Michigan State University Dairy Department. The milk was heated to 95-100F (37-38C) and separated by means of a Westphalia Model LWA 205 separator. The cream obtained after separation contained approximately 35-40% fat and was standardized to 20% fat with fresh skimmilk. In standardizing, the required amount of added skimmilk was reduced as needed to compensate for the weight of other additives required to complete the formulation. Each standardized batch contained approximately 30% total solids.

Normal commercial practices were followed in the preparation of the cultured creams for freeze-drying with the exception of certain variables to be evaluated. In all trials, unless otherwise noted, the following procedure was employed.

1. The raw cream (20% fat) was heated under constant agitation to 130-135F (54-57C).
2. Additives such as casein, nonfat dry milk, stabilizers and emulsifiers, if used, were added at this point and dispersed by means of a 4 Qt Waring blender. Although the

incorporation of air that resulted from using the Waring blender could be potentially detrimental to the body properties of the resulting product, such incorporated air was driven off during subsequent heating.

3. The cream with additives was held at 130-135F (54-57C) under constant agitation for a minimum of 30 minutes to facilitate hydration and dispersion of the additives.
4. The hydrated cream was pasteurized by heating to 175F (80C) and holding at that temperature for 30 minutes.
5. The pasteurized cream was then double homogenized at 2000 psi at a temperature of 160-165F (71-74C).
6. The processed cream was cooled rapidly with a minimum of agitation in an ice bank can cooler to 72F (22C), inoculated with 2% (w/w) of an excellent frozen culture and incubated at 72-74F (22-23C) to a titratable acidity of one per cent, expressed as lactic acid.
7. Following culturing the cream was aged for 24 hours. In order to maintain an even fill depth (1/2 inch) in each tray, ten pounds of the inoculated liquid cream was weighed into each of 5 trays (18-1/4 x 23-3/4") and cultured in

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the tray. Thermocouples (copper-constantan) were placed at the bottom of each tray before the addition of the sample.

These thermocouples were fitted with connectors so that they could be connected to thermocouples permanently installed in the vacuum chamber of the freeze-dryer. The temperature of the platen and product could thus be monitored throughout the drying cycle. Composite fresh samples of each batch were collected in beakers, covered with aluminum foil and stored at 40F (4.4C) to await initial analysis for fat, total solids, titratable acidity, diacetyl, and body and texture properties.

The trays of cultured cream were rapidly frozen in the freeze dryer by direct contact with the platens which were cooled to -85F (-63C). Freezing was usually accomplished in 90-120 minutes.

Freeze-Drying

The freeze dryer used in this research was a Virtis REPP Model FFD42 WS (Fig. 1) equipped with automatic controls for condenser and platen temperatures, vacuum adjustment, weight system recorder for determination of drying curves, constant recording of absolute pressure and thermocouple connections. The capacity of the freeze dryer is 50 lbs of ice removal per run. The adjustable temperature range for the platen was -100 to 250F (-73 to 117C).

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Eight thermocouple connections for temperature measurements were permanently installed inside the vacuum chamber. The thermocouples were connected to a Honeywell Electronic Multipoint Strip Chart Recorder (Fig. 1). The temperature of the platen and the temperature of the product at the bottom and at the surface were constantly measured. The process of freeze-drying was terminated when the temperature measured at the inside bottom of the tray was between 75-80F (24-26C) or ambient temperature. It was observed that if the product were evenly spread throughout the tray and heat was applied uniformly, the final rapid rise in temperature between 50-75F (10-24C) indicated the complete removal of ice as sublimed water vapor from the frozen cream.

Radanovics (1969) showed that breaking the vacuum in the freeze dryer with an inert gas (nitrogen) significantly reduced the formation of hydroperoxides and improved the flavor of the sour cream. Therefore, after the completion of the freeze-drying cycle, nitrogen (W/P) rather than air was introduced into the vacuum chamber until the inside pressure reached equilibrium with the outside atmosphere. The samples were then maintained in the chamber under a slight nitrogen pressure for a minimum of 30 minutes. During this holding period, the heat on the platens was turned off and the condenser temperature was reduced to maintain the chamber free of water vapor.

Fig. 1.--RePP freeze dryer equipped with temperature and pressure recorders.

The freeze-dried cultured cream was triturated with a Patterson Kelly eight quart liquid solids blender model LB 4598 and stored at 40F (4.4C) or 72F (22C) in plastic bags or screw top jars for analysis.

Preparation of Samples for Rheological Study

The freeze-dried cultured cream was reconstituted to the original total solids of the fresh cultured cream with distilled water at 72F. The powder required to produce 1800 grams of reconstituted product was weighed on a top loading Mettler balance. The volume of water required for reconstitution was mixed with the powder in a Waring blender. In order to achieve constant conditions, since the detrimental effect of excessive mixing on the body of cultured cream was observed early in this research, a routine procedure was devised for mixing the powder and water. The blender motor was switched on and off, once per second, 60 times, with a total mixing time of approximately one minute. The on-off motion was introduced to reduce the incorporation of air. After every ten consecutive mixings the sides of the blender were scraped to ensure complete dispersion of all the powder which sometimes adhered to the wall of the vessel during mixing. The reconstituted cream was portioned equally into each of three 600 ml beakers, covered with aluminum foil and refrigerated (40F, 4.4C) for a minimum of twelve hours before testing.

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The viscosity of the reconstituted cream was determined by two instruments: (1) American Society for Testing and Materials (ASTM) precision penetrometer, using a standard grease cone to determine the penetrometer value. (2) Brabender viscomamylograph (VAG) to determine relative viscosity (Fig. 2).

Analytical Techniques

Determination of Penetrometer Value (PV)

The chilled reconstituted sample which had been held quiescently for at least 12 hours at 40F was tested with a Precision penetrometer. The tip of the cone was aligned to the center of the 600 ml beaker and the cone was allowed to penetrate the surface of the cultured cream for twenty seconds before recording the penetration in tenths of millimeters (Appendix Table 1).

Determination of Relative Viscosity by the Brabender Viscoamylograph (VAG)

The VAG was adapted for the evaluation of the rheological properties of cultured cream by virtue of its reproducibility and its sensitivity to small changes in the properties of the product. The VAG is a fully recording instrument for measuring and recording apparent viscosity at fixed or varying temperatures. The determination is made by means of a suspended sensing element immersed in

Fig. 2.--Brabender Visco amylo Graph for determination of relative viscosity.

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the material under test and connected through a precise measuring spindle to a pen arm controlled by an interchangeable linear calibrated spring cartridge. The VAG is a rotational instrument which permits continuous determination of viscosity. The rotating sample cup contains a number of fixed vertical pins and is driven by a variable speed synchonous motor.

A circular metal disc with metal pins projecting vertically downward into the sample serves as a sensing element. Rotation of the (viscous) sample exerts a force on the sensing element which is dynamically balanced by a calibrated torsion spring. Application of the restoring force is accompanied by an angular deflection of the sensing element shaft which is recorded continuously on a strip chart recorder. The sample cup is positioned in an electrically heated air bath and a cooling coil projects into the cup. Heating and cooling are controlled by a mechanically operated thermoregulator which maintains a constant temperature or which increases or decreases the temperature at a constant rate of 1.5C per minute. The range of measurement of the instrument may be increased by the addition of weights to suppress the zero, or by substitution of a less or more sensitive cartridge. Shear rates can be changed by varying the speed of rotation of the bowl.

Use of this instrument afforded the opportunity of obtaining an initial penetrometer value of the quiescent

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sample as well as more objective appraisal of the resistance of the product to varying degrees of agitation for increasing lengths of time. For the evaluation of the body or viscosity of the reconstituted or fresh culture cream, test conditions were standardized. A cartridge of 700 centimeter gram of torque (cmg) with a preload of 350 cmg was used unless otherwise indicated. The range on the strip chart (0-1000), therefore, represented 0-100% of the measuring range of the cartridge or 0-700 cmg, plus the preload (350 cmg) which changed the zero, increasing the range to 1050 cmg. A reading of 500 Brabender units on the strip chart would be equivalent to 525 cmg. The speed of rotation of the bowl was set at 25 rpm to control the effect of shear rate.

The arbitrary Brabender units were not reported because of the different factors of time and shear rate involved. The different treatments evaluated were compared on the basis of the response or pattern of breakdown that resulted from the prescribed treatment. To analyze a 450 gram sample at an initial temperature of 40F (4.4C) periods of agitation of 5-60 minutes sufficed to obtain a curve which represented the initial and final body of the stirred product. The sample was maintained cold by the circulation of cold water to offset the slow rise that would occur due to frictional heat and prevailing atmospheric conditions. Typical duplicate determinations on two identical samples

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of reconstituted freeze-dried cultured cream (Appendix Fig. 1) show the excellent reproducibility of the test. Penetrometer values were used to affix a numerical value to the body properties of the quiescently stored fresh and reconstituted products.

Fat

The separated cream containing 35-40% fat was standardized to 20% fat on the basis of the official Babcock test. The per cent fat of the cultured cream was determined by means of the official Roese-Gottlieb test (AOAC, 1965), using the Mojonnier apparatus.

Moisture

The per cent moisture of the freeze-dried powder was determined by the Karl Fischer titration, using a Beckman KF-2 aquameter (Beckman, 1965).

The fresh cultured cream was analyzed for total solids by a vacuum oven method, using the Mojonnier apparatus.

Titratable Acidity

Titratable acidity expressed as lactic acid was determined on a 9.0 gm sample by titrating with accurately standardized 0.1N sodium hydroxide to the phenolphthalein endpoint (pH 8.3).

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The pH measurements on the control, the reconstituted freeze-dried and the direct acidified samples were made with a Beckman Zeromatic pH meter using a calomel half cell and glass electrode standardized to read accurately in the range of pH 4.0-7.0. The results were expressed to the nearest one-tenth of a pH unit.

Diacetyl Determination

The colorimetric method of Prill and Hammer (1938) was used to determine the diacetyl content of fresh, freeze-dried and reconstituted cultured cream samples. The diacetyl contents reported for the freeze-dried cultured cream were based on determinations carried out on the powder, due to possible errors introduced by converting acetylmethylcarbinol to diacetyl during reconstitution.

In testing the fresh or the reconstituted cultured cream, 20 g aliquots were accurately weighed into a 500 ml, two necked distillation flask, and 50 ml of distilled water was added. The flask was connected to the distillation apparatus (Fig. 3) and a slow stream of CO_2 was passed over the sample and through the apparatus for at least five minutes. Steam was then admitted under reflux to displace any remaining air and CO_2 from the system. When bubbles of gas ceased to appear in the collection trap, distillation was permitted to proceed at a slow rate for 30 minutes, collecting 10.0 to 10.2 ml distillate in 2 ml hydroxylamine

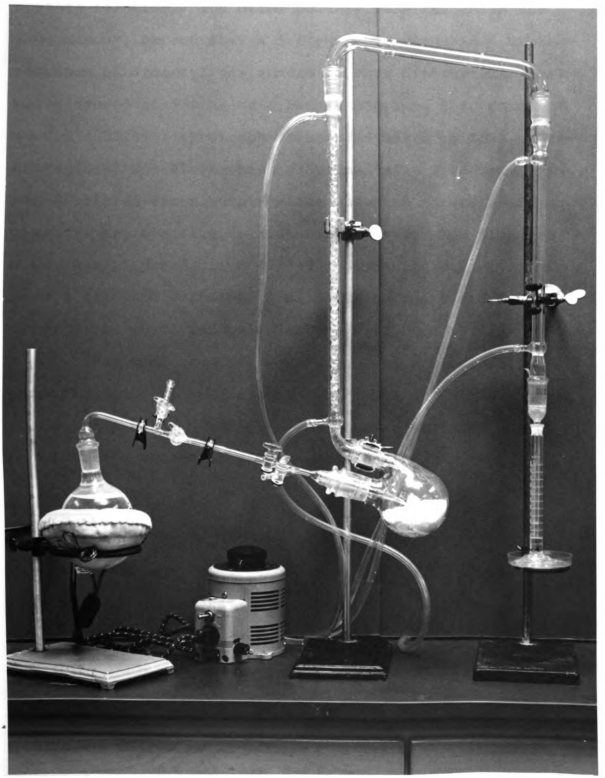

Fig. 3.--Distillation apparatus for diacetyl determinations.

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acetate solution. The dried powders were tested in the same manner, except that a 6.0 g sample was used. The absorbance of diacetyl (as ammono ferrous dimethylglyoxime) was measured at 530 mμ on a Bausch and Lomb Spectronic 20 (Model #3322995) spectrophotometer adjusted to 100% transmittance with a blank which contained all of the reagents and distilled water. This value was then converted to mg diacetyl by referring to a standard curve. The standard curve was prepared using 0.1349 g dimethylglyoxime to prepare a standard solution, 1 ml of which contained 0.1349 g of dioxime, which is equivalent to 0.1000 mg of diacetyl.

Per cent recovery was determined periodically on a prepared diacetyl solution and with each set of determinations the color development of a known standard was used to ascertain the quality of the reagents.

Free Fat

The method of Thomas et al. (1957) was used to determine the mgm free fat/gm total fat in the freeze-dried cultured cream powders.

Dispersibility

The method outlined by Stone et al. (1954) was modified, in view of the high fat content and added stabilizers, to determine the dispersibility of the freeze-dried cultured cream. A 10 g sample of each powder was blended with 90 ml distilled water at 25C for 30 seconds in a micro

high speed blender. Immediately after mixing, the dispersion was filtered under high vacuum using a coarse porosity sintered glass funnel. The resulting filtrate was transferred to a 100 ml volumetric flask and made up to the mark with distilled water. The solids content of 10 g aliquots of this filtrate was determined by the Mojonnier method and the results reported as per cent powder dispersed/100 g sample.

Sensory Evaluation

Sensory evaluations of freeze-dried reconstituted cultured cream were carried out concommitantly with physical and chemical analyses, both initially and following storage, to evaluate stability under different conditions. The samples were prepared as previously described and evaluated by a selected panel of 4-6 trained judges or were placed in small cups in a random pattern for evaluation by panels selected at random from the personnel of the Food Science Department at Michigan State University.

The selected trained panel used a hedonic preference scale with a range of 1-13 in evaluating the samples. The ranking method and a combination of ranking and the triangular test was the basis of evaluation by the consumer panel.

RESULTS AND DISCUSSION

The delicate flavor, body, and texture of cultured cream, which are readily altered or degraded during dehydration, have complicated the preparation of a completely acceptable dehydrated product. Efforts to reduce weight, improve flavor, and enhance storage stability at room temperature have renewed interest in freeze-drying as a method of preservation. Desai (1966) showed freeze-drying to be superior to spray drying as a method of dehydrating cultured cream because of the greater retention of volatile flavor components and lessened heat damage. Radanovics (1969) also showed that the flavor components of cultured cream were not entirely removed during freeze-drying and that the use of antioxidants and inert gas packing would greatly improve the storage stability of the freeze-dried powder. An acceptable product from the viewpoint of the consumer has not been produced because many of the desirable rheological properties of the fresh cultured cream are destroyed during the process of freeze-drying.

The Effects of Processing Variables on the Body Characteristics of Freeze-Dried Reconstituted Cultured Cream

Processing variables have been shown to play an important role in the final quality of the dehydrated food (Bradley and Stine, 1964; Goldblith et al., 1963). Since processing variables are often interrelated, the systematic evaluation of one variable such as heat treatment, homogenization pressure or added ingredient, requires that all other processing parameters be carefully standardized and controlled. Trials were therefore repeated and parameters were individually varied to eliminate or compensate for possible interactions. This permitted conclusions to be drawn with regard to each parameter tested without the aid of statistical analysis.

The Effect of Pasteurization Treatments on the Final Body and Texture of Freeze-Dried Cultured Cream

Early research on the effect of pasteurization treatment on properties of fresh cultured cream (Guthrie, 1952, 1963) had shown that heating the cream to 165F (74C) for 30 minutes was optimal for building a good body in fresh cultured cream. Heat treatments of 145F (62C) or 180F (82C) for 30 minutes resulted in weak and grainy products.

Several batches of cultured cream were prepared to produce powder which exhibited different body and texture properties on reconstitution and to study the effect of pasteurization. In these trials one large batch was

processed to the point of pasteurization. The cream was homogenized at 165F (74C) prior to pasteurization and was then divided into three batches. Each batch was pasteurized at one of the following temperatures: 165F (74C), 175F (80C), or 185F (85C) for 30 minutes. The temperatures and times of pasteurization were carefully monitored.

Table 1 contains results representative of these trials in which the three heat treatments were evaluated. A pasteurization temperature of 175F (80C) for 30 minutes was selected as the most effective means of improving the body and texture characteristics of the cultured cream without adversely affecting the flavor. The acceptability of the intermediate treatment over the extremes was based on different factors. Exposure to the higher temperature 185F (85C) for 30 minutes proved to be too severe a heat treatment. An undesirable cooked flavor developed, the body was weak, and the texture tended to be grainy. The results in Fig. 4 corroborate the data presented in Table 1 and demonstrate the sensitivity of the VAG to seemingly small differences in the properties of the product. On this curve, time is represented in minutes on the abscissa and arbitrary Brabender viscosity units, ranging from 0-1000, are represented on the ordinate. The VAG reflects the breakdown of the product with agitation by the decline in Brabender viscosity units with time. The rate of breakdown decreases with time of agitation and the slope of the

Table 1.--The effect of different pasteurization temperatures on some of the proper

Table 1.--The effect of different pasteurization temperatures on some of the properties of freeze-dried cultured cream.

Heat Treatment (°F/20 mins)	Trial	Penetrometer Values (mm/10)		Free Fat	Titratable Acidity (%)	Sensory Evaluation		
		Quiescent	Stirred	mg/g Total Fat		Flavor	Body (40F)	Texture
165	1	290	395	976.0	0.98	Good	Good	Smooth
	2	406	508	997.0	0.90	Good	Sl weak	Smooth
	3	392	482	994.8	0.88	Good	Sl weak	Smooth
175	1	275	372	963.2	0.98	Good	Good	Smooth
	2	364	423	979.5	0.90	Good	Good	Smooth
	3	385	455	976.8	0.88	Good	V sl weak	Smooth
185	1	300	448	965.6	0.98	Cooked	Weak	Grainy
	2	383	530	969.3	0.90	Cooked	Weak	Grainy
	3	410	549	972.8	0.88	Cooked	Weak	Grainy

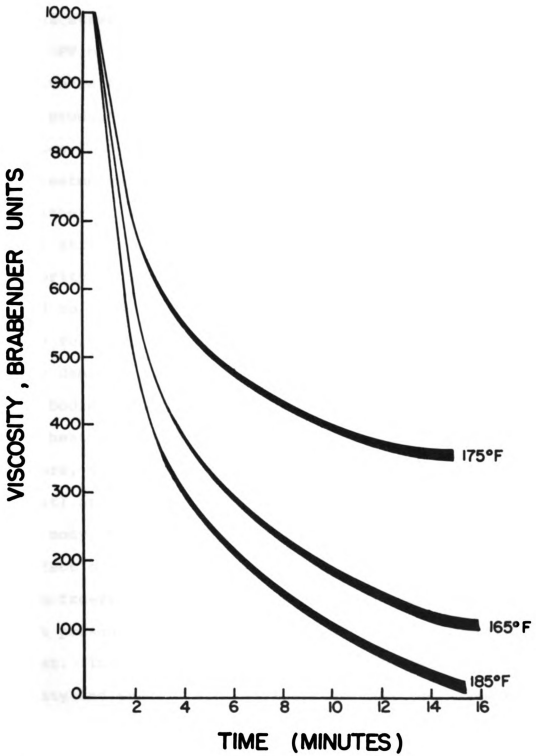


Fig. 4.--The effect of pasteurization temperatures on the viscosity of reconstituted freeze-dried cultured cream.

curve, therefore, provides an index of resistance to the physical stress. A comparison of the VAG curves (Fig. 4) and the PV of the stirred product (Table 1) showed that excessive heat treatment (185F, 85C) resulted in a weak bodied product which broke down rapidly on agitation. The PV values (Table 1) on the products prepared by the lower heat treatments suggested that the body properties obtained with either treatment were similar and that the stirred product still retained much of its original body. The superiority of the body of the reconstituted product heat treated to 175F (80C) for 30 minutes following quiescent storage for a minimum of 12 hours and after mixing was clearly demonstrated by the VAG curves (Fig. 4). The weaker bodies of the cream heated to either the lower and higher heat treatment were also evident. The results, therefore, showed that pasteurization of processed cream (20% fat) at 175F for 30 minutes produced a product with better body, texture and flavor. Table 1 also shows that the effect of heat treatment on free fat was minimal and that on freeze-drying a cream of medium fat content (20%), the fat present in the powder existed almost exclusively as free fat. The effect of different heat treatments on the viscosity and stability of milk systems has been studied by many researchers. Douglas et al. (1968) showed that heating skimmilk concentrates to temperatures above 185F (85C) decreased the whey protein nitrogen. This change was

accompanied by a marked increase in viscosity. Heating to 158F (70C) for 25 seconds did not denature the whey protein nitrogen or affect the viscosity. Prentice and Chapman (1969) found that heat treatment was second only to homogenization in giving rise to differences in viscosity of the creams tested. Creams which were inadequately heat treated by heating to 151F (66C) were almost twice as viscous as those heated to 165F (74C). Guthrie (1963) also noted this increase and decrease in viscosity of fresh cultured cream with different temperatures of heat treatment and showed that the history of preheat treatment governed the selection of a pasteurization temperature consistent with good body characteristics.

Sommer (1952) showed that exposure of milk to temperatures of 165-171F (74-77C) for 30 minutes results in:

1. increased hydration of the protein fraction;
2. development of cooked flavor;
3. decrease in the oxidation-reduction potential of the milk.

Hall and Hedrick (1966) reviewed the effect of heat treatment on curd firmness and noted that the curd softening action of relatively high heat treatments above 180F (85C) was due principally to changes in solubility brought about among the calcium and phosphate salts in milk.

The heat treatment of a medium fat cream (20%) fortified with MSNF, milk proteins, and stabilizers was

selected to provide the optimal characteristics in the resulting product. A pasteurization treatment of 175F (80C) for 30 minutes was required for adequate pasteurization of the high total solids viscous cream. Pasteurization reduced the competition from foreign organisms during culturing. This treatment also increased hydration of the proteins and decreased the oxidation reduction potential of the milk without the development of excessive cooked flavor. Hence maximum viscosity and improved body properties were obtained in the freeze dried reconstituted cultured cream.

The Effect of Homogenization Pressure on the Body and Texture of Reconstituted, Freeze-Dried Cultured Cream

The cream used in these trials was pasteurized as a single batch prior to portioning into smaller volumes for homogenization. This eliminated the possibility of processing variables which might be induced by individually heating and mixing smaller volumes of cream for each homogenization. In these experiments the hot cream was pumped through the first stage homogenizer valve only (single pass, single stage) or was circulated twice through this valve (double pass, single stage). The results of these trials (Table 2, Fig. 5) show that the body and texture of the reconstituted sour cream were greatly affected by either single or double pass homogenization. Sensory evaluation of the fresh cultured cream corroborated Guthrie's findings (1952, 1963)

Table 2.--The effect of homogenization treatment and pressures on some of the properties of freeze-dried cultured cream.

Trial	Homogenizing Pressure (psi)	Penetrometer Values (mm/10)		Free Fat	Sensory Evaluation	
		Quiescent	Stirred	mg/g Total Fat	Body (40F)	Texture
Trial 1 Single Pass Single Stage	1000	269	375	999.3	Good	Smooth
	1500	242	345	993.8	Good	Smooth
	2000	262	348	984.1	Good	Smooth
	2000*	260	359	970.6	Good	V Smooth
	2500	262	356	982.0	Good	Sl Grainy
Trial 2 Double Pass Single Stage	1000	285	384	999.0	Good	
	2000	320	418	967.8	Good	V Smooth
	3000	328	458	911.2	Sl Weak	Grainy
	4000	333	BTM	914.5	Weak	Grainy

*Double homogenized.

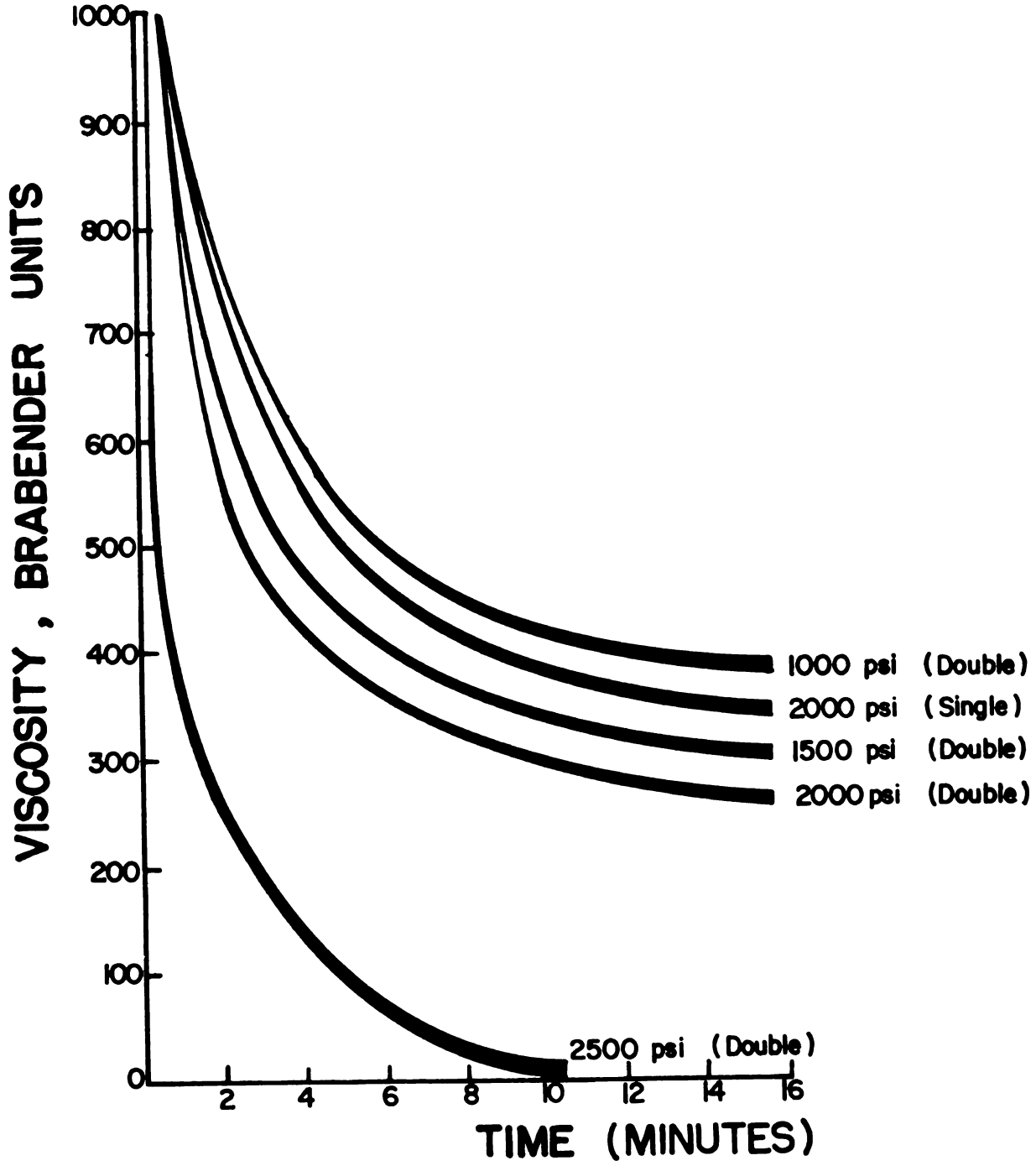


Fig. 5.--The effect of increasing homogenization pressure on viscosity of reconstituted freeze-dried cultured cream.

that pressures in the range of 3000 to 4000 psi produced a firm bodied product. After freezing and dehydration however, the creams which had undergone high homogenization pressures tended to be noticeably weaker than other samples in the series. The undesirable effect produced at 4000 psi was evidenced by the complete penetration of the stirred product by the penetrometer cone. The VAG curves (Fig. 5) of reconstituted cultured creams processed at different homogenization pressures further elucidate the effect of these pressures. These results provide an excellent example of the difference in sensitivity of the two instruments used for evaluating the body properties of the cultured cream. The PV results shown in Table 2 suggest that there is comparatively little difference between the reconstituted cultured creams homogenized between 1000-2500 psi. The weaker body of the reconstituted cream treated at 2500 psi was clearly evident in the VAG curves shown in Fig. 5.

Sensory evaluation of the various reconstituted products suggested that homogenization at medium pressures (1500 to 2000 psi) produced the most desirable physical characteristics. Lower pressures (1000 psi) produced a firmer body that was more resistant to agitation, whereas somewhat higher pressures (2000 psi) produced a cream with smoother texture. Homogenization at higher pressures appeared to improve the dispersibility of the freeze dried powders. This was at first assumed to be the result of

less free fat in such powders. The results (Table 2) however, showed that even pressures of 4000 psi were ineffective in reducing the free fat content of the freeze dried sample. Nickerson et al. (1952) and Rutz (1953) had previously observed destabilization of the milk fat emulsion during lyophilization. Homogenization of whole milk, concentrated milk and cream, with fat contents of up to 20%, has been shown to minimize the damaging effects of freezing such products (Webb and Hall, 1935; Trout, 1950). Homogenization pressures of 3000 psi have been recommended by these researchers. Evidence obtained from these studies shows that pressures in excess of 2000 psi destroyed the body properties of the reconstituted freeze dried product without reducing the level of free fat. These observations suggest that freezing and dehydration both function to destroy the milkfat emulsion, which adversely affects the body and texture properties of the reconstituted cultured cream.

The Effect of Freezing Rate and Fat Content on the Physical Properties of Freeze-Dried Cultured Cream

Earlier research in this laboratory (Stine et al., 1967) indicated that the freezing rate had no effect on the body of the reconstituted freeze dried cultured cream. The freezing conditions employed (Stine et al., 1967) ranged from slow freezing at temperatures of 10F (-12C) to rapid freezing at -110F (-79C). Similar trials were repeated in this research since the total solids and fat content of the

cultured cream used in this study were higher. Difficulty encountered with the freezing of the cultured cream at the slow freezing temperatures (10F, 12C) required that the temperature for slow freezing be lowered to 0F (-18C). The trials were designed to determine whether increasing the freezing rate would reduce the high level of free fat found in freeze-dried cultured cream. In addition, information was sought regarding the possible effects of smaller ice crystals formed during rapid freezing on the gel structure of the fresh cultured cream. Rapid freezing reduces the concentrating effect of slow freezing and the smaller ice crystals produced during rapid freezing may exert less pressure, due to expansion, on the system. This phenomenon could result in less mechanical damage to the fat globule membrane and have some bearing on the free fat in the freeze-dried powders.

A large batch of cultured cream was prepared for these trials. The batch was divided into 10 lb portions and frozen. In rapid freezing the product was frozen by direct contact with a refrigerated platen which was maintained at -85F (-65C). The slow freezing condition was achieved by placing the tray of cultured cream in a static air cold box (0F, -18C) with no air movement or direct contact with any surface containing refrigerant.

The results of these trials, shown in Table 3, clearly indicate that the freezing rate had no effect on

Table 3.--Effect of freezing rate and increasing fat content on some of the properties of the resulting freeze-dried reconstituted cultured cream.

Freezing Rate	Per Cent Total Fat	Free Fat	Penetrometer Values (mm/10)		Sensory Evaluation	
		mg/g Total Fat	Quiescent	Stirred	Body (40F)	Texture
Rapid	15	950.0	390	BTM	Sl Weak	
	20	969.0	289	400	Good	Creamy
	25	999.0	246	372	Good	V Smooth
Slow	15	978.0	390	BTM	Sl Weak	
	20	983.0	305	397	Good	Creamy
	25	995.0	258	380	Good	V Smooth

either the incidence of free fat in the freeze-dried powders or the viscosity of the reconstituted cultured cream. Under both conditions of freezing more than 95% of the total fat in the powder existed as free fat. The penetrometer values also do not indicate any significant difference between the effects of the rates of freezing.

Webb and Hall (1935) reported that the content of free fat increased directly with the fat content of frozen cream which had been thawed. Increases in the fat content of fresh cream did not result in an increase in free fat in the dried cultured cream powders. A 66.6% increase in fat content (15-25%) resulted in only a 2-4% increase in free fat (Table 3). The total destruction of the milk fat emulsion during freeze drying indicated that even with an increase in fat content of the cream no further increase in free fat could be expected.

Since the amount of free fat in any freeze-dried dairy product is very high, small changes are unlikely to have much effect on the physical properties and stability of the dried product. Only a major reduction in the amounts of free fat could conceivably produce desirable changes in dispersibility characteristics and the storage stability of the freeze-dried cultured cream powders.

An increase in the fat content of the cream resulted in a corresponding increase in viscosity of the reconstituted product (Fig. 6, Table 3). This relationship was not

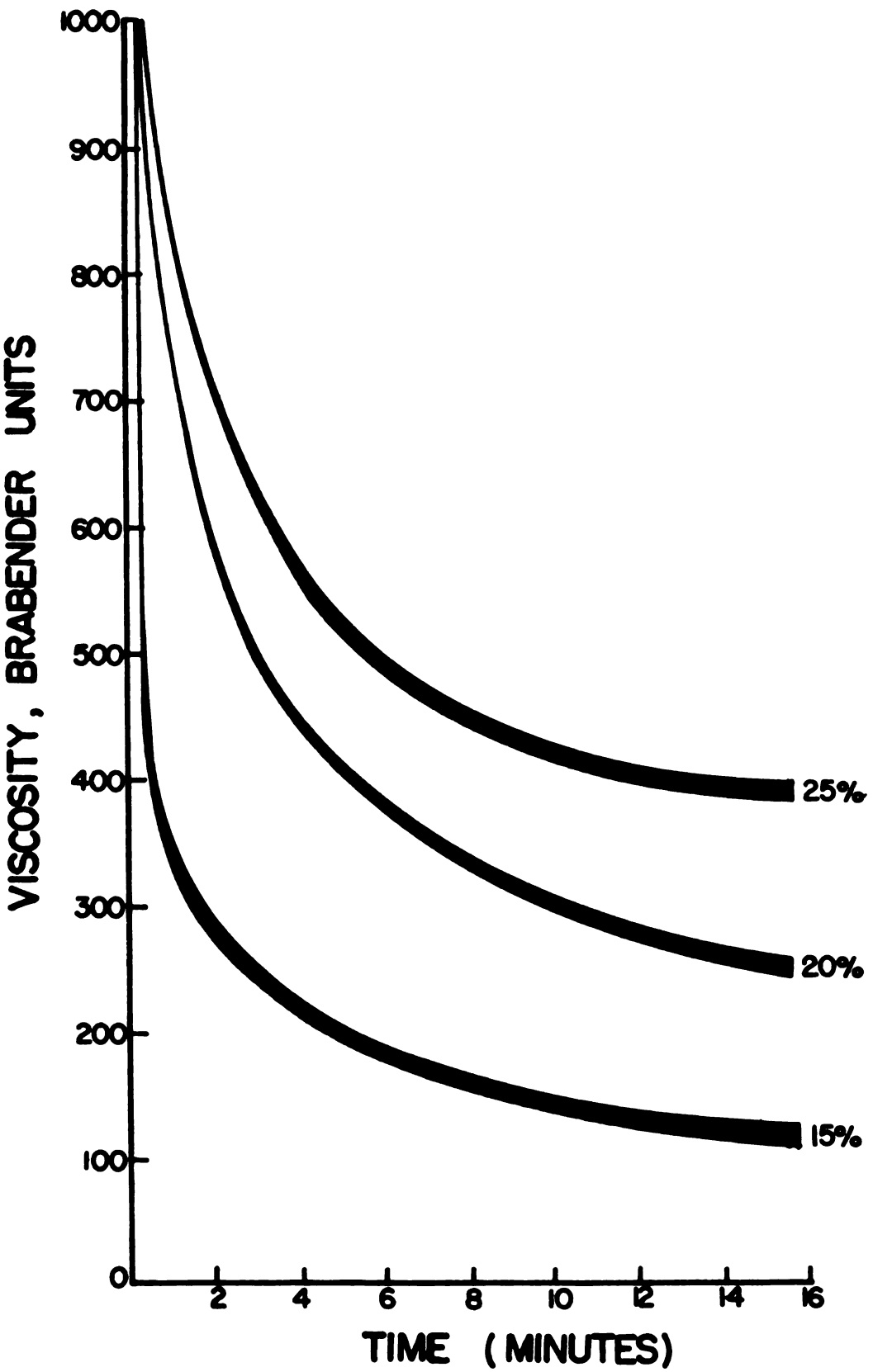


Fig. 6.--The effect of increasing the fat content of the fresh cream on the viscosity of the reconstituted freeze-dried cultured cream.

linear and a greater increase in viscosity was observed from 15-20% (Table 3). Rothwell (1966) and Prentice and Chapman (1969) have previously observed this effect in their work with fresh high fat creams. The definite interrelationship which was observed between fat content, heat treatment and viscosity permitted the construction of nomograms (Phipps, 1969). The results of the experiments described here suggest that a higher fat content will produce a firmer bodied reconstituted product. However a 20% fat cream was chosen for subsequent trials because at this fat content the maximum increase in viscosity was obtained, and higher levels of fat resulted in poorer dispersibility.

Faustoba and Vlodavets (1955) defined the destabilizing effect of freezing and dehydration which resulted in fat clumping and protein flocculation as "a reduction of the fat dispersion due to fusing of the fat particles." The expansion of water on freezing and the possible denaturation of the membrane proteins ruptures the fat globule membrane. This results in the liberation and fusion of globular fat causing a subsequent loss in viscosity (Knoop and Wortmann, 1959). Rapid freezing could minimize this effect if the rate of contraction of the fat was the same as the rate of expansion of the water. Slow freezing would inflict greater damage on the structure of the cream because of the pressures exerted within the system by the larger ice crystals.

Lagoni and Peters (1961) showed destabilization to be largely dependent on the rate and magnitude of the temperature change and to be favored by low temperature storage of high fat products.

Rapid freezing has been shown to increase the drying rates (Mackenzie and Luyet, 1963; Quast and Karel, 1968). The increase in rate of drying was attributed to the smaller ice crystals formed, permitting the formation of "micro channels" for rapid diffusion of the water vapor through the dry material.

In this study, no appreciable difference was noted between the free fat contents of the dry powders prepared from cream containing different fat levels and frozen at different rates. This may be due to the very high fat content of the powder (64-66%) and the lessened heat damage encountered during freeze drying. There was a definite increase in viscosity as the fat content was increased from 10-25%.

The variation in fat content did not result in any significant difference in the incidence of free fat. Rapid freezing of a 20% fat cream was none-the-less adapted for subsequent trials because the theories which postulated the advantages of rapid over slow freezing strongly suggested its superiority. In addition, the results of these trials did suggest slight trends in favor of rapid freezing although no significant differences were observed.

The Effect of Added MSNF on the Physical Properties of Freeze-Dried Cultured Cream

Since increasing the fat content improved the body properties of the reconstituted cultured cream, investigations were directed towards an evaluation of the basic composition of the cream on the properties of the reconstituted product. Guthrie (1963) pointed out that increasing the milk solids-not-fat (MSNF) of a low solids cream increased the viscosity of the fresh cultured cream. Mickle (1966) showed that a more significant reduction in free fat was obtained with added MSNF than by the use of emulsifiers. Finally, an increase in the total solids of the cultured cream would reduce the amount of water to be removed, thereby increasing the efficiency of the drying operation. Therefore in an effort to reduce free fat and increase the viscosity of the freeze-dried reconstituted product, the effect of varying the MSNF content of the fresh cream prior to culturing was investigated. The level of MSNF was varied from 0-6% (w/w) in repeated trials. The standardized cream with added stabilizers was divided into batches and the different levels of MSNF were incorporated prior to hydration of all the ingredients. The batches of cream were then pasteurized and homogenized. Initially the control, with no added MSNF was compared with 4 different levels of added MSNF in each trial. Unequal acid development between the control, with no added MSNF, and the samples containing other levels of addition later required that a 2% addition

be adopted as the control. The results shown (Table 4, Fig. 7) are representative of these trials and clearly show the undesirable effect of the higher levels of MSNF addition. The optimum change in viscosity and body properties under the conditions studied was observed when the added MSNF content was increased to 2% (Fig. 7). The higher levels of addition (4-6%) resulted in a weaker body, grainy texture and undesirable salty flavor. Another interesting observation was the increase in viscosity that occurred during the viscosity determination in the Brabender visco-amylograph (VAG). After the initial breakdown in body, which occurred within ten minutes of agitation, there was a secondary increase in viscosity during continued agitation. This increase in viscosity was found to be due to the positional arrangement of the vertical pins in the rotating sample cup and the sensing element. Since all the pins are

Table 4.--The effect of addition of MSNF of the body of the reconstituted freeze-dried cultured cream.

Amount of MSNF (%)	Penetrometer Values (mm/10) Quiescent	Stirred	Body and Texture at 40°F	
2.0*	217	350	Good	Smooth
2.0	224	357	Good	Smooth
4.0	220	367	Good	Sl Grainy
6.0	337	365	Weak	Grainy

*2.0% starch added to basic formula.

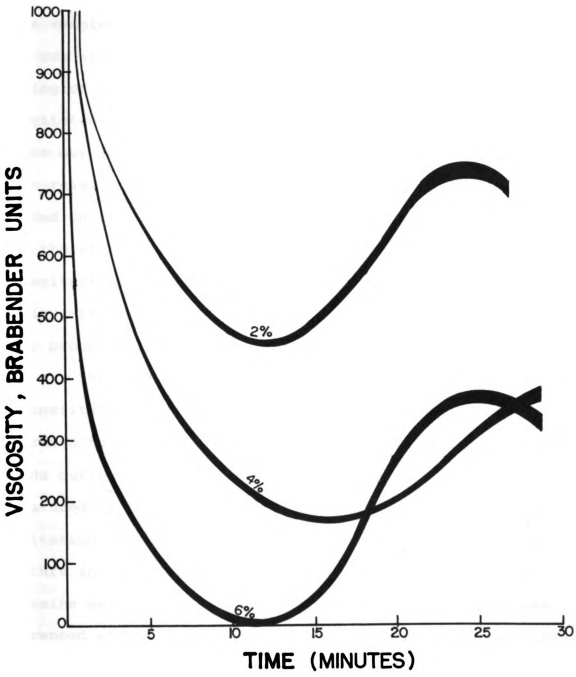


Fig. 7.--The effect of added MSNF on the viscosity of reconstituted freeze-dried cultured cream.

located towards the outer edge, proper mixing of the sample within the sample cup is not achieved when testing a viscous sample. The sample located in the outer portion of the cup will breakdown initially with only a minimum of mixing of the remainder of the sample. Eventually, that unagitated portion of the sample in the center of the cup moves out and mixes with the less viscous cultured cream. This resulted in the increase in viscosity which was recorded on the strip chart. Interpretation of the results was therefore based on the initial breakdown of the sample on agitation. Since the increase in viscosity was due to faulty mixing, only a viscous cultured cream with desirable body properties produced this effect.

The lower viscosity and grainy texture observed in reconstituted creams with the higher levels of MSNF were presumed to be due to incomplete hydration of the milk proteins during processing. The dehydrated proteins seem to be denatured by the acid produced during fermentation. Precipitation of these denatured proteins produced a grainy texture in the cultured cream. At the 2% level all the proteins were properly hydrated and the aqueous phase was entrapped within the casein network to produce a smooth gel. Hayes et al. (1968) studied the viscosity of caseinates and observed that at the higher calcium levels the preparations were of very low viscosity. These high calcium preparations settled out on standing. This would suggest the possibility

of a salt imbalance also, increasing the calcium percentage increased the gelation temperature. Destabilization might result from the increased MSNF which could not be contained within the gel structure and precipitated out. Repeated trials showed this phenomenon of precipitation and a grainy textured product at the higher levels of MSNF. Acid production was enhanced by the addition of MSNF, which at the 2% level imparted no undesirable flavors to the reconstituted cultured cream. The results (Table 4) also show that cold dispersible starch added at the rate of 2% (w/w) produced no significant improvement in the body of the reconstituted cultured cream.

The Effect of Stabilizers and Other Bodying Agents on the Physical Properties of the Reconstituted Freeze-Dried Cultured Cream

The use of stabilizers in the manufacture of fresh cultured cream has increased significantly over recent years in an effort to improve the uniformity of the fresh product. Guthrie (1963) showed that in general, stabilizers increased the plasticity, rigidity and viscosity of the cultured creams. Additions of stabilizers to cream before frozen storage considerably improved physical stability during storage and subsequent thawing (Bell, 1947). Several patents recently issued include stabilizers as part of the basic formula of both direct acidified and cultured creams (Little, 1968; Noznick, 1967; Edwards, 1969; Loter, 1967). The use of stabilizers and other bodying agents have been

shown to be effective in improving the viscosity of direct acidified sour creams (Desai and Harper, 1968).

In this research stabilizers were required which could withstand the effects of heat treatment, homogenization, low pH, freezing and dehydration. Commercial stabilizer suppliers were therefore contacted for samples of stabilizers which would operate efficiently under some, if not all of these conditions. Different levels of stabilizers were mixed into the standardized cream prior to hydration, as described with MSNF trials. Initial investigations were designed to determine the level of addition which produced the maximum viscosity in the freeze-dried reconstituted product. The viscosity of the reconstituted product was found to increase with increasing levels of stabilizer. An optimal level of addition of 0.5% was adapted, because the lower levels of addition did not consistently produce an acceptable product. At levels of above 0.5% addition, although there was an increase in viscosity, the reconstituted product was described as firm and dough-like. The dilution effect of these additives on the flavor of the freeze-dried reconstituted product and the difficulty encountered in the processing of the high viscosity creams prior to freeze-drying combined to make the higher levels of addition undesirable. All the stabilizers tested in this study increased the final viscosity of the reconstituted product to some extent. Some were more effective than

others. The changes observed, however, clearly indicated that added stabilizers were required for the manufacture of an acceptable freeze-dried reconstituted cultured cream.

During the initial investigations, repeated trials showed large variations in the properties of the reconstituted freeze-dried cultured cream made from the same formulations. The body ranged from a heavy, sticky, doughy product which could withstand the effects of agitation to an almost fluid cream with a grainy texture. These findings indicated that small variations in processing such as improper incorporation or hydration of additives were resulting in much larger variations in the final product. A study of proper incorporation of additives, methods of mixing and allowing for proper hydration of the additives before pasteurization and homogenization showed that these variations could be overcome. Proper incorporation of the additives required proper dispersion by means of a high input of energy, followed by constant agitation during hydration. In this study, based on the recommendation of the supplier, the ingredient was dispersed in either hot (135F, 57C) or cold (60F, 15.6C) skimmilk by means of a 4 Qt Waring blender. The properly dispersed suspension was then mixed with the entire batch at a temperature of 135F (57C) and hydrated with constant agitation for 30 minutes. This allowed for complete hydration of each particle dispersed throughout the cream.

The results (Table 5) show the effect of some of the stabilizers used in this study, added at the rate of 0.5% (w/w), which gave the most favorable results. The importance of added stabilizer (0.5%) is clearly shown (Table 5) by comparing the results of the test with the control containing no added stabilizer. The control was completely fluid on reconstitution even after quiescent storage for 12 hours at 40F (4.4C). These results further show two stabilizers to be superior to the others tested, namely glucosyl glucan (0.5%) and the locust bean gum (0.4%) and guar (0.1%) combination. The use of guar at the recommended 0.08-0.1% rate of addition in combination with other stabilizers to obtain the required 0.5% level of addition greatly increased the viscosity of the reconstituted cultured cream. Addition of guar at levels greater than 0.1% however, resulted in a weaker bodied product.

The penetrometer values on the quiescent samples (Table 5) range from 270-348, whereas the stirred samples showed slightly more variation, from 350-450 mm/10. The VAG (Fig. 8) shows curves representative of stabilizers used in these trials. The preload associated with each curve shows the variations that were required in the test conditions in order to obtain all four curves within the range of the strip chart. The most desirable body with the greatest resistance to agitation was obtained when glucosyl glucan was used as a stabilizer under these conditions.

Table 5.--The effect of added stabilizers on the body of the reconstituted freeze-dried cultured cream.

Type of Stabilizer	Amount of Stabilizer (%)	Penetrometer Values (mm/10)		Sensory Evaluation	
		Quiescent	Stirred	Body and Texture (40°F)	
Control--No Added Stabilizer	--	BTM	BTM	Fluid	Grainy
Glucosyl Glucan	0.50	273	350	Good	Smooth
Glucan and Methyl Cellulose	0.25 0.25	328	439	Sl Weak	Smooth
Gelatin and Locust Bean Gum	0.30 0.20	286	402	Good	Smooth
Guar and Locust Bean Gum	0.10 0.40	270	400	Sl Doughy	Sl Sticky
Sta-Rite*	0.50	318	400	V Sl Weak	Smooth
Olympic*	0.50	327	418	Sl Weak	
Hydroxy Methyl Cellulose	0.50	348	450	Weak	Sl Grainy
Sodium Alginate	0.50	336	422	Sl Weak	

*Commercial Stabilizers--Germantown Manufacturing Co., Broomall, Penn.

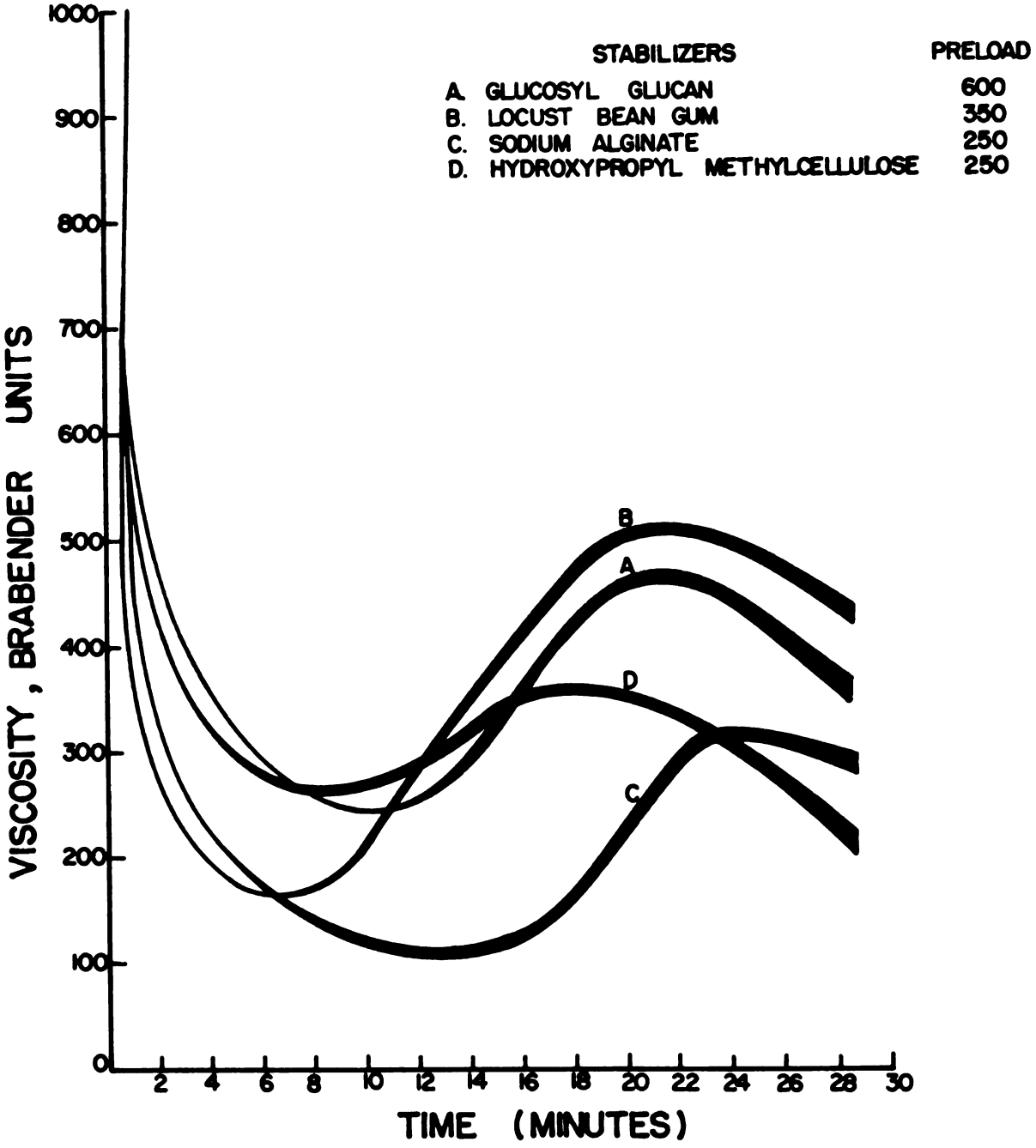


Fig. 8.--The effect of different added stabilizers and combinations of stabilizers on the viscosity of the reconstituted freeze-dried cultured cream.

Reconstituted freeze-dried cultured creams which exhibited desirable body and texture properties could be prepared with any of the following stabilizers: (Listed in the order of preference) glucosyl glucan (0.5%), locust bean gum (0.4%) + guar (0.1%) and locust bean gum (0.3%) + gelatin (0.2%).

Even addition of MSNF (2%) and stabilizer (0.5%) in conjunction with improved processing techniques of pasteurization and homogenization did not produce a completely acceptable body in the reconstituted freeze-dried product. However the further addition of sodium or calcium caseinate to the formulation yielded a desirable reconstituted freeze-dried cultured cream. Added starch with or without added caseinate was also evaluated in a manner similar to that described previously to determine its effect on the viscosity of the reconstituted product. Although added cold dispersible starch imparted definite body properties to the cultured cream, the coincidental development of a starchy flavor limited the maximum acceptable level of addition to 2%, which failed to impart the desirable physical properties to the product. Comparison of the results shown in Tables 6 and 7 and Figs. 9 and 10 indicate that 2% added starch produced no significant effect on the viscosity of the resulting product. The PV results (Tables 6 and 7) show that a weaker body was obtained with added starch. For proper comparison of the VAG results the difference in

Table 6.--The effect of varying the levels of sodium caseinate (with a fixed amount of added starch) on the body of the reconstituted freeze-dried cultured cream.

Amount of Sodium Caseinate (%)	Penetrometer Values (mm/10)		Sensory Evaluation	
	Quiescent	Stirred		
0	320	550	Weak	Smooth
0.5	310	435	Weak	Smooth
1.0	294	386	Good	Smooth
2.0	317	513	Weak	Grainy

Table 7.--The effect of varying the levels of sodium caseinate (in the absence of added starch) on the body of the reconstituted freeze-dried cultured cream.

Amount of Sodium Caseinate (%)	Penetrometer Values (mm/10)		Sensory Evaluation	
	Quiescent	Stirred		
0	320	560	Weak	
0.5	256	403	Good	Smooth
1.0	252	392	Good	Smooth
1.5	286	530	Sl Weak	Sl Grainy

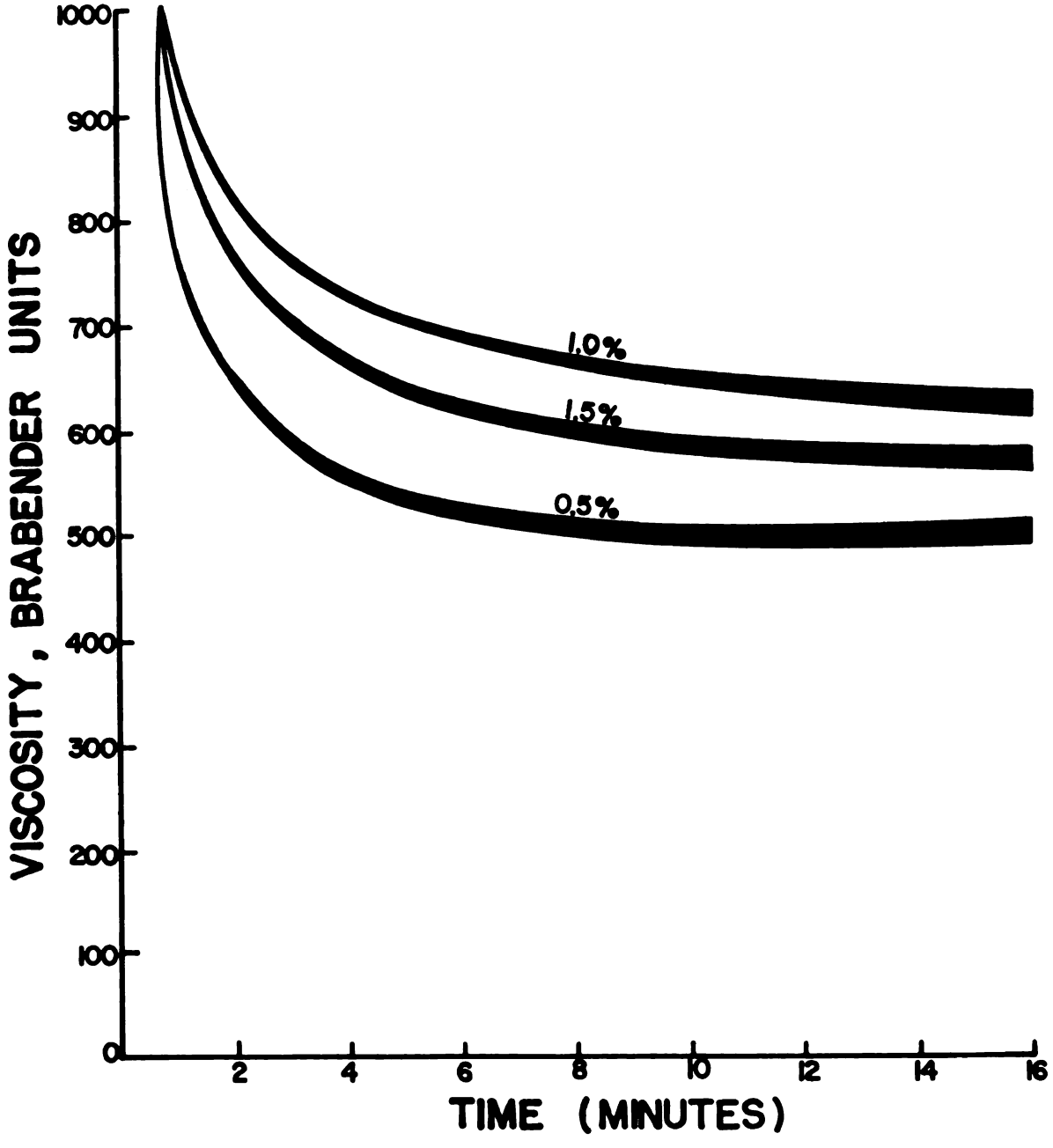


Fig. 9.--The effect of added sodium caseinate in conjunction with a fixed amount of starch (2%) on the viscosity of reconstituted freeze-dried cultured cream.

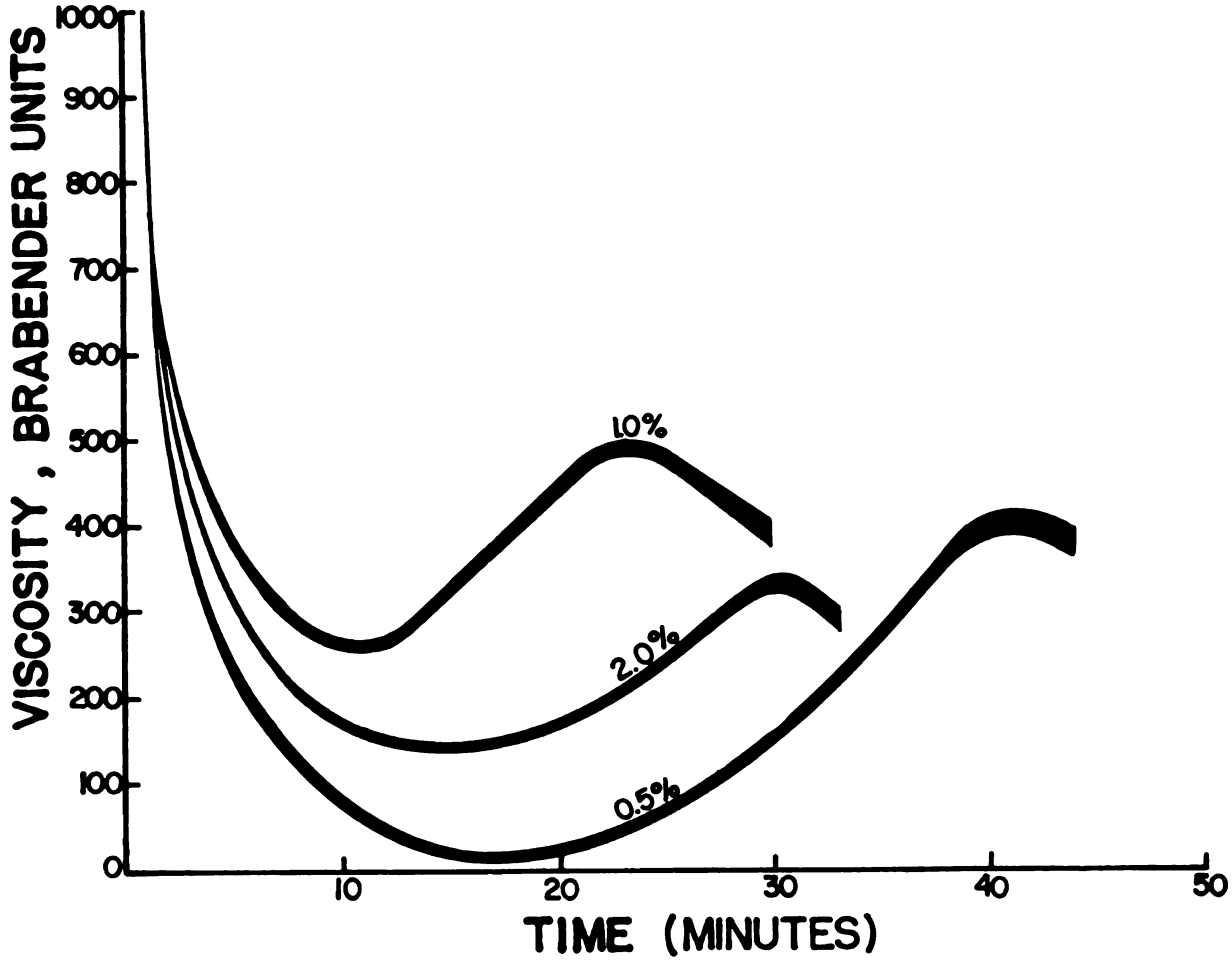


Fig. 10.--The effect of added sodium caseinate on the viscosity of reconstituted freeze-dried cultured cream.

preload or test conditions should be noted. In Fig. 10 the preload was increased to 600 cmg. whereas there was no preload used while testing the samples reported in Fig. 9. This accounts for the difference in the extent to which the samples were broken down by the test conditions. Both sets of results show that starch was ineffective in increasing the viscosity when compared with sodium caseinate. The most important observation to be made with those results was the trend indicated in both Figs. 8 and 9 where 1% added sodium caseinate proved to be the optimum level of addition. Higher and lower concentrations produced a weaker body.

These results parallel those obtained from the addition of MSNF and also suggest that incomplete hydration and destabilization caused by the imbalance of salts and low pH of the medium are responsible for the inferior body noted in certain samples. The fact that calcium and sodium caseinate produced very comparable results tends to suggest that a salt imbalance is not the sole explanation.

The specific environmental conditions that prevail before, after and during coagulation will determine the nature of the coagulum formed. The coagulum may assume a gel structure, a partially coagulated grainy condition or it may separate entirely from the whey serum as a flocculant precipitate. In these trials the effects of heat, acids and freezing on the stability of the caseinate were standardized. Extreme concentration however, due to the higher addition

of caseinate in conjunction with the concentrating effect of freezing probably produced a partially coagulated grainy condition which precipitated out at the lower pH levels (Jenness and Patton, 1959).

A reconstituted freeze-dried cultured cream exhibiting highly desirable body and texture properties was prepared from 20% fat fresh cream fortified with MSNF (2%), caseinates (1%) and an appropriate stabilizer (0.5%). This method of producing a desirable product with little variation between batches thus allowed for the retesting of all parameters and additives as they affect this new process. The results reported in this research are taken from trials where only the factor being examined was changed, suggesting that under the ideal conditions of manufacture all the parameters being tested behaved as has been described.

The Effect of Added Emulsifiers on Some of the Physical Properties of a Freeze-Dried Cultured Cream

In an effort to reduce the high incidence of free fat which developed as a result of the freeze drying process, various emulsifiers were incorporated into the formulation prior to culturing. Trials were based on the hydrophilic-lipophilic balance of emulsifiers (HLB) to determine their effect on dispersibility and free fat in the freeze-dried powders. In these trials, the different levels of emulsifier were dispersed into the cream prior to homogenization by means of an electric stirrer. The emulsifier was weighed

into an 800 ml beaker and mixed with approximately 400 ml of cream. After proper dispersion, the cream plus emulsifier was mixed with the remainder of the 10 lb batch and the mixing was continued as the batch was fed into the homogenizer. Each batch with added emulsifier was double homogenized at 2000 psi. This method of mixing was adopted to ensure proper incorporation of the emulsifier. The amount of added emulsifier varied from 0.1%-0.75% (w/w). Although the higher levels sometimes imparted bitter off flavors to the product, these higher levels were none-theless studied to obtain information regarding their effects at high concentrations. The emulsifiers used were obtained from commercial sources. The results of these trials (Table 8) clearly indicate that emulsifiers at any of the concentrations studied did not reduce the amount of free fat in the powder. The VAG results in Fig. 11 show the detrimental effect of added emulsifiers (0.5%) on the viscosity of the reconstituted product. The addition of emulsifiers resulted in a weaker bodied product. The product emulsified with the lower HLB (3.2) emulsifier was slightly firmer than that of the higher HLB (14.9), although there was not much difference between the two products. Other monoglyceride emulsifiers also investigated include Myvatex 8-20 and Myverol 18-7, 18-30 and 18-85. The results of those trials are not shown since no reduction in free fat was noted. The absence of a distinct water phase in the

Table 8.--The effect of added emulsifier on some of the properties of freeze-dried cultured cream.

Type of Emulsifier	HLB	Rate of Addition (%)	Total Fat (%)	Free Fat mg/g Total Fat	Dispersibility g/100g Sample	Sensory Evaluation Body and Texture (40°F)	
Control	--	--	66.5	945.0	2.9		Sl Grainy
Tween 60	14.9	0.20	66.4	988.0	18.6	V Smooth	Sl Weak
		0.75	66.5	981.0			
Tween 65	10.9	0.20	66.4	991.0	10.0	V Smooth	Sl Weak
		0.75	66.5	956.0			
Span 60	4.7	0.20	66.4	940.0	8.6	Smooth	Sl Weak
		0.75	66.5	936.0			
Atmos 150	3.2	0.20	66.4	957.0	6.8	Smooth	V Sl Weak
		0.75	66.5	981.0			

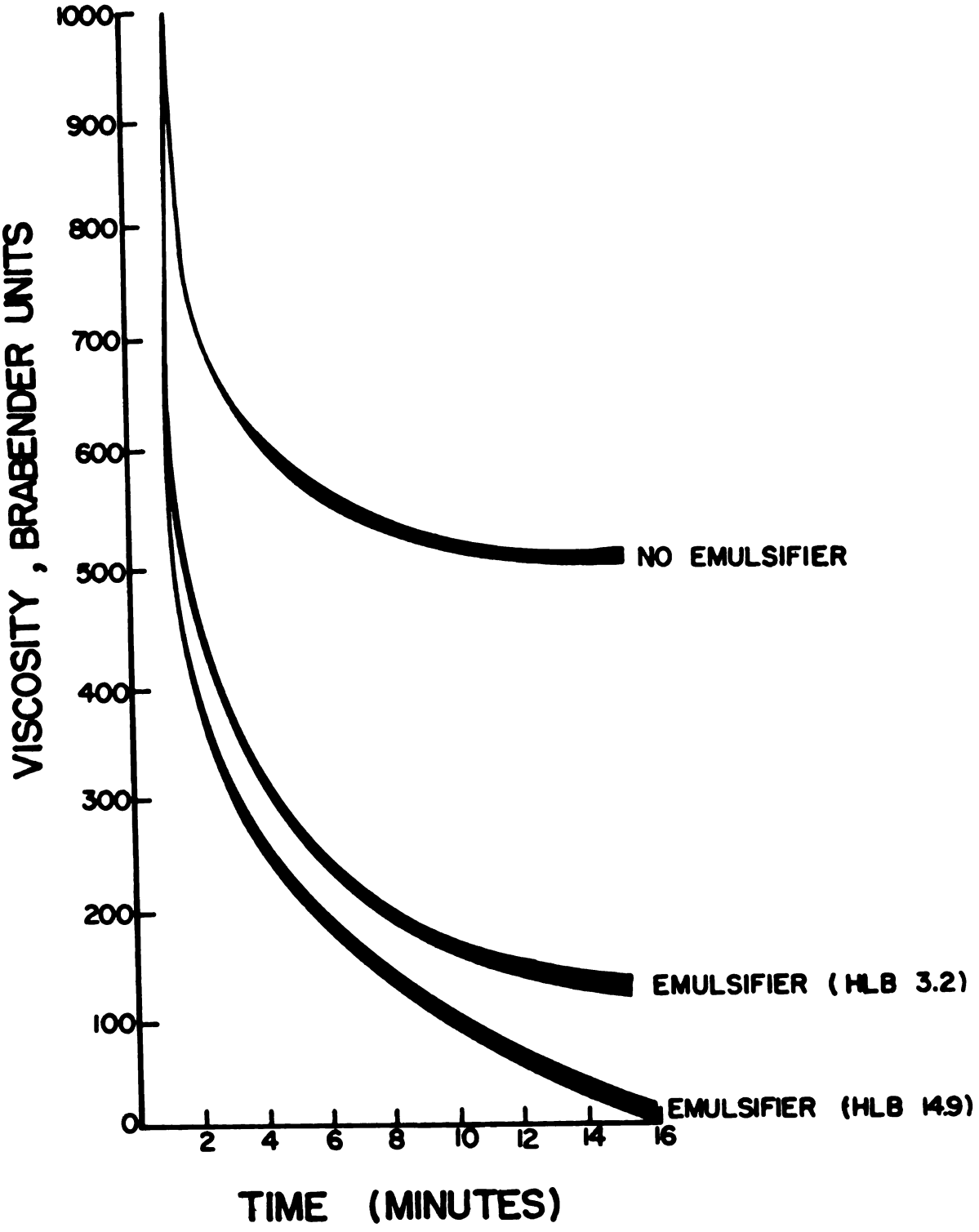


Fig. 11.--The effect of emulsifiers on the viscosity of reconstituted freeze-dried cultured cream.

dry powder possibly accounts for the ineffectiveness of the emulsifiers tested in reducing free fat.

The use of emulsifiers did prove beneficial, however, as they greatly improved the dispersibility of the freeze-dried powders. Fig. 12 shows the difference in texture obtained with added emulsifier. These freeze-dried powders were reconstituted by means of an electric hand mixer, employing a mixing time of three minutes. Examination of the reconstituted cream on the spatula reveals the grainy texture of the control in contrast to the smoother texture of the powders containing added emulsifier. The difference in viscosity between the reconstituted creams with added emulsifiers of different HLB ratings is also apparent. The higher HLB (14.9) produced the weaker bodied product. The weak body of the control is due to poor dispersibility and incomplete hydration of the stabilizers on reconstitution under these conditions. The results in Table 8 corroborate these findings since the dispersibility was increased from 2-6 times that of the control with the aid of added emulsifier.

Freeze Drying of Cultured Cream

Freeze drying has been shown to be superior to spray drying as a method of dehydrating cultured cream (Desai, 1966). Although the flavor of the highly fortified reconstituted cultured cream obtained after freeze drying was superior to the spray dried powder, the flavor was still

Control, no added emulsifier

0.5% Tween 60 (HLB 14.9)

0.5% Tween 65 (HLB 10.9)

0.5% Span 60 (HLB 4.7)

0.5% Atmos 150 (HLB 3.2)

Fig. 12.--The effect of added emulsifier on the dispersibility of a freeze-dried sour cream.

described as flat. Radanovics (1969) had shown that there were inherent losses of the major flavor components during the freeze-drying of model sour cream systems. Trials were therefore carried out to ascertain whether the flat flavor noted in the freeze-dried cultured cream was due solely to the effect of freeze drying or due in part to dilution of the main flavor components by the additives.

Stabilized and unstabilized cultured cream was prepared and freeze dried under the same freeze drying conditions. Diacetyl determinations were run on the fresh and the freeze dried samples, and results were reported on the basis of the total solids content of the fresh product.

The results shown in Table 9 are in agreement with those obtained by Radanovics (1969), suggesting that there is a uniform percentage loss of diacetyl during freeze drying regardless of the initial level. The retention of diacetyl was consistently better in the stabilized than in the unstabilized creams, with an average loss over 10 trials of 49 and 61% respectively. Efforts to reduce these losses and to increase the efficiency of drying were investigated in order to improve the commercial feasibility of this process. Fig. 13 shows a typical drying curve obtained during the freeze drying of cultured cream. Changes in absolute pressure noted in these trials, where the dryer was operating near maximum capacity, served as another indication of the end of the drying cycle. Other workers

Table 9.--The effect of added stabilizers and MSNF on the retention of diacetyl during freeze-drying.

Trial Number	Diacetyl Content (ppm)				
	Fresh Cultured Cream	Freeze-Dried Cultured Cream			Loss (%)
		Stabilized	Loss (%)	Unstabilized	
1	1.00	0.62	38.0	0.48	52.0
2	1.21	0.73	40.0	0.67	45.0
3	1.30	0.73	43.8	0.52	60.0
4	2.40	1.26	47.5	0.98	59.2
5	2.90	1.26	56.5	0.96	67.0
6	3.10	1.65	46.8	1.23	60.3
7	3.10	1.41	54.5	1.03	67.0
8	4.65	2.15	53.8	1.86	59.2
9	5.70	2.82	50.5	1.96	65.6
10	6.05	2.53	58.2	1.35	77.3

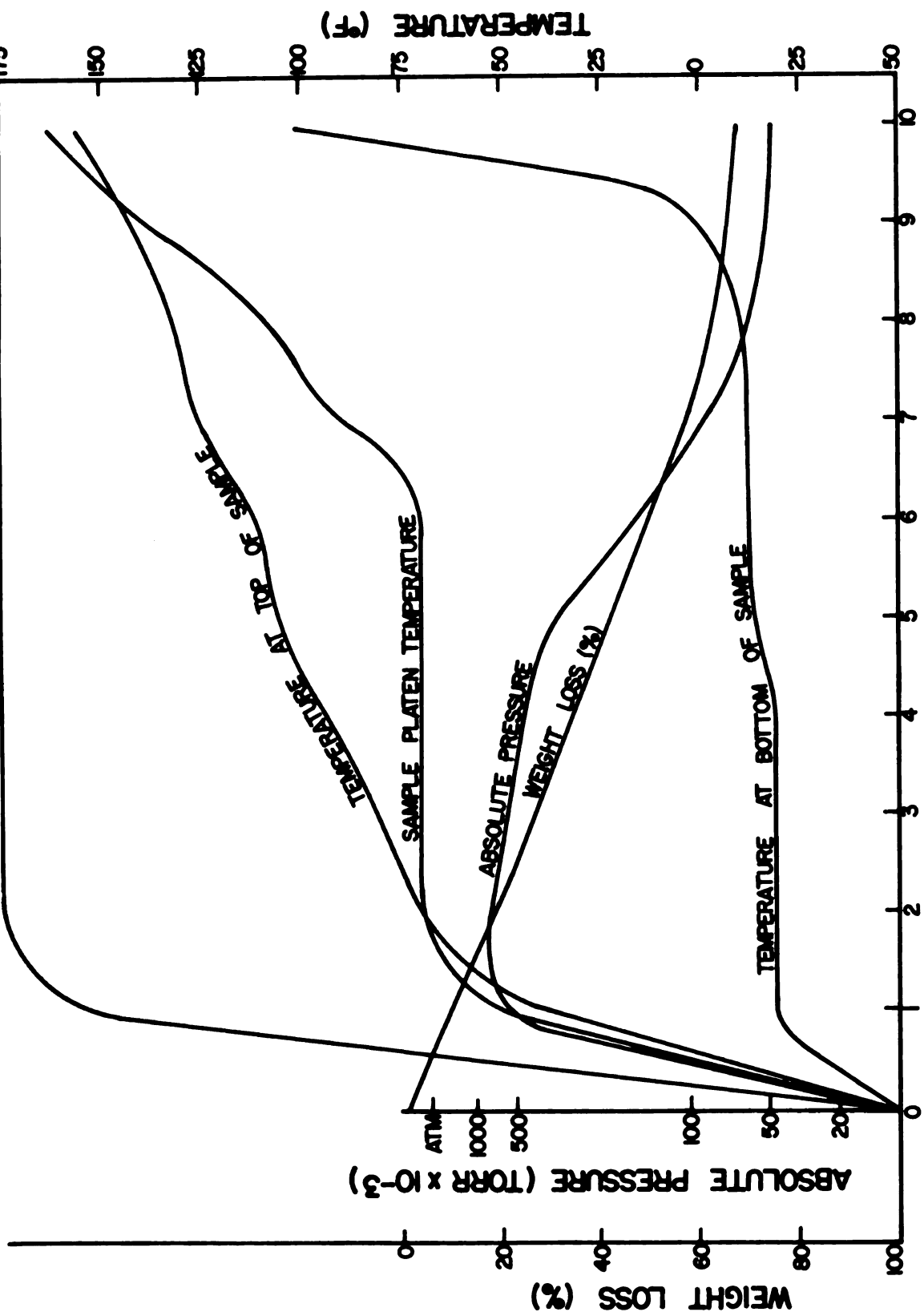


Fig. 13.--Typical drying curve with pressure and temperature profiles of a cultured cream freeze-dried at a platen temperature of 175°F (80°C).

(Saravacos, 1965; Radanovics, 1969) have shown that at relatively high vacuum, variations in chamber pressure did not significantly affect either the freeze drying time or the retention of flavor volatiles. The main parameters to be considered (Fig. 13) are the platen and product temperatures, since proper definition of these temperatures still presents some confusion in the literature. These temperature profiles (Fig. 13) show close similarity to those reported by Radanovics (1969). These results show four different temperatures within the drying chamber after a drying time of 6 hours: namely;

1. the control platen, which attained the set platen temperature of 175F (80C),
2. the sample platen, 70F (20C) where some of the sensible heat has been used to supply the heat of sublimation,
3. the product temperature at the bottom of the tray (12F, -24C) where some of the product was still frozen,
4. the product temperature monitored at the top of the tray, 110F (42C), where the product was dry but still had not attained the temperature of the platen.

These temperatures therefore show that the product was dry before the set platen temperature (175F, 80C) was attained (product at top of tray). An increase in

temperature above that of the ambient air temperature signified that the product was dry, since the cooling effect of the subliming ice phase had been removed. The end of the drying cycle was therefore selected as that point at which the temperature of the product at the bottom of the tray reached 75F (22C). For this situation to be true, it was necessary that the temperature recorded at the bottom of the tray be representative of the sample. To accomplish this, the thermocouple was placed in the geometric center of the tray. The uniform thickness of the product layer was maintained throughout the entire tray, and heat was evenly distributed. This "endpoint" was used after conducting several trials at different platen temperatures which established that under these conditions all the ice in the product was sublimed and the moisture content of the product had equilibrated at between 0.5-1.5%. Temperatures of 200F (93.3C) and above produced a burnt flavored powder which indicated that interstitial melting or heat induced degradation of the powder had occurred. Therefore, the drying method employed at a temperature of 200F (93.3C) and above would no longer be representative of good freeze drying. It was therefore established that 50 pounds of cultured cream could be freeze dried with platen temperature of 175F (80C) to produce a low moisture powder (0.5-1.5%) in ten hours. The results in Table 10 show that increasing the platen temperature from 75F (22C) to 175F (80C) resulted in a

Table 10.--The effect of platen temperature on the loss of diacetyl during the freeze-drying of cultured cream.

Platen Temperature (°F)	Trial Number	Drying Time (Hours)	Diacetyl Content (ppm) Before / After Freeze Drying		Diacetyl Loss (%)
75	1	24.0	6.90	4.00	42.0
	2	25.0	5.50	3.12	43.3
	3	24.0	1.50	0.60	60.0
100	1	20.0	6.90	4.20	39.1
	2	22.0	5.50	2.80	49.0
	3	20.5	1.50	0.55	63.3
150	1	12.25	6.90	4.00	42.0
	2	13.0	5.50	2.80	49.0
	3	12.0	1.50	0.55	63.3
175	1	10.0	6.90	4.30	37.7
	2	10.5	5.50	2.90	47.3
	3	10.5	1.50	0.57	62.0

greater than 50% decrease in drying time from 22 hours to 10 hours. This reduction of freeze drying time as the platen temperature was increased is well documented in the literature (Harper and Tappel, 1957; Burke and Decareau, 1964) where time of drying appears to be a logarithmic function of platen temperature. The results of diacetyl determinations show (Table 10) that when using the endpoint previously described there was no change in diacetyl loss or retention as a function of platen temperature. Even extended exposure to low absolute pressure at platen temperatures between 75F (22C) and 100F (38C) did not increase the diacetyl losses. This finding is contrary to that reported previously (Radanovics, 1969) but is substantiated by Saravacos and Moyer (1968). They found that increased losses in freeze drying was caused by higher product temperatures during the advanced stages of drying. The volatile components, when subjected to high temperatures for extended times, obviously penetrated the membrane barriers as a result of both high vapor pressures and decreased membrane strength. In these trials only diacetyl, one of the major flavor components of cultured cream, was measured. Radanovics (1969) showed that the loss of acetoin resulting from increased platen temperature was higher than that of diacetyl. Boudreau et al. (1966) found the loss of homologous fatty acids from butter was a function of boiling point. Several researchers have demonstrated that the loss

of flavor volatiles was a function of solids concentration (Rey, 1960; Sivetz et al., 1963 and Reineccius et al., (1969).

The retention of flavor volatiles during the dehydrating process at present is attributed to two forces: selective permeability and adsorption. Issenberg et al. (1968) demonstrated the importance of adsorption in the retention of volatiles and suggested that the affinity of the volatile compound for the system in which it was present would determine its ability to adsorb. Since the diacetyl retention in a freeze-dried cultured cream is attributed to adsorption, it is not surprising that greater retention was noted in the stabilized cream (Table 9). The increased losses reported in direct acidified creams (Radanovics, 1969) might also be accounted for by the lower solids level used (with less total adsorbing surface), in addition to excessive exposure to higher temperatures.

Effect of Method of Drying

Two trials were conducted to evaluate the effect of drying methods on some of the physical properties of the resulting cultured cream powder. In these trials one large batch of cultured cream was prepared and divided; one part was freeze-dried and the other spray-dried. The portion to be spray-dried had to be diluted in a 1:1 ratio with water to facilitate drying. The results of these trials are shown in Table 11. The higher loss of diacetyl due to

Table 11.--Effect of method of drying on some of the physical properties of cultured cream.

Method of Drying	Diacetyl Content (ppm)	Penetrometer Values (mm/10)		Free Fat	Sensory Evaluation		
		Quiescent	Stirred	mg/g Total Fat	Flavor	Body (40°F)	Texture
Freeze	1.61	280	368	966.0	Good	Good Firm	V Smooth
Spray	0.35	354	BTM	654.0	Oxid.	Weak Whey	V Grainy

spray-drying indicates as one possibility the effects of exposure to higher temperatures on the retention of diacetyl. The fact that in spray drying the water and water soluble volatile constituents are liquid throughout the constant and falling rate periods may also have an influence on losses during dehydration. The original diacetyl content of the fresh cultured cream was 3.2 ppm resulting in approximately 40% difference in losses between the two methods of drying tested. The spray-dried powder on reconstitution produced a weak body and grainy texture. As a result of spray drying there is much less free fat in the powder, since the disruptive effect of freezing has been avoided. The spray dried powder was non-dispersible, possibly due to partial coating of the exterior of the particle with fat. Variable heat damage during drying and resulting insolubilization of the particle is another possible cause of the poor dispersibility.

Bradley and Stine (1964) observed that the retention of flavor volatiles was enhanced by lower powder temperatures, and that the retention of volatiles increased directly as the particle size. The higher retention was attributed to the volatiles being trapped in the particles. The freeze-dried powder produced during this study were relatively more porous and larger in size than the corresponding spray-dried powders. This larger particle and the lower temperatures utilized in freeze drying, which

complement the adsorption theory, would therefore account for the higher losses of diacetyl in spray drying.

There was a distinct difference in the physical appearance of the cultured cream dehydrated by the two drying methods tested. The deeper yellow color of the freeze-dried powder appeared to be due to the amount of fat liberated as free fat and the state of its dispersion. The lower free fat content of the spray-dried powder would suggest that the effect of visible fat on the color of the powder was reduced. Berlin et al. (1964) observed by fluorescence microscopy, that the free fat in spray dried powders was present on the surface in the form of lakes and pools. Microscopic examination by Nickerson et al. (1952) showed that free fat in freeze-dried whole milk was dispersed throughout the mass of the particles. The ease of dispersibility of a dehydrated product is greatly influenced by its lipid content (Ashworth, 1955). Investigations by Tamsma et al. (1958) indicated a positive relationship between free fat content of foam spray-dried whole milk and its dispersibility. The dispersibility was unaffected by amounts of free fat up to 40%, but decreased as the levels increased from 40 to 95%. The greater increase in dispersibility noted with the higher HLB emulsifiers (Table 8) more strongly suggests the orientation of the emulsifier around the fat at the surface of the particle. The exposed hydrophilic portion of the emulsifier molecule readily

associates with the water, resulting in rapid dispersion of the fat and hydration of the protein.

Nickerson et al. (1952) further showed that although the mechanism of oxidation appeared to be the same with spray-dried and freeze-dried whole milk powders, the rates of oxidation were different. Even with the high levels of free fat in freeze-dried powders, the spray-dried powders became tallowy and developed peroxides much more rapidly. Therefore the high free fat content of the freeze-dried cultured cream powders might not result in any appreciable difference in the stability of the stored powders.

The Flavor of the Reconstituted Freeze-Dried Cultured Cream

No study directed toward product development in the food industry is complete without consumer acceptance, since palatability of the commodity in question largely influences its success on the consumer market.

The reconstituted freeze-dried cultured cream prepared according to the procedures previously discussed has exhibited excellent and desirable characteristics of body and texture. One criticism, however, has been the mild, somewhat "flat" flavor of the reconstituted product when compared to the fresh counterpart. This flat flavor was attributed in part to the 50% loss of diacetyl which occurred during the freeze-drying process. Hempenius et al. (1969b) however, indicated that the volatile acidity content

was a more accurate indicator of the flavor level present in cultured cream than diacetyl.

Appropriate experiments were designed to determine if the concentration of flavor constituents of the fresh cultured cream could be increased prior to dehydration in order to achieve higher residual levels of these essential volatile materials in the dehydrated product.

Preliminary flavor evaluations were made on reconstituted freeze-dried cultured creams which contained different levels of diacetyl and titratable acidity. The samples were evaluated by a trained panel of judges using the evaluation sheet (Appendix Fig. 2). The evaluation sheet (Amerine et al., 1965) was designed to ascertain which factor, in the opinion of each judge, attributed more to his idea of a desirable cultured cream flavor and which factor or combination of factors would result in the upgrading or downgrading of the product. Table 12 shows results that are representative of these trials. Trial 1 shows the comparison of a stabilized and unstabilized fresh cultured cream. Although the stabilized cultured cream contained higher levels of both the flavor components, namely diacetyl and acid, the unstabilized product received a higher overall score. This was attributed to the dilution or masking effect of the stabilizer additives on the flavor of the stabilized cultured cream.

Table 12.--Sensory evaluation of freeze-dried cultured cream as influenced by the diacetyl content and the titratable acidity.

Trial		ppm Diacetyl	Titratable Acidity	Sensory Evaluation**				
				Diacetyl	Acid	Flavor Intensity	Freshness	Overall
1	unstabilized	0.73	0.72	10	10	10	12	10
	stabilized	0.85	0.90	8	10	9	8	8
2	stabilized	4.0*	0.82	11	10	11	12	11
	stabilized	2.33	0.81	8	11	7	9	9
	stabilized	1.20	0.81	8	10	8	7	8
	stabilized	4.00	0.82	10	8	9	10	9
3	stabilized	0.38	0.87	4	5	4	5	5
	stabilized	0.40	0.72	3	3	1	2	2
	stabilized	0.80	0.90	8	9	9	8	8
	stabilized	1.70	1.00	11	11	12	11	12
4	stabilized	1.83	1.10	11	11	11	12	12
	stabilized	1.23	1.20	11	11	10	11	11

*Identical samples presented to judges at the same time in the order reported.

**Mean scores of five judges based on Hedonic Scale 1-13.

Trial 2 compared the effect of different levels of diacetyl at fixed titratable acidity on the overall flavor. These results show that the higher diacetyl content of reconstituted cultured cream was generally preferred. This trial also included two identical samples, which, although they were not discerned by all the judges, indicated the anticipated trend.

The product was assumed to be acceptable on each flavor component if it received a score of 9 or more when using a hedonic scale of 1-13, as shown on the evaluation sheet (Appendix Table 2). Each value represents the average score of 5 or 6 judges corrected to the nearest whole number. The trends indicated by these trials are in agreement with other work done with flavor thresholds of cultured cream (Hempenius et al., 1969b) and suggested that a titratable acidity of 0.9-1.0% was required for desirable flavor in the reconstituted sample. The judges invariably scored flavor intensity on the basis of diacetyl; a sample with low titratable acidity was however, invariably downgraded. In addition to the appreciation of the preferences of the judges, it was also observed that the higher titratable acidity always produced a firmer bodied product on reconstitution. This was also shown by Guthrie (1963) with reference to fresh cultured creams.

The Effect of Different Pretreatments on the Production of Acid and Diacetyl

A frozen culture concentrate obtained from a commerical source added at the rate of 2% was found to be highly dependable in producing a clean flavored product under the conditions used. However, in an effort to enhance the production of the flavor components, trials similar to those reported in the study of viscosity were carried out to determine the effects of heat treatment, additives and culturing techniques on the production of diacetyl. Fortification of the sweet cream with 2% MSNF was observed to be an optimal in enhancing the production of acid since the higher concentrations, although permitting high acid production, contributed salty flavors and a weaker body.

The pasteurization treatment of 175F (80C) for 30 minutes was maintained, as higher heat treatments are recommended for the inactivation of phosphatase and the equivalent of 99.9% destruction of coliform organisms in high fat creams (Hening and Dahlberg, 1943).

Proper incorporation of the frozen culture and careful monitoring of temperatures and times of incubation permitted the development of the required titratable acidity (0.9-1% expressed as lactic acid).

To enhance the production of diacetyl, several methods were investigated. These included:

1. addition of sodium citrate and manganese (Man and Galesloot, 1962; Seitz et al., 1963b);

2. reducing the pH by direct acidification prior to culturing (Yano and Ozawa, 1956);
3. use of hydrogen peroxide and catalase (Pack et al., 1968).

The results of these trials (Tables 13 and 14) show that all these treatments increased the production of diacetyl. The use of citric acid to reduce the pH from 6.5 to 5.0 (Table 14), resulted in the greatest increase in diacetyl production. Very favorable results were also obtained with the addition of 0.2% sodium citrate in conjunction with 2 ppm manganese. The addition of hydrogen peroxide and catalase was not as effective in increasing the production of diacetyl as the other methods tested. In addition, the cultured cream reconstituted from powder treated in this way invariably exhibited a weaker body.

Table 13.--The effect of different pretreatments on the production of diacetyl in fresh stabilized cultured cream.

Pretreatment	Addition (ppm)	Diacetyl Produced (ppm)	Sensory Evaluation		
			Body	Texture (40F)	Flavor
Control	---	1.38	Good	Smooth	Flat
Sodium Citrate	2000				
Manganese	2	4.35	Good	Smooth	Good
Hydrogen Peroxide	30				
Catalase	As Reqd.	2.10	Weak	Grainy	Good

Table 14.--The effect of reducing pH by direct acidification prior to culturing on the production of diacetyl.

Acid Used	Acidified pH	Diacetyl Produced (ppm)
Control	6.5	1.20
Lactic	6.0	1.90
	5.0	2.60
Hydrochloric	6.0	1.70
	5.0	2.34
Citric	6.0	3.00
	5.0	6.07

Of these treatments, the addition of 0.2% sodium citrate and 2 ppm manganese was adapted for subsequent use because of the ease of this treatment when compared with the others. In other applications, however, direct acidification might be preferred to the addition of sodium citrate. Since sodium citrate emulsified the product, reducing the viscosity (Vujicic et al., 1968), whereas the direct acidification was shown to produce a firmer bodied reconstituted product. The results shown in Fig. 14 are representative of trials in which the body produced by natural fermentation was compared with that produced by direct acidification to the same pH. These results indicate that a better body was obtained by direct acidification, but as reported by other researchers (Freeman and Bucy, 1969), the flavor of non-culture sour cream is not equal to that made by conventional culturing.

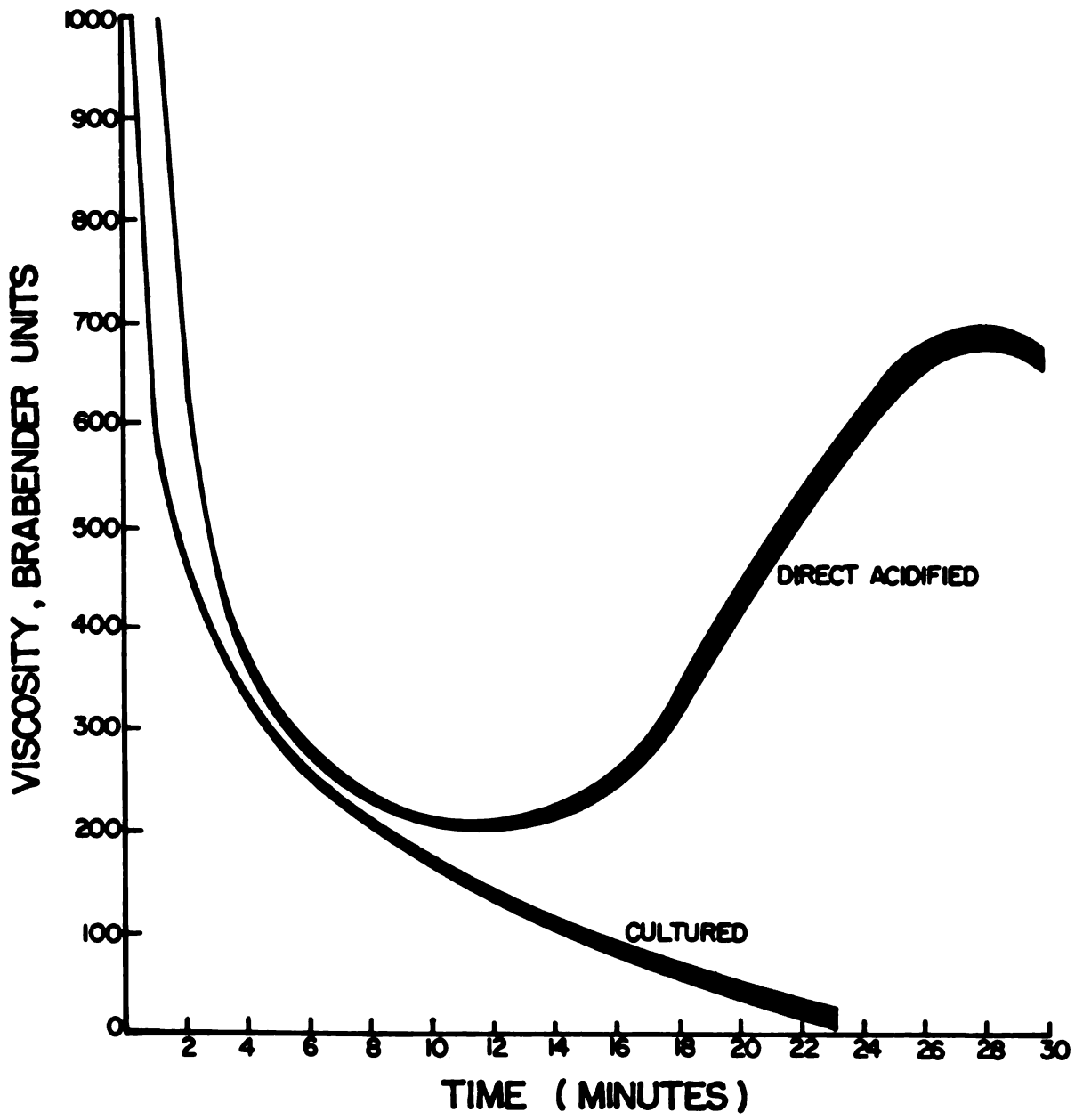


Fig. 14.--A comparison of the viscosity of reconstituted freeze-dried sour creams prepared by direct acidification or natural fermentation.

The flavor of cultured cream is dependent on a fermentation process involving acid and flavor producing organisms where sweet pasteurized cream provides the substrates. Therefore, the quality of the original raw milk is important (Greene and Jezeski, 1957a). Anderson et al. (1955) also indicated that there was a correlation between the peptide fraction in milk and starter activity. The nature of the subsequent treatment has also been shown to affect the suitability of the milk as a starter medium. Certain manufacturing processes, especially heating, were shown by Greene and Jezeski (1957b) to alter the substrate rendering it stimulatory in some cases and inhibitory in others. Milk as it is secreted contains very little non-protein nitrogen, hence the nitrogen requirements of the cultures have to be met by hydrolysis of protein (Speck, 1964). Since all organisms are not proteolytic, heat treatment which results in hydrolysis of protein can improve the growth of organisms, while grossly overheating the milk might have a deleterious effect on the same organism (Foster, 1952). Horrall and Elliker (1950) reported that reconstituted milk promoted more constant activity in starters than did selected whole milk. Increasing the MSNF content of reconstituted NFDM was found to stimulate acid production (Yano et al., 1960) possibly because of the buffering action, and a higher concentration of growth factors in the milk.

Present day starter cultures employ either L. citrovorum or S. diacetilactis or both for the production of aroma compounds desirable in cultured cream. The leuconostocs grow best in association with any of a variety of the strains of S. lactis or S. cremoris, which produce lactic acid from lactose. The presence of either S. lactis or S. cremoris is beneficial to sufficiently reduce the pH of the medium and thereby initiate leuconostoc activity.

The schemes for diacetyl and acetoin synthesis by L. citrovorum (Andersen, 1959) and S. diacetilactis (Seitz et al., 1963b) were found to be the same. The interconversion of diacetyl, acetoin and 2,3-butanedial is based on their relationship through an oxidation reduction mechanism. The amount of oxidized or reduced substances in the medium, therefore, determines the corresponding proportion of these compounds formed.

The most potent diacetyl-producing organisms are paradoxically, the ones most active in its destruction. Thus, of the lactic streptococci, S. diacetilactis exhibits the strongest diacetyl reducing potential (Seitz, 1963a). This ability is attributed to the presence of certain enzyme systems with which the bacteria are endowed and which are activated under favorable conditions. In the process of manufacture of a freeze-dried cultured cream, however, these enzyme systems appear to be destroyed. Fig. 15 shows a comparison of the loss of diacetyl in fresh commercial and

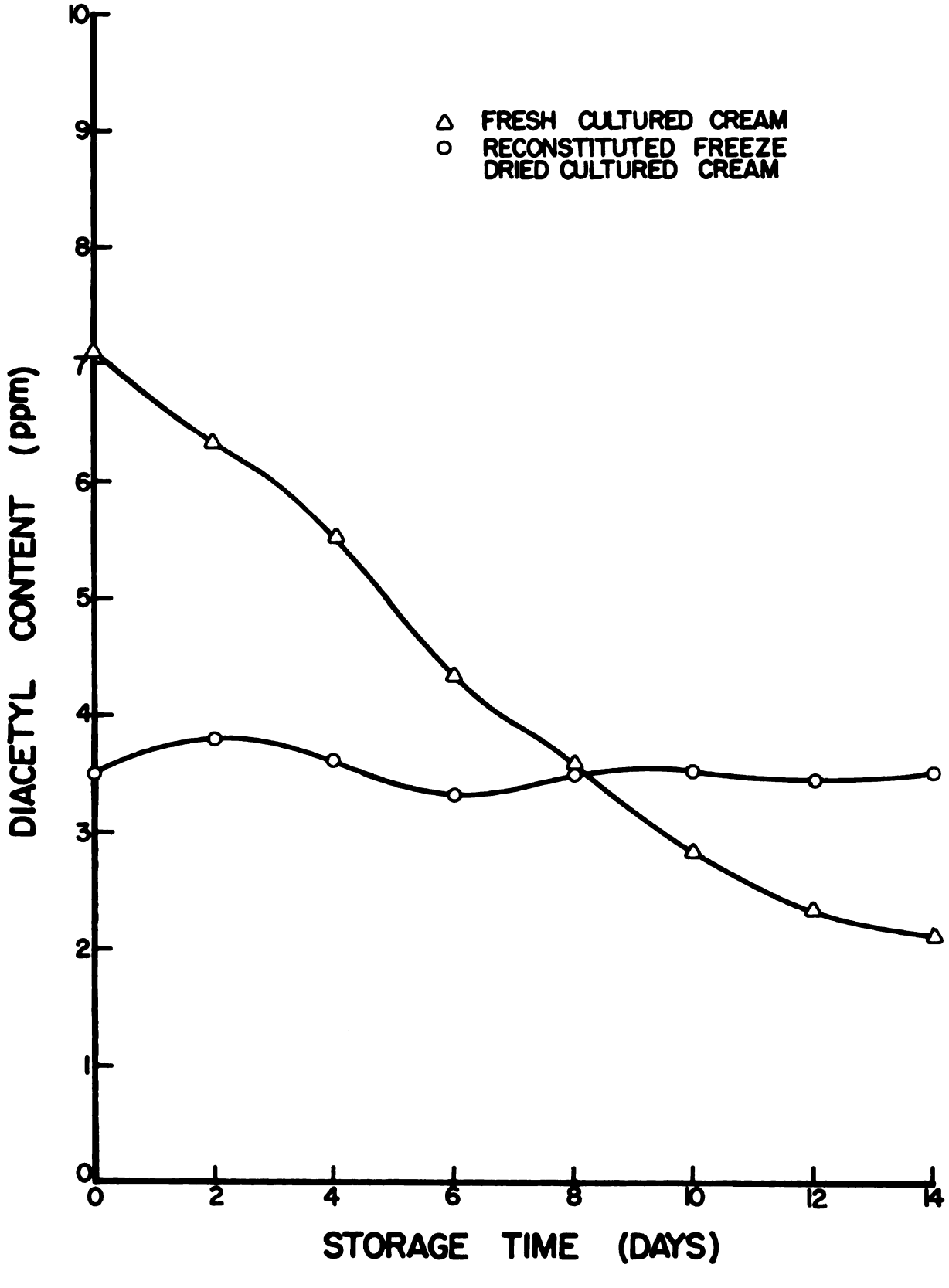


Fig. 15.--The loss of diacetyl in fresh and reconstituted freeze-dried cultured cream during storage at 40F (4.4C).

reconstituted freeze-dried cultured creams stored at 40F (4.4C). The rapid loss of diacetyl (70%) from 7.1 to 2.1 ppm in a 14 day period is indicative of the activity of the diacetyl reductase enzyme system in the fresh cultured cream. On the other hand, although the diacetyl content of the reconstituted cultured cream was lower initially, it remained constant throughout the storage time over which it was tested. These results corroborate the findings of Stine et al. (1967). This suggests greater stability of the reconstituted cultured cream over and above the fresh cream. The fresh commercial creams in contrast to the reconstituted creams showed other definite signs of deterioration, such as whey formation and a decrease in viscosity.

The adsorption and desorption of various flavor components, as the moisture of the freeze-dried product was raised, was observed by Flink and Karel (1969). This prompted the investigation of the adsorption and retention of diacetyl by low moisture freeze-dried cultured cream powders. The powder was exposed to an atmosphere saturated with diacetyl for varying lengths of time in a closed container maintained at 72F (21C). Each treatment was standardized, so that the conditions within the container were the same in all trials. The results (Table 15) show the rapid adsorption of diacetyl, such that in 15 minutes the powder was supersaturated with diacetyl. After 30 minutes of exposure, equilibrium conditions appeared to have

Table 15.--The adsorption of diacetyl by low moisture powders exposed to an atmosphere saturated with diacetyl.

Time of Exposure (mins)	Diacetyl Content (ppm)					
	Trial 1		Trial 2		Trial 3	
	Before	After	Before	After	Before	After
0	1.8	--	3.55	--	2.1	--
15	1.8	19.43	3.55	13.83	2.1	13.00
30	1.8	8.20	3.55	12.10	2.1	5.00
60	1.8	9.16	3.55	12.38	2.1	6.46
90	1.8	9.32	3.55	12.00	2.1	6.30

been attained. Longer times of exposure of 60 and 90 minutes did not result in any further change in diacetyl content. An adequate picture of the retention of the diacetyl was not obtained, as it appeared to be dependent on the extent to which the package was opened and closed and the atmospheric conditions that existed during sampling. Even after regular sampling for up to 3 months, most of the powder still retained a nearly constant level of diacetyl 2-3 times that of the original powder. These observations suggested that diacetyl could have adsorption-desorption isotherms similar to those of water vapor. There was, however, a certain level of diacetyl that was strongly adsorbed to the freeze-dried cultured cream.

Based on the results obtained in this and other research carried out in this laboratory (Radanovics, 1969) the desired level of volatile flavors can be adjusted in the final product by appropriately increasing the initial concentration of such materials. This increase in the original concentration can be achieved by the proper selection of raw materials, improving culturing techniques in conjunction with the addition of essential metabolites, or by the addition of natural or chemical flavor components.

The Storage Stability of Freeze-Dried Cultured Cream

For a freeze-dried cultured cream to be commerically attractive, the dehydrated product would have to possess good stability in storage at ambient temperatures of 72F (21C) to 80F (27C) for periods of up to 6 months. Such stability requirements would encompass both chemical stability towards lipid oxidation and retention of desirable flavor characteristics and physical stability with regard to body, texture and dispersibility properties. Several large batches of good quality cultured cream were freeze-dried, triturated and packaged in large glass jars. These powders were stored in large wooden constant temperature cabinets. The storage studies were designed to determine the effect of level of moisture and temperature of storage on some of the physical and chemical properties of the

freeze-dried product. Sensory evaluations, viscosity and diacetyl content were used as criteria of quality.

The effects of moisture content and temperature of storage on the stability of dehydrated products have been studied extensively. Too high a moisture content initiates such chemical reactions as browning. Reduction of moisture below the level necessary to form a monolayer accelerates the oxidation of fats presumably by permitting direct contact with the oxygen of the air (Aceto et al., 1965, 1966). These changes have been shown to be accelerated by increases in temperature (Tamsma et al., 1963).

Preliminary storage studies on each batch of cultured cream freeze-dried throughout this study indicated:

1. there was a loss of body and a decrease in viscosity in cultured cream reconstituted from stored powders;
2. the stored powders produced a grainy texture;
3. the development of off flavors in the stored powder, masked the true cultured cream flavor; and
4. powders with a higher titratable acidity and a lower moisture content exhibited better storage stability.

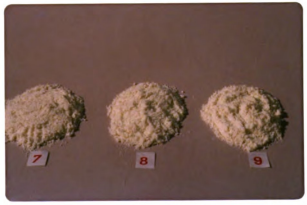
Four large batches were therefore carefully prepared, controlling all processing conditions to minimize variation in the finished product. The powders from these batches

were then air packed and stored at temperatures of 40, 75 and 100F. The moisture contents of portions of each powder from each batch were increased, to vary the moisture contents of the powders from 0.85-5.2%. The initial moisture, diacetyl content and viscosity or body properties were then determined and the powders placed in storage.

At regular intervals, portions were removed from storage for examination. The results of these trials are shown in Table 16 and Figs. 16-20. The effect of increasing moisture content is shown in Figs. 16-18. The poor quality of the higher moisture powders was based on greatly accelerated Maillard browning during storage of the powders. Fig. 16 shows the absence of Maillard browning in those powders of: (1) 0.85, (2) 1.25, and (3) 1.85% moisture stored at 40, 75 and 100F for 6 months. In Fig. 17, the moisture content of the powders has been increased to: (1) 2.35%, (2) 3.38% and (3) 4.89% and the effect of higher temperatures at these higher moisture levels is clearly seen. At storage temperatures of 75F (22C) the (5) 3.38% and (6) 4.89% show definite browning. Cultured cream reconstituted from these brown powders was described as objectionable and stale, with pronounced off-flavors. The same effect of moisture and temperature of storage is shown in Fig. 18. In this figure, the moisture contents range through (1) 1.75%, (2) 3.83% and (3) 5.22% and an optimal level of below 2% was indicated. Even at 40F (4.4C) the

Moisture content (%): (1) 0.85, (2) 1.25, (3) 1.85.
Powders stored for 6 months at 40 F (4.4C)

Moisture content (%): (4) 0.85, (5) 1.25, (6) 1.85.
Powders stored for 6 months at 75F (22C)



Moisture content (%): (7) 0.85, (8) 1.25, (9) 1.85.
Powders stored for 6 months at 100F (38C)

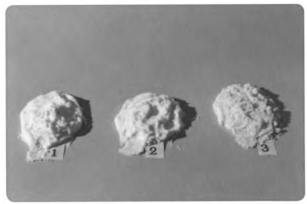
Fig. 16.--The effect of temperature and time of storage on Maillard browning in low moisture freeze-dried cultured cream powders.

Moisture content (%): (1) 2.35, (2) 3.38, (3) 4.89.
Powders stored for 6 months at 40F (4.4C)

Moisture content (%): (4) 2.35, (5) 3.38, (6) 4.89.
Powders stored for 6 months at 75F (22C)

Moisture content (%): (7) 2.35, (8) 3.38, (9) 4.89.
Powders stored for 6 months at 100F (38C)

Fig. 17.--The effect of temperature and time of storage on Maillard browning in high moisture freeze-dried cultured cream powders.



Moisture content (%): (1) 1.75, (2) 3.83, (3) 5.22.
Powders stored for 6 months at 40F (4.4C)

Moisture content (%): (4) 1.75, (5) 3.83, (6) 5.22.
Powders stored for 6 months at 75F (22C)

Moisture content (%): (7) 1.75, (8) 3.83, (9) 5.22.
Powders stored for 6 months at 100F (38C)

Fig. 18.--A comparison of the effect of low and high moisture contents and different storage temperatures on Maillard browning in freeze-dried cultured cream powders.

browning reaction was apparent in the higher moisture powders after storage for 6 months. At 75F (22C), browning was more pronounced at the higher moisture levels and was described as objectionable. The powders stored at 100F (38C) indicate clearly that at moisture levels of 1.75% (1) there was no evidence of browning even after storage for 6 months. The higher moisture powders, on the other hand, had deteriorated rapidly. The absence of evident browning in lower moisture powders even at higher temperatures of storage was shown in Fig. 16. Interim observations and examination of the powder during the storage period also indicated that the rate of development of browning was indicated by the degree of color developed at the end of 6 months. Powders of 3-5% moisture all showed signs of browning within the first 3 weeks of storage, while the samples with 2.35% (1) moisture did not show signs of browning until after 2 months at 100F (38C). The presence of browning in the powder of 2.35% moisture at temperatures of 100F (38C), however, indicates that this was too high a moisture for optimum storage stability, since there was enough free moisture present to initiate the browning reaction. This did not occur in the powders tested which contained less than 2% moisture. The chemical changes that result from the Maillard browning reaction interferred with both the diacetyl and the viscosity determinations on the stored powders. At the initiation of browning the diacetyl

determinations indicated marked increases in diacetyl content due to interferring colored compounds that were formed. Maillard browning also resulted in denaturation of the proteins in the stored powder, hence the dispersibility of the powder was impossible, rendering any determination of viscosity or body properties quite meaningless.

The results of the loss of diacetyl and change in viscosity of powders, with an initial moisture content of 1.53% are shown in Figs. 19 and 20. These results (Fig. 19) indicated that at temperatures of 75F (22C) and 40F (4.4C) the loss of diacetyl was minimal over a storage period of 6 months. The powders with an initial diacetyl content of 1.7 ppm retained 1.1 and 1.4 ppm diacetyl respectively after 6 months of storage. There was also a loss of moisture in these stored powders which was related to the temperature of storage. From an initial moisture content of 1.53% after 6 months, the powders stored at 100F (38C), 75F (22C) and 40F (4.4C) contained 0.96, 1.23 and 1.42% moisture, respectively. This loss of moisture was attributed to improper packaging. Proper packaging, for example canning in an inert atmosphere, was not investigated because of the amount of sample required for viscosity and diacetyl determinations and the lack of available storage space. In addition, previous work in this laboratory had shown improved storage stability with improved storage techniques. The sensory evaluation, the results of which are shown in

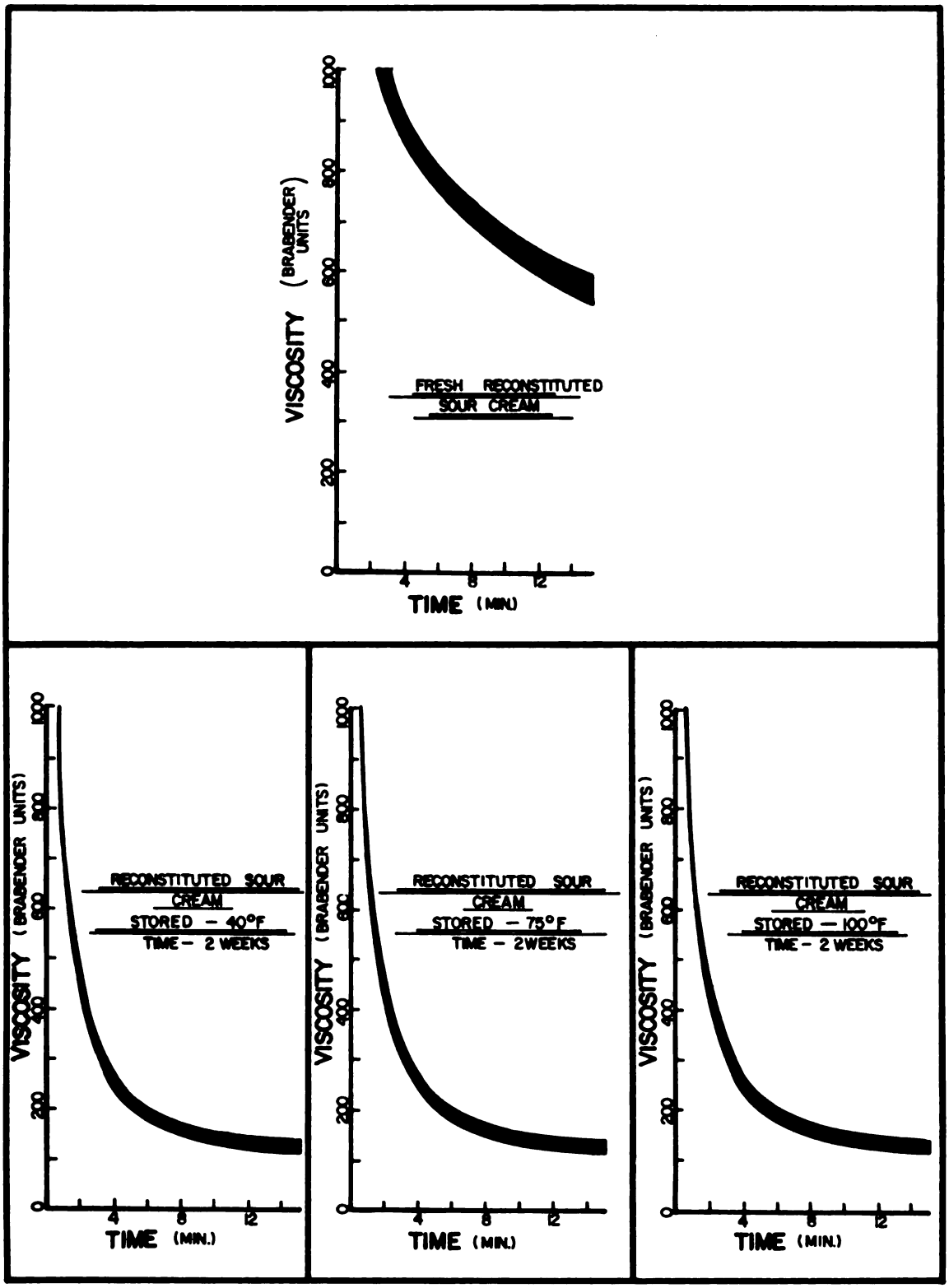


Fig. 19.--The change in viscosity of cultured cream reconstituted from powders stored at different temperatures.

Table 16, did show that there was little difference between the effect of different storage temperatures on the storage stability of the powders of such low moisture. These results are reported as the mean scores of 5 or 6 judges based on the hedonic rating of 1-13 shown in Appendix (Table 2). The mean values are reported since statistical analysis did not indicate any further differences not apparent in the table. Throughout the storage period, the cultured creams reconstituted from the stored powders was considered by the judges to possess a desirable flavor, body and texture and was only downgraded on the characteristic or property described as freshness. After 6-12 weeks the powders stored at 100F (38C) were downgraded and judged unacceptable by virtue of an overall mean score of less than 9.

The results of changes in viscosity of the cultured cream reconstituted from the stored powders are shown in Fig. 20. This decrease in viscosity after 2 weeks storage was representative of the entire storage study. There was no further discernible change in viscosity for the duration of the storage time (6 months). This breakdown in body and the difficulty encountered with dispersibility of the stored powder has suggested that these problems in storage may be due to a loss of solubility in the powder. This loss of solubility may be due to one or a combination of several factors, for example:

Table 16.--Sensory evaluation of cultured cream reconstituted from freeze-dried cultured cream (1.5% moisture) stored 40F (4.4C), 70F (21C) and 100F (38C) for 24 weeks.

Time in Storage (Weeks)	Temperature of Storage (F)	Mean Hedonic Scores of Five Trained Judges[1]						
		Diacetyl	Acid	Flavor Intensity	Freshness	Body	Texture	Overall
0	--	11	11	12	11	11	12	12
1	40	10	11	10	9	10	11	11
	70	10	10	10	10	11	12	10
	100	10	11	10	9	11	11	10
2	40	9	10	9	8	8	9	10
	70	8	9	8	7	9	9	8
	100	9	9	8	8	9	10	7
4	40	10	10	10	9	10	11	9
	70	9	10	9	9	9	10	9
	100	9	9	9	6	9	11	7
6	40	9	11	10	7	10	10	8
	70	10	11	10	10	10	9	10
	100	10	10	9	6	11	11	7
8	40	10	10	10	10	11	10	10
	70	10	10	10	9	10	9	9
	100	9	9	9	8	9	10	9
12	40	10	10	10	9	10	10	10
	70	9	10	9	9	9	10	9
	100	9	10	9	8	9	10	8
18	40	10	11	10	10	10	10	10
	70	10	10	9	9	11	11	10
	100	8	10	8	6	11	8	8
24	40	10	11	10	9	10	10	10
	70	10	9	9	8	10	11	9
	100	8	9	9	6	10	9	7

[1]Hedonic range 1-13.

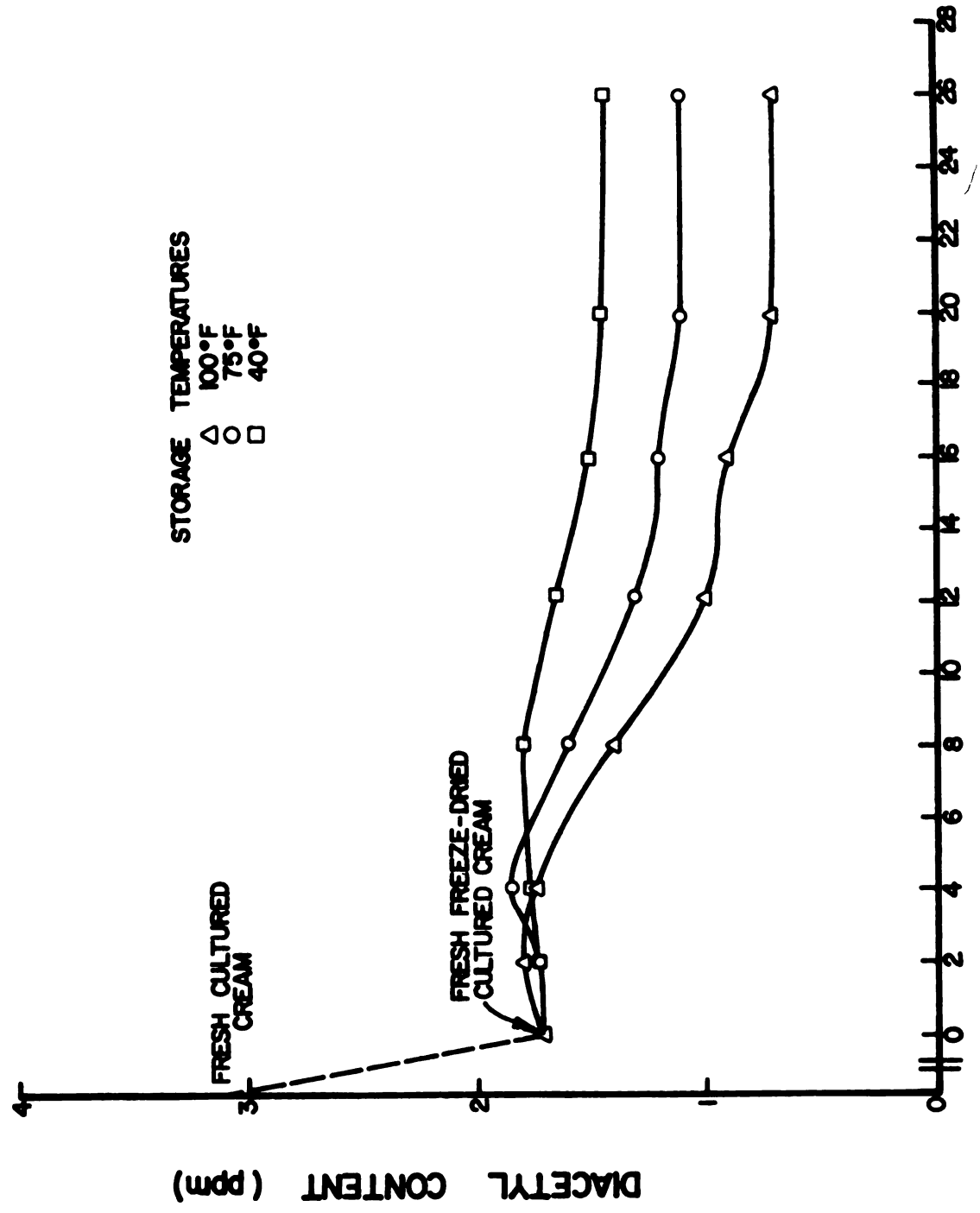


Fig. 20.--The effect of different storage temperatures on the diacetyl content of low moisture freeze-dried cultured cream.

1. breakdown of the stabilizers;
2. orientation of polymorphic forms of fat at the surface of the particles;
3. Maillard browning reaction;
4. combinations of the above.

The VAG results (Fig. 7) seem to suggest that there was some breakdown of stabilizer involved since the high viscosity upon agitation was completely destroyed in storage. However, if the concentration of free fat at the surface of the low moisture powder prevented complete hydration of the stabilizer, then the true stabilizing effect would not be realized.

The improved dispersibility of the stored powders obtained with the use of added emulsifier seems to attach more significance to the orientation of polymorphic forms of fat to the surface of the particle as the main cause of insolubility developing in the stored powder. There was, however, some breakdown in stabilizing effect as a result of the freeze-drying process, accounting for the great difference in viscosity between the fresh cultured and fresh reconstituted cultured cream.

The improved storage stability of the freeze-dried cultured cream powder can be attributed to many factors which are all well documented in the literature. Various processing treatments have been shown to improve the storage stability of dehydrated products. Manus and Ashworth (1948)

showed that air packed whole milk powder prepared from milk which had been preheated to 180F (82C) for 10 minutes or 170F (76.6C) for 30 minutes possessed slightly better palatability for up to 10 months of storage. The activation of sulfhydryl groups during preheating served to retard the development of oxidation. Tarassuk and Jack (1946) showed that adequate preheat temperatures are essential for reducing the potential for staling. Bradley and Stine (1964) showed that cheese powders receiving the higher preheat treatment during manufacture developed less stale flavor.

The ease of dispersion of a dehydrated product is greatly influenced by its lipid content (Reinke and Brunner, 1959). The dispersibility of dried milks was observed by Ashworth (1955) to decrease with increasing total fat. Investigations by Tamsma et al. (1958) indicated a positive relationship between the free fat content of foam-spray-dried whole milk and its dispersibility. The use of hydrophilic emulsifiers should, therefore, improve the dispersibility of powder with high levels of free fat, as was noted in this research. Similarly, if the insolubility of the stored powders was completely due to destabilization of the protein, the proper dispersion of fat with the aid of emulsifiers would not markedly improve dispersibility of the stored powder.

The results obtained suggest that while there was some breakdown in the stabilizing systems, the main reason for the development of this degree of insolubility in the stored powder was due to orientation of different polymorphic forms of fat at the surface of the powder during storage. It is also possible that these changes taking place in the physical structure of the fat might account for the decrease in viscosity or body properties noted in the first two weeks of storage. Careful study of the effects of freeze-drying on the crystalline structure of milk fat might furnish much needed information regarding this loss of viscosity. The fat content represents approximately two-thirds of the total solids present in cultured creams, and therefore would be expected to greatly affect the viscosity of the system. In addition, of all the individual components known to be present in the cultured cream, it appeared that the fat phase underwent the greatest changes.

Sensory Evaluation of the Reconstituted Freeze-Dried Cultured Cream

Since the standards of identity set for dairy products by the professional graders might actually indicate their preferences rather than those of the consumer, the reconstituted freeze-dried cultured cream was tested for consumer acceptability.

Cultured cream reconstituted from freeze-dried powder (previously referred to in Table 16) which had been

stored for 4 weeks at 75F (22C) was evaluated for consumer acceptability by 92 women. This sensory evaluation was designed to determine the overall acceptability of the product. Each lady was given approximately a 3/4 ounce sample of the chilled (40F, 4.4C) cultured cream in a portion cup along with a plastic spoon, and asked to fill out the questionnaire shown in Appendix Fig. 3. The results of the evaluation (Table 17) indicate quite clearly that this group of housewives found the product acceptable. Of the 92 housewives tested, 88 scored the product acceptable and only 2 thought it unacceptable. Two of five women who were not regular users of cultured cream did not evaluate the product. As the usage of cultured cream increased, the number of preferences for individual characteristics increased. This tends to suggest that as the women became more trained (from the regular use of cultured cream), they were better able to perceive the individual factors which contributed to the quality of the product, which, in turn, attaches more significance to their acceptability of the product.

In other trials, panels selected at random from the faculty, graduate students and secretarial staff in the Department of Food Science, Michigan State University, were used to determine the acceptability of the freeze-dried reconstituted product. Based on the results of paired and triangular tests, these trials showed that the panels could

Table 17.--Sensory evaluation of instant freeze-dried cultured cream by university women.

Amount of Sour Cream Used/Month	Consumer Evaluation							
	Number of Consumers	Number Scored Acceptable	Number Scored Unacceptable	No Response	Number of Scored Preferences For			
					Flavor	Body	Texture	All Factors
0	5	3	0	2	3	1	1	1
1/2 Pint	28	27	1	0	20	18	20	20
1 Pint	40	38	1	0	33	24	29	19
1 Quart	16	16	0	0	15	14	14	12
2 Quarts or More	3	3	0	0	3	3	3	3
Total	92	88	2	2	71	59	66	47

not consistently differentiate between a fresh commercial sample and the freeze-dried reconstituted sample. These results, not reported, suggested one of two alternatives--either the panel members could not differentiate between the samples presented, or there was no significant difference between the samples.

In order to test these alternatives, a ranked-paired test was designed in which four samples were presented to each panel member along with a reference sample. The reference sample in each case was identical to one of the four samples--this constituted the pair. The panel members were then given a score sheet as shown in Appendix Fig. 4. By comparison with the reference sample, each sample was ranked according to preference. If two samples were ranked together, the total score was divided between them. Only the evaluations of those panel members who correctly identified the paired samples by indicating "No Difference" were included in the analysis for significance. The samples were examined by 20 panelists in each trial.

The four samples evaluated were: a fresh commercial cultured cream; a freeze-dried cultured cream reconstituted from powders of 1.75% moisture, stored for 4 months at 75F (22C); and two commercial spray-dried sour cream bases (Appendix Table 2).

The results of these evaluations (Table 18) show that of the 80 panelists tested, only 59 could correctly

Table 18.--Sensory evaluations comparing reconstituted freeze-dried cultured cream with commercial fresh and dehydrated sour cream samples by rankings.

Trial	Number of Panelists[1]	Total of the Ranked Scores				Totals Req'd. For Significance P/.01[2]
		Commercial Fresh	Freeze-Dried Reconstituted	Commercial Dehydrated		
				Sample 1	Sample 2	
1	11	13	24	35	38	15 - 29
2	14	21	24	46	49	25 - 45
3	18	24.5	30	63.5	62	32 - 58
4	16	24.5	23.5	56.5	55.5	28 - 52

[1]Number of (20) panelist who correctly identified the paired samples.

[2]Totals Represent: Lowest insignificant rank total, any treatment and Highest insignificant rank total, any treatment.

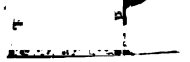
identify the paired sample. Analysis of the ranked totals of each trial indicated that the freeze-dried was significantly better than the spray-dried at the 1% level of significance. The results also showed that there was no significant difference between the freeze-dried reconstituted cultured cream tested and the fresh cultured creams purchased in the supermarket.

SUMMARY AND CONCLUSIONS

1. A desirable freeze-dried cultured cream was prepared which possessed excellent flavor and body properties upon reconstitution.
2. Retention of the typical body and texture properties of the fresh cultured cream in the freeze-dried reconstituted cultured cream required fortification of the fresh cream with stabilizers and stabilizing additives.
3. The addition of MSNF and milk proteins (sodium and calcium caseinate) demonstrated that an optimal level of addition existed, above which further addition resulted in a weaker bodied, destabilized reconstituted freeze-dried cultured cream.
4. The addition of stabilizer resulted in an increase in viscosity in the reconstituted product.
5. Pasteurization at 175F (80C) for thirty minutes was optimal because it permitted proper hydration of proteins producing the highest viscosity in the reconstituted product without adversely affecting the flavor.

6. Homogenization pressures in excess of 2000 psi completely destroyed the body properties of the reconstituted freeze-dried cultured cream.
7. Freeze drying at a platen temperature of 175F (80C) proved to be the most favorable drying condition with regard to both product quality and drying efficiency.
8. Maximum retention of diacetyl can be achieved at platen temperatures of 175F (80C) if the product is not exposed to these higher temperatures after being dried.
9. During the normal drying cycle there is a variation in absolute pressure within the drying chamber which increases with increased platen temperatures.
10. Freeze drying, which involves freezing and dehydration, completely destroys the milk emulsion resulting in a high level of free fat.
11. The incidence of free fat could not be significantly reduced by either higher homogenization pressures or the use of added emulsifiers.
12. The resulting flavor of the reconstituted freeze-dried cultured cream can be enhanced by increasing the level of the major flavor components in the fresh cultured cream prior to freeze drying.
13. The retention of diacetyl during the freeze drying process is dependent on the total solids of the fresh cultured cream.

14. Diacetyl will readily adsorb to the freeze-dried powder saturating the powder within fifteen minutes of exposure.
15. Freeze-dried cultured cream powders of less than 2% moisture were scored acceptable after storage for 6 months at a temperature of 72F (22C) and lower.
16. The loss of diacetyl during storage of low moisture powders at a temperature of 72F (22C) and lower was minimal for up to 6 months.
17. The viscosity or body properties of the fresh cultured cream was greatly reduced during freeze drying and the first two weeks of storage; during later storage there was no further appreciable change.
18. A moisture content of greater than 2% in the freeze-dried powder facilitates rapid development of Maillard browning in the stored powder even under regrigerated conditions.
19. The use of emulsifiers improved the dispersibility of the powder initially and following storage.
20. Consumer sensory evaluations show that there was no significant difference between the freeze-dried reconstituted cultured cream and the fresh cultured cream purchased in the supermarkets. The freeze-dried reconstituted and the fresh cultured creams were significantly better than spray dried preparations on the market.



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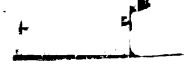

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APPENDIX

Table A-1.--Variation in penetrometer values (mm/10) with with time of penetration.

Time (Seconds)	Penetrometer Values (mm/10)			
	Fresh Cream (Direct Acidified)		Reconstituted Cultured Cream*	
0	245	222	279	298
5	248	262	316	303
10	279	267	317	305
15	280	287	321	330
20	294	292	326	325
25	295	293	322	328
30	290	292	323	325

*Reconstituted freeze-dried cultured cream.

Table A-2.--Comparison of analysis of commercial and freeze-dried cultured creams.

Cultured Cream	Titratable Acidity (%)	pH	Diacetyl Content (ppm)
Freeze Dried	0.90	4.8	1.85
Spray Dried I	1.30	5.0	1.45
Spray Dried II	1.10	4.8	0.28
Fresh	0.78	4.3	2.10

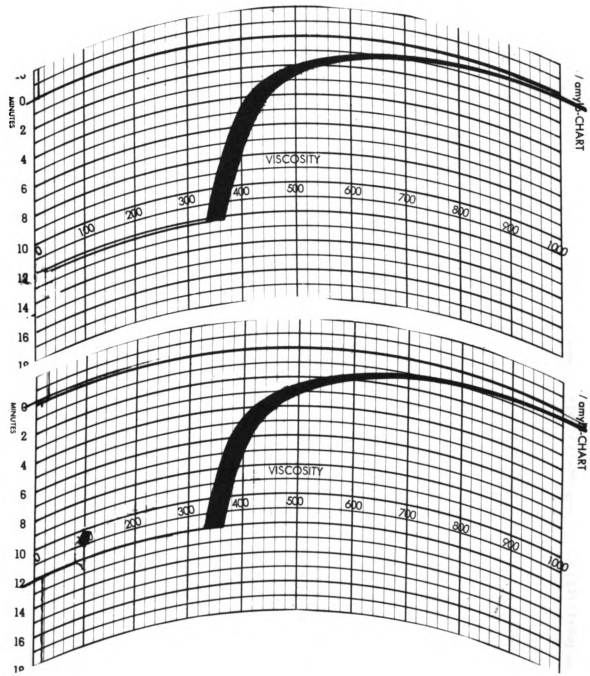

Fig. A-1.--Brabender/Visco Amylo/Graphs of duplicate samples of reconstituted freeze-dried sour cream.

Fig. A-2.--ORGANOLEPTIC EVALUATION OF FREEZE-DRIED RECONSTITUTED SOUR CREAM

NAME _____ SAMPLE # _____ DATE _____

	Diacetyl	Acid	Flavor Intensity	Freshness	Body	Texture	Over-all Evaluation
3	Very Pronounced	Very Pronounced	Very Pronounced	Very Fresh	Very Desirable	Very Smooth	Very Desirable
2							
1	Moderately Pronounced	Moderately Pronounced	Moderately Pronounced	Moderately Fresh	Moderately Desirable	Moderately Smooth	Moderately Desirable
0							
9	Slightly Pronounced	Slightly Pronounced	Slightly Pronounced	Slightly Fresh	Slightly Desirable	Slightly Smooth	Slightly Desirable
8							
7	Perceptible	Perceptible	Perceptible	Neutral	Neutral	Neutral	Neutral
6							
5	Moderately Perceptible	Moderately Perceptible	Moderately Perceptible	Slightly Stale	Slightly Undesirable	Slightly Grainy	Slightly Undesirable
4							
3	Slightly Perceptible	Slightly Perceptible	Slightly Perceptible	Moderately Stale	Moderately Undesirable	Moderately Grainy	Moderately Undesirable
2							
1	Imperceptible	Imperceptible	Imperceptible	Very Stale	Very Undesirable	Very Grainy	Very Undesirable

Number _____

g. A-3.--CONSUMER EVALUATION OF INSTANT FREEZE-DRIED SOUR CREAM

1) Do you use sour cream?

yes ☐

no ☐

If yes, please check where applicable.

2) What quantity of sour cream do you use per month?

1/2 pint ☐

1 pint ☐

1 quart ☐

2 quarts or more ☐

3) How do you normally use sour cream?

chip dips ☐

baked potatoes ☐

salad dressings ☐

baking ☐

cooking ☐

4) Is this product ...

acceptable ☐

unacceptable ☐

5) If product was acceptable, which factors did you like?

flavor ☐

body ☐

texture ☐

6) If product was unacceptable, which factors did you dislike?

flavor ☐

body ☐

texture ☐

NAME_____ TEST CODE_____

Fig. A-4.--Score sheet.

Flavor Difference Evaluation

Instructions:

1. Enter at the head of each column the code number of each sample in the test.

2. Determine by flavor comparisons with the REFERENCE SAMPLE the degree of flavor difference for each numbered sample.

   a. If you do not detect any flavor difference, place a check in the box opposite the words "No Difference".

   b. If in your judgement any flavor difference exists, place a check in one of the other five boxes opposite or between the terms which best describe the degree of flavor difference.

3. After rating the flavor difference place a check in one of the two boxes at the bottom of the column indicating, whether the flavor of the numbered sample is "Acceptable" or "Not Acceptable" to you.

N. B. Retaste reference sample as often as necessary to determine flavor differences.

Degree of Flavor Difference	Sample Number				
	____	____	____	____	____
Much Better	☐	☐	☐	☐	☐
Slightly Better	☐	☐	☐	☐	☐
No Difference	☐	☐	☐	☐	☐
Slightly Worse	☐	☐	☐	☐	☐
Much Worse	☐	☐	☐	☐	☐
Acceptable	☐	☐	☐	☐	☐
Non-acceptable	☐	☐	☐	☐	☐

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