NEUTRALIZED, HYDROLYZED, FLUID COTTAGE CHEESE WHEY IN FROZEN DAIRY DESSERTS

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ABSTRACT

The objective of this research was to utilize fluid cottage cheese whey as an ingredient in organoleptically acceptable frozen desserts. Acid whey was neutralized to either pH 6.5 or 6.8 with either KOH or $Ca(OH)_2$. Whey lactose was hydrolyzed, through the action of lactase enzyme, to either 50% or 75% of complete conversion to glucose and galactose. A 2^3 factorial design experiment was conducted to determine the most acceptable combinations of these three variables in fluid whey used as the bulk-supplying ingredient in ice cream.

Based on chemical, physical, and sensory considerations, KOH-neutralized ice creams were preferred to those prepared with $Ca(OH)_2$-neutralized whey. Analysis of sensory panel test data by theory of signal detection and analysis of variance showed a highly significant ($P < .001$) interaction between the three variables. These sensory data indicated that, with KOH neutralization, pH 6.8 was preferred regardless of degree of lactose hydrolysis; using $Ca(OH)_2$, 75% hydrolysis was preferred at pH 6.5.

Ice creams were free from sandy texture development for four months of storage. Problems with off-flavors, coarse texture, poor melt-down, and shrinkage were potentially correctable with minor changes in formulations and processing methods. The ice creams met all requirements for the 1960 standards of identity except for the use of the treated acid whey. This ingredient probably would have been acceptable

under the "safe and suitable ingredient" clause of the proposed 1977 standards revision, which is currently set aside by regulatory authorities.

INTRODUCTION

The dairy industry currently faces a problem of considerable magnitude regarding disposal and utilization of whey. Throughout history, this important cheese by-product has been used as livestock feed, as a social drink, or as a preventive medicine, but in more recent times, whey has been regarded more as a waste product and a nuisance. Strict environmental regulations, however, now prohibit disposal by dumping in many areas, and a great deal of research on utilization is currently under way.

Both sweet and acid whey contain about 93% water. Lactose, protein, minerals, and small amounts of fat comprise the solids. Cottage cheese whey presents a particularly difficult utilization problem because of its high acidity or low pH. Due to its perishable nature, cottage cheese is often produced in smaller to medium-size local plants. For this reason, acid whey is frequently not utilized because the relatively small volume makes processing uneconomical.

Cheese production in the United States has increased substantially in the past decade. This has resulted in a corresponding increase in whey output. Dried and modified whey is successfully used in a great many food and feed products today, and current total whey utilization is over 50%. There are very effective techniques available to demineralize, delactose, deproteinize, concentrate, and dry whey, but these methods are expensive in terms of required equipment and energy,

and only the large producers can usually justify the investment and processing expense. As a result, an all-too-common practice is to dump whey output as sewage.

It would appear, therefore, that further development of whey-based foods is justified, particularly if the new products can be manufactured by smaller operations. Further, it would be advantageous to utilize the whey at the site of its production, to eliminate transportation costs.

Surprisingly, there has been little research on utilization of fluid cottage cheese whey with only minor treatment or modification. Consequently, the objective of this study was to utilize fluid acid whey as a primary ingredient in frozen dessert formulations. The two obvious problems in using cottage cheese whey are its relatively high acidity and high lactose content. It was proposed to minimize these problems by alkali neutralization and enzymatic hydrolysis, respectively.

The current standards of identity for ice cream do not permit the high levels of whey used in this study. Under new standards proposed by the Food and Drug Administration, unlimited amounts of whey would be permitted as long as fat, protein, and solids levels were within the federal guidelines. During the course of this study, the proposed standards changes were withdrawn, in response to the understandable economic objections of milk producers. It is expected the issue will be pursued, however, and the results of this study will remain a viable alternative if agreeable standards modifications are made in the future.

It may be of interest to note that this study was carried out during the period from summer 1976 through spring 1978.

REVIEW OF LITERATURE

Whey Composition and Properties

Acid whey is a by-product of cottage cheese manufacture, in which lactose of fluid skim milk or reconstituted non-fat dry milk (NFDM) is fermented by a starter culture of lactic acid-producing bacteria. The high acidity that develops coagulates the casein, and the resulting whey is high in acidity and low in pH.

Table 1 lists the approximate composition and mineral content of fluid acid whey. The whey composition is contrasted with skim milk, for which whey could be considered as the ice cream ingredient substitute in this study.

The protein content of whey is about one-fourth that of skim milk. Casein is the major protein of milk, and exists as a micelle of calcium caseinate, phosphate, calcium, magnesium, and citrate (Gordon and Kalan, 1974, p. 88).

Removal of casein by coagulation with acid or rennet leaves the soluble whey proteins, the lactoalbumin and lactoglobulin fractions. At pH 6.3-6.5, the whey proteins are coagulated at 90 C (Gordon and Kalan, 1974, p. 112). Nutritionally, whey protein is of even higher quality than casein, having Protein Efficiency Ratios of 3.0-3.2 and 2.5, respectively (Schingoethe, 1976). The small amount of protein in fluid whey, however, makes it an inferior substitute for an equal volume of milk, from the standpoint of total protein content.

Table 1. Proximate composition and mineral content of fluid and acid whey and fluid skim milk.

Component	Fluid Acid Whey[1]	Fluid Skim Milk[2]
	%	
Moisture	93.5	92.0
Protein	0.9	3.5
Fat	0.1	0.08
Lactose	4.7	5.0
Ash	0.6	0.7
Total solids	6.5	8.56
Titratable acidity, as lactic acid	0.5	0.16
	mg/100 g	
Na	46	58
K	143	150
Ca	101	113
Mg	9.1	9.8
P	70	102
pH	4.6	6.6

[1]Compiled from Arbuckle and Singh (1971) and Feeley et al. (1972).

[2]Compiled from Shukla (1975) and Feeley et al. (1972).

Table 1 also shows that the water, fat, and ash contents of whey and skim milk are about the same. The major component of the solids of whey is lactose, while in skim milk, the same amount of lactose is present together with more protein.

Milk is a good source of calcium, phosphorus, and magnesium, but these minerals are slightly reduced in whey because of their association with the casein coagulum (Vaughan, 1970). The B vitamins, particularly riboflavin, are largely retained in whey.

Whey proteins are susceptible to heat-denaturation, but form a fine, soft, easily dispersible coagulum (Webb and Whittier, 1948). The proteins also have good whipping properties, and improve the body of sherbets (Webb, 1970b; Potter and Williams, 1949). Lactose accentuates flavors, absorbs pigments, and contributes mouthfeel in foods (Webb, 1970b; Nickerson, 1978).

Enzymatic Hydrolysis of Lactose

Sandiness Defect

Lactose is the major component of the solids in whey and milk. Avoidance of lactose crystallization in the sandiness defect of stored ice cream has traditionally been of primary concern in calculating mix composition. Use of significant amounts of whey solids to replace a portion of the milk solids-not-fat (MSNF) has presented a particular problem in avoiding sandiness because of the small amount of protein "dilution" of lactose in whey solids, as compared to dry milk solids.

The basic reason for lactose crystallization in ice cream is that there is insufficient water available under the prevailing low

temperature conditions to hold the poorly soluble lactose in solution (Doan, 1958). When the length of the crystals approaches 30 μm, the product feels gritty or mealy in the mouth (Shukla, 1975).

At the start of the freezing operation, ice cream mix is normally at refrigerator temperatures, 1.6-4.4 C (35-40 F) (Whittier, 1933). At this temperature range, the lactose concentration is usually below its saturation point (11% lactose). During freezing, however, as a portion of the water of the mix is frozen, the lactose becomes concentrated beyond the saturation point. At the extremely low temperatures of the hardening room (-18 C; 0 F), lactose becomes supersaturated in the unfrozen water and very susceptible to nucleation and crystal growth.

Other conditions favoring sandiness are heat shocking, high freezer drawing temperatures, dilute condition of the unfrozen water, and presence of crystal nuclei (Arbuckle, 1977, p. 326). In recent years, however, effective vegetable and marine gum stabilizers and efficient milk clarification methods have greatly reduced the danger of sandiness (Nickerson, 1962). Partial replacement of cane sugar with corn sugar also helps control this defect (Arbuckle, 1977, p. 326).

There has been interest in producing high-solids ice cream because of consumer demand for low-fat ice milks with heavy, chewy body and texture, and because of a desire to utilize surplus whey solids (Dahle, 1955). Thus, avoidance of sandiness is still of considerable concern in the manufacture of ice cream containing whey.

Lactase Technology

The advantages of producing low-lactose milk products for use in ice cream have been known for a long time, but suitable methodology has been available only recently.

There were a few early attempts at producing low-lactose products which were little more than laboratory curiosities. Tracy and Corbett (1939) produced a low-lactose/high-casein milk by separating the casein from skim milk with a carob bean stabilizer. Nickerson (1951) made ice cream with a spray-dried milk powder in which a portion of the lactose had been removed by crystallization.

The most satisfactory way to reduce lactose is to split the disaccharide into simpler sugars, and this can be accomplished by hydrolysis with acid or with the enzyme lactase (Nickerson, 1974a, p. 308). Lactase is the common name for β-D-galactosidase or β-D-galactoside galactohydrolase, with an enzyme number 3.2.1.23 (Shukla, 1975). Lactase breaks the β-D-galactosidic bond of lactose and in other substrates such as ortho-nitrophenyl-β-D-galactoside; both reactions may be used to assay lactase activity (Bouvy, 1974; Shukla, 1975). Bouvy (1974) illustrates these chemical structure changes.

Lactase occurs naturally in a variety of molds, yeasts, and bacteria, as well as almonds, soybeans, coffee, chickens, pigs, man, and many other sources (Pomeranz, 1964). The primary commercial lactases available today are from yeast (Saccharomyces lactis), mold (Aspergillus niger), and bacteria (Escherichia coli) (Shukla, 1975). Pomeranz (1964) reviewed the history of isolation and purification procedures for this enzyme. The lactase preparation should have high activity and low cost,

it should be active at typical substrate pH, and should not be contaminated with toxins, lipase, protease, zymase, or bacteria (Olling, 1972).

There are five changes in properties of milk that occur when lactose is hydrolyzed: 1) decrease in lactose concentration, with an increase in glucose and galactose; 2) increase in sweetness; 3) change in crystal arrangement, possibly affecting texture; 4) lowered freezing point; and 5) changes in viscosity and moisture retention (Nickerson, 1974b). In addition, condensation reactions between the sugars may occur, producing small amounts of oligosaccharides (Nickerson, 1974a, p. 309).

An early commercial yeast lactase was used by Potter and Webb (1951) to reduce lactose in condensed sweet whey (15% solids). The authors achieved 50% hydrolysis in three days at 40 F.

Sampey and Neubeck (1955) hydrolyzed 20-30% of the lactose in skim milk with commercial yeast lactase, and produced up to 14.5% MSNF ice cream which was resistant to sandiness. Albrecht and Gracy (1956) added this same commercial lactase directly to ice cream mix containing 17% MSNF, and after holding at 45 F for five days, the mix was pasteurized and frozen. They concluded that a two-hour incubation period at 112-121 F would have prevented the flavor problems associated with bacterial growth, and that hydrolysis was beneficial in preventing sandiness.

Jasewicz and Wasserman (1961) prepared crude lactase from thirty different microbial cultures, and assayed the lactase activity. Optimum enzyme activity depended on substrate lactose concentration, amount of lactase added, and time, temperature, and pH of incubation.

Wierzbicki and Kosikowski (1973d), in evaluating twenty-three different microorganisms for lactase-production potential, found that molds produced better cell yield than yeasts and bacteria, but that lactic acid bacteria had the highest lactase activity.

Wendorff, Amundson, and Olson (1970a) determined that Saccharomyces fragilis lactase production at 28 C was optimum at pH 4.0-4.7 using a Cheddar cheese whey medium. When this yeast lactase was used to hydrolyze skim milk concentrates and pizza cheese whey, forewarming the substrate at 85 C for 30 min enhanced the degree of hydrolysis (Wendorff, Amundson, and Olson, 1970b). Earlier work (Wendorff et al., 1971) had shown that enzyme suppressors in the nonlactose milk solids must be heat-inactivated for optimum hydrolysis.

Most of the lactase work reported so far in this review has been done with self-prepared enzymes. Borglum and Sternberg (1972) state that, on the basis of properties and ease of preparation, the lactases of Aspergillus and Saccharomyces would be most useful industrially. This prediction has been reflected in the fact that the two primary commercial lactases used today are: "Lactase LP" (Wallerstein Co.), from Aspergillus niger, and "Maxilact" (Enzyme Development Corp.), from Saccharomyces lactis. It should be noted that Lactase LP has been replaced by "Lactase N," and that both the yeast and fungal lactases are now marketed by GB Fermentation Industries, Des Plaines, Illinois (personal communication, GB Fermentation Industries). The primary difference between these two products is that Lactase LP is most active at pH 3.5-4.9, while the optimum pH for Maxilact is 6.5-7.0 (Technical

Bulletins, GB Fermentation Industries). The latter enzyme was used in this study.

The activity of Maxilact is irreversibly lost below pH 5 (Bouvy, 1974). Manganese is essential for active enzyme structure, and potassium and ammonium ions enhance its activity, while sodium and heavy metal ions are inhibitory. Activity is reduced if the substrate contains cheese starter organisms or is of poor microbiological quality. Galactose is a competitive inhibitor of lactase, and at high lactose concentrations (such as in condensed products), transgalactosidase activity increases.

Kosikowski and Wierzbicki (1973), in lactase treatment of raw and pasteurized milk, found that 4 C/48 hr incubation was preferable to 30-37 C/5 hr from the standpoint of bacterial growth and flavor deterioration, and because of the usual industrial practice of holding cold milk in tanks for some time before pasteurization and packaging. A lactase concentration, in pasteurized milk, of either 25 or 100 mg/l, resulted in 80% and 95% lactose conversion, respectively. Because degree of hydrolysis in raw milk was only slightly reduced, the authors concluded that pasteurization before lactase treatment was unnecessary.

The Agricultural Research Service of the U.S. Department of Agriculture has tested a variety of Maxilact-treated products on a pilot-plant scale (Holsinger and Guy, 1974; Guy et al., 1974; Holsinger and Roberts, 1976). Guy and co-workers (1974) found that fluid milk was acceptable to adults even with 90% lactose conversion, although the panel preferred the less sweet products at lower hydrolysis levels. In a panel comparison with sucrose solutions, it was found that 30% lactose hydrolysis equals 0.3% sucrose, 60% conversion approximates 0.6%

sucrose, and 90%, about 0.9%. Other USDA research examined Maxilact-treated yogurt, buttermilk, and cottage and Cheddar cheese (Holsinger and Guy, 1974).

Engel (1973) and O'Leary and Woychik (1976) found that yogurt manufacture was well-suited to lactase use because the starter organisms utilize glucose faster than lactose. Other advantages were reduction in amount of sweetener required, automatic inactivation of the enzyme with sugar fermentation to lactic acid, and different flavor profiles.

Cheddar cheese made with lactase-hydrolyzed milk aged faster and had better flavor, body, and texture than the control (Thompson and Brower, 1976). The hydrolyzed whey by-product could be dried or used in other products with no further treatment. Use of lactose-reduced milk resulted in increased yield of cottage cheese because of minimized curd shattering (Gyuricsek and Thompson, 1976).

Lactase-treated dairy ingredients in candy would enhance desirable browning reactions and allow reduction in sucrose levels (Holsinger, 1976).

Fungal lactase was used to convert acid whey into a clear, golden, sweet food syrup (Wierzbicki and Kosikowski, 1973a). An ice cream sweetener containing galactose, glucose, fructose, lactose, and 10% solids was prepared from acid whey (Weetall et al., 1974). In this system, immobilized lactase hydrolyzed the lactose, and then the glucose was partially isomerized to fructose with immobilized glucose isomerase.

In surveying the microbial lactases available commercially, Woychik and Holsinger (1976) concluded that those from Aspergillus niger and Saccharomyces lactis are most suitable for food use. Further,

A. niger lactase is best used in immobilized form for acid whey, while S. lactis works best for milk in soluble form. The current high enzyme cost makes lactase treatment commercially feasible only when immobilization on insoluble carriers allows repeated use (Pitcher, 1975).

Shukla (1975) has reviewed the major immobilization procedures for lactase, including polyacrylamide gel entrapment, collagen and cellulose attachment, encapsulation, and binding to porous glass. The latter method, although subject to high enzyme denaturation rates, is preferred because of its stability, versatility, and nondegrability by microbial contamination.

Lactase catalyzes transgalactosidation, or the non-hydrolytic transfer of the galactose moiety of lactose to other sugars or alcohols (Shukla, 1975). The oligosaccharides produced pose a potential problem because of their indigestibility to humans (Shukla, 1975). Wierzbicki and Kosikowski (1973b) found five oligosaccharides in acid whey treated with A. niger lactase, while the number identified in milk treated with lactase from S. fragilis is eleven. The number and type of oligosaccharides formed is determined by substrate lactose concentration, source of enzyme, temperature, pH, and presence of inorganic ions.

Potential oligosaccharide problems could be prevented by the use of multi-enzyme systems, which would convert the oligosaccharides to monosaccharides (Wierzbicki, Edwards, and Kosikowski, 1974).

Determination of Degree of Hydrolysis

A considerable range of techniques was found in the literature to determine the extent of lactose hydrolysis. There is no difficulty

in measuring the amount of lactose before hydrolysis because lactose is the only significant carbohydrate present. Glucose, galactose, and lactose all contain a free aldehyde group, and undergo the same types of reduction reactions. Thus, in the presence of all three sugars during the course of lactose hydrolysis, there is interference because the reducing reagent reacts with all three sugars.

In measuring the extent of hydrolysis, many researchers assume that one mole of lactose yields one mole each of glucose and galactose. In this way, lactose can be measured before hydrolysis and either glucose or galactose after hydrolysis, using an enzymatic procedure which recognizes only one of the sugars. Degree of hydrolysis is calculated as the appearance of glucose from a measured amount of lactose (Weetall et al., 1974). However, because of the formation of small amounts of oligosaccharides, a reaction involving only galactose, there will always be slightly more free glucose than galactose (Bouvy, 1974). This difference from the theoretical amount of glucose is assumed to be insignificant.

An enzymatic procedure specific for glucose is the Glucostat Reagent Set (Worthington, Diagnostics, Freehold, New Jersey), which has now been replaced by the Worthington Statzyme Glucose Kit. Both methods utilize glucose oxidase to oxidize the glucose in a deproteinized sample (Package Insert, Worthington Diagnostics). The hydrogen peroxide produced by the reaction is enzymatically coupled with a chromogen to produce a colored product which can be colorimetrically analyzed.

Table 2 lists some suggested methods, most of them colorimetric, for determining lactose and its hydrolytic products. Brobst and Lott

Table 2. Review of methods for determining degree of lactose hydrolysis in milk or whey.

Authors	Lactose Method	Glucose Method
Potter and Webb (1951)	Calculated from glucose/galactose	Potter (1950)
Wendorff, Amundson, and Olson (1970a)	Marier and Boulet (1959)	Tauber and Kleiner (1932)
Kosikowski and Wierzbicki (1973)	Randerath (1963)	Randerath (1963)
Wierzbicki and Kosikowski (1973c)	Nelson (1944), modified, following optimum lactose conversion	Nelson (1944), modified
Wendorff et al. (1971)	Lawrence (1968)	Glucostat Reagent
Borglum and Sternberg (1972)	Brobst and Lott (1966)	Barton (1966), auto-analyzer
Giacin et al. (1974)	Dubois et al. (1956)	Glucostat Reagent
Guy et al. (1974)	Folin and Wu (1919)	Tauber and Kleiner (1932)
O'Leary and Woychik (1976)	Jasewicz and Wasserman (1961), following conversion of lactose by excess lactase	Jasewicz and Wasserman (1961), modified galactose by galactose-dehydrogenase (O'Leary and Woychik, 1976)
Current study	Teles et al. (1978)	Glucostat Reagent

(1966) detail a gas-liquid chromatographic method, and Randerath (1963) describes thin-layer chromatography. Recently, Nickerson, Vujicic, and Lin (1976) published a colorimetric method for following lactose hydrolysis which was a modification and improvement of two earlier methods.

In the current study, lactose was determined by the colorimetric method of Teles et al. (1978), and glucose by the Glucostat Reagent.

Use of Whey in Ice Cream

About 40% of the dry sweet whey and 25% of the modified whey products used for human food in 1975 were used by the dairy industry (Clark, 1976). The federal standards of identity for ice cream allow sweet whey replacement of up to 25% of the nonfat dry milk (Singleton, 1972). Acid whey is not recognized as an optional ingredient in the current (1960) standards, although some states allow it (Arbuckle and Singh, 1971). Under the proposed revisions of the federal guidelines, acid whey in unspecified amounts could be used under the "safe and suitable ingredient" clause (Keeney, 1976).

Leighton (1944) suggested that, because of its high lactose and low protein content, whey solids would be more acceptably utilized as additional, rather than as substitute, solids in wartime ice cream. To avoid sandiness, he suggested a maximum 2.8% whey solids substitution or 2.3% supplementation to the composition of a 10% fat mix. In sherbet, there would be no need to limit whey addition because of the lower solids of this product.

Reid and Shaffer (1947) concluded that vanilla ice cream containing as much as 10% dry sweet whey could be stored satisfactorily for 50 days in the hardening room.

Potter and Williams (1949, 1950) found that excellent sherbets could be made with fluid, condensed, dried, or sweetened condensed sweet and acid wheys. Sherbets containing 4-5% whey solids were equal or superior to conventional sherbets in body, texture, flavor, and overall impression. An advantage of whey sherbets is that whey proteins do not curdle with citric acid addition and, furthermore, acidification to impart a tangy flavor was not necessary in acid-whey containing sherbets. The masking effect of casein on fruit flavors is avoided in whey sherbets (Anonymous, 1955).

Use of spray-dried whey in ice cream lowered costs, and improved whipping, body, texture, melt resistance, and flavor (Rosenberger and Nielsen, 1955). Whey-containing ice creams have a firm, dry appearance at the freezer.

Nielsen (1957) discussed the value of using high-grade dry whey and whey-milk blends as sources of serum solids in frozen desserts. Crowe (1960) recommended that, to avoid sandiness, no more than 50% of the ice cream mix solids should come from dry sweet whey. In contrast, as much as 94.5% of the serum solids in sherbet could be replaced by dry cottage cheese whey with no detriment to flavor, body, and texture (Blakely and Stine, 1964).

The natural acidity of cottage cheese whey was used advantageously in nutritional popsicles by Guy, Vettel, and Pallansch (1966) and by Stull et al. (1977). In the latter study, fresh, fluid cottage

cheese whey was used. In the former study, the authors speculated that the whey popsicles were superior to conventional popsicles because, based on previous work, the calcium and phosphate content of whey could help reduce tooth erosion.

The biggest drawback in using whey in ice cream at permissible levels is the possibility of imparting off-flavors from poor-quality whey (Frazeur, 1965). Flavor of whey-containing frozen desserts could be improved if the whey was first electrodialyzed to reduce the mineral content (Frazeur, 1967). In large-scale consumer tests, several quality grades of whey were tested in several types of frozen desserts (Frazeur and Harrington, 1967). This research showed that excellent-flavor dry whey could be used in sherbet, ice milk, soft-serve ice milk, and milk-shake mix, but electrodialyzed whey should be used in ice cream. Average-flavor whey should not be used except in sherbet.

Tobias (1970) discussed the advantages and disadvantages of using dry whey as a replacement for NFDM in frozen desserts. Although the protein content is low in whey solids, the whey proteins have good water-binding and whipping properties. The higher ash content may impart salty flavors, and the calcium and phosphorus content is lower than in dry milk. High lactose content poses the threat of sandiness, but also accentuates flavors. Whey solids lower the freezing point of the mix and produce a dry ice cream with improved body, texture, and melt-down.

Arbuckle and Singh (1971) give formulations for sherbets, ice- and fudge-type stick novelties, and quiescently frozen dairy confections made with liquid, condensed, or dried acid whey. To avoid curdling, the

dairy ingredients were pasteurized separately from the whey and non-dairy ingredients. Good flavor was maintained when bicarbonate and phosphate neutralizers and buffers were added to the formulation.

As a result of a study by Igoe et al. (1973), frozen dessert standards in Pennsylvania were amended to allow the use of cottage cheese whey in ice cream. Concentrated acid wheys (29% solids), either unneutralized or adjusted to pH 6.5 with NaOH or KOH, were used to replace up to 27% of the MSNF. Based on taste panel data, the authors recommended a maximum substitution for MSNF of 9% unneutralized or 18% neutralized acid whey solids. The neutralized whey ice creams were very similar to those made with sweet whey. The low protein-to-lactose ratio of whey solids theoretically reduced the protein content and afforded less resistance to sandiness and heat shock, but improved stabilizers, emulsifiers, and use of low DE corn sweeteners minimize these problems in practice.

Loewenstein (1975) compared the composition of whey protein concentrates, lactose-hydrolyzed concentrates, and partially demineralized and delactosed whey products. Variations in ice cream composition and properties resulted when these modified wheys were used as solids suppliers. When hydrolyzed (50%) concentrated cheese whey was used to supply essentially all of the MSNF, the ice cream properties were acceptable, but the protein content was too low (1.82%) to be legal under the proposed standards of identity.

Guy et al. (1974) replaced 25% of the MSNF of ice cream with either fluid Cheddar cheese or KOH-neutralized cottage cheese whey, following 83% hydrolysis of lactose. Taste panel scores after eleven

weeks were similar to those for ice cream made with hydrolyzed skim milk, and the ice creams were not sandy. Ice cream containing acid whey had a longer, less uniform melt-down than the sweet whey product, however.

A taste panel at the University of Vermont preferred whey-containing ice creams to a commercially available control ice cream (Nilson, 1975). There were only minor differences in scores for ice creams containing 25-75% replacement of dry milk with either dry sweet whey or demineralized sweet whey.

Loewenstein et al. (1975) compared the properties of ice creams containing one of three types of neutralized acid whey concentrates: 1) ultrafiltered to 11.5% solids, 2) low-temperature concentration to 30-35% solids, or 3) 50% lactose hydrolysis prior to low-temperature concentration. Following neutralization with KOH, whey was decanted or centrifuged to remove precipitated protein. It was found that any of the concentrates could be used to replace 20% of the dry milk, but at 50 or 100% substitution levels, flavor was undesirable. The best overall ice cream was made with the hydrolyzed concentrate.

Arnold, Evans, and Kreshel (1976) found that dried sweet whey was a better MSNF replacement ingredient than was partially delactosed whey because, with removal of two-thirds of the lactose, the mineral content of the resulting whey was unpalatable.

In anticipation of the proposed changes in ice cream standards, numerous whey blends have appeared on the market. These products may contain combinations of whey, nonfat dry milk, whey and milk protein

concentrates, caseinates, and other ingredients, and are designed to be used as NFSM replacers (Anonymous, 1976).

Federal Standards of Identity for Ice Cream

The Federal Food, Drug and Cosmetic Act of 1938 is the fundamental law regulating foods in the U.S. today and arose out of an apparent need to protect consumers against fraud and hazardous products in interstate commerce. The Food and Drug Administration (FDA) was given the responsibility of establishing "standards of identity" for manufactured foods to insure that the public was offered unadulterated products of honest value.

The current standards of identity for ice cream were published in 1960 (FDA, 1960). The standards specify that ice cream can contain not less than 10% milk fat, 20% total milk solids, and 6% MSNF by weight of the finished product. The finished ice cream must contain at least 1.6 lb/gal total solids and weigh at least 4.5 lb/gal. Artificial or natural coloring may be added with no label declaration. Only specified optional ingredients are allowed, including concentrated or dried cheese whey with a titratable acidity of less than 0.16 or 0.18%, respectively. The sweet whey may be added at a level of not more than 25% of the total nonfat milk solids, by weight of the finished product. Caseinates are permissible when added to a mix containing at least 20% total milk solids. The stabilizers, emulsifiers, and other permissible salts are specified, as is the maximum quantity of each class of additives.

Since 1960, there has been a trend in the Food and Drug Administration to simplify the regulatory machinery, in order to lessen the

economic burden of compliance by industry (and ultimately the consumer) (Gutterman, 1977). The amended regulations would ideally protect the consumer from fraud and unsafe food, while allowing the food manufacturer the latitude to make formulation changes and improvements without the need for prior regulatory permission. Concurrent with this thinking, there has been a growing awareness about food quality, nutrition and health in this decade as a result of White House Conferences on the subject. This has tended to increase consumer interest in nutritional labeling.

In October 1973, the International Association of Ice Cream Manufacturers (IAICM) petitioned the FDA to provide for nutritional labeling in the standards of identity for ice cream. The proposal by IAICM was in anticipation of probable FDA proceedings of the same nature (IAICM, 1974). The ice cream manufacturers felt from past experience that they would have a better chance of getting final regulations closer to established industry practice if they initiated the proposal, rather than to wait for FDA to propose the inevitable changes in regulations. The basic request of IAICM was that generic groupings of dairy ingredients, egg ingredients, and sweetening agents be established so that interchange of ingredients within a class could be made without label changes. In addition, the manufacturers wanted to be able to list dairy ingredients as "milk products" and to use the generic names "stabilizers" and "emulsifiers" instead of the common or usual names of these custom-made additives.

Accordingly, on July 25, 1974, the FDA published a proposal to revise ice cream standards of identity, based on the IAICM petition and

upon their own initiative (FDA, 1974). The important changes included: 1) full ingredient listing on the label, 2) use of "any safe and suitable ingredients," and 3) replacement of the old unenforceable (because of the lack of reliable laboratory tests to distinguish between milk and non-milk solids) minimum level of milk solids-not-fat with minimum protein. As a result of the "safe and suitable ingredient" provision, all forms of milk-derived materials could be incorporated in any amount, including whey and caseinates.

The 1974 proposal generated a great deal of discussion and speculation about new opportunities and the potential for abuse. On April 12, 1977, the final action was published for Part 135 (formerly Part 20) of the Federal Food, Drug and Cosmetic Act, subject to filing of objections and petitions for hearings (FDA, 1977). The regulations provided for: 1) use of safe and suitable ingredients including milkfat, nonfat milk solids, and milk-derived ingredients; 2) minimum of 1.6 lb/gal total solids; 3) minimum 4.5 lb/gal weight of finished ice cream; 4) minimum 10% milkfat; 5) not less than 2.7% protein having a PER not less than whole milk protein; 6) label declaration, in decreasing order of prevalence, the sources of milkfat or milk solids-not-fat; and 7) declaration of all optional ingredients.

However, the National Milk Producers Federation filed objections and on July 8, 1977, the FDA stayed portions of the standards which were objectionable to allow study of the need for public hearings (FDA, 1978; Anonymous, 1978). A sixty-day period was designated for interested parties to submit new data regarding the impact of the new standard on physical and nutritional characteristics of ice cream (FDA, 1978).

Congressional leaders rushed to the support of the milk producers, and the media provided broad coverage of the issue (Anonymous, 1978).

As a result of further study, the FDA concluded in December 1977 that "under the new standard some ice cream formulations could have lesser amounts of some nutrients than under the current standard" (FDA, 1978, p. 4597). Accordingly, on February 3, 1978, FDA revoked the proposals regarding safe and suitable ingredients and minimum protein requirement, reinstated the whey and caseinate limitation, and finalized the requirement for nutritional labeling as of July 1, 1979. The Commissioner announced his intention to hold hearings on food labeling and to further examine the concept of safe and suitable ingredients versus the strict recipe approach in designing food regulations.

The basic issue in the standards controversy has been one of economics. The substitution of more whey and caseinates for nonfat dry milk would provide potential ingredient cost relief to ice cream manufacturers faced with skyrocketing dry milk prices. The milk producers were understandably reluctant to allow any possible infringement on their dry milk markets. Superimposed on this issue is the influence of the milk price support system, which complicates the responsiveness in the law of supply and demand (Saal, 1978). Another often-overlooked point is that the use of whey and caseinates has both quantitative and qualitative self-limiting aspects because of undesirable effects on flavor, body, texture, and storage characteristics (Hutton, 1977).

The ice creams developed in this investigation could be considered legal in a broad interpretation of the "safe and suitable ingredients" clause of the proposed new regulations. The revocation of the

whey ingredient portion of the standards places the immediate commercial applicability of these products in the potential feasibility status. It is expected that changes in ice cream standards will remain a viable activity on the part of the several groups involved. Indeed, the IAICM has filed objections to FDA's decision and requested hearings on the standards revocation (IAICM, 1978). In a second petition, the organization has requested approval for the use of acid and modified whey in ice cream, within the 25% MSNF minimum. The grounds supporting this second petition are that, according to the data filed by FDA in support of the standards revocation, limited use of acid and modified whey would not adversely affect nutrient content of ice cream. It should be noted that ice cream standards in Pennsylvania presently permit the use of either neutralized or unneutralized acid whey at levels of 25% of the MSNF (Igoe et al., 1973).

It is hoped that the results of this study will provide technical and scientific information of value in future standards considerations.

Standardization of Whey Acidity

Neutralization of ice cream mixes was a common practice in the days of inadequate refrigeration, when cream acidity was likely to be higher than desirable. There was a temptation to use neutralizers to mask old or inferior ingredients, however, and as a result, neutralization has been regarded suspiciously by regulatory bodies.

Today's use of cottage cheese whey in foods has created a new use for acid standardization, because unneutralized whey has a

disagreeable "whey taint" flavor. This flavor is due to the lactic acid built up during fermentation in cheesemaking.

The word "neutralization" is a misnomer because the process involves reducing the titratable acidity from about 0.5% to a final value of 0.1-0.3% (Dahle, 1926). Although "acid standardization" is a better term, both terms will be used synonymously here.

Fresh whole milk has a normal acidity of 0.14-0.16%, due to dissolved salts, casein, and carbon dioxide. This "apparent acidity" is proportional to the amount of serum solids or MSNF (Josephson, 1947). The acidity of ice cream mix components also contributes to final mix acidity (Dahle, 1926).

"Developed acidity," on the other hand, is due to bacterial fermentation of lactose to lactic acid, and this abnormally high acidity changes mix properties and ice cream quality (Josephson, 1947). High-acid mixes are usually excessively viscous and difficult to whip, and casein coagulation can cause curdling or feathering during homogenization or pasteurization (Dahle, 1926; Josephson, 1947). High-acid ice creams frequently have flavor defects and curdy, wheyed-off melt-downs.

The purpose of neutralizing high-acid mixes, therefore, includes prevention of protein coagulation, efficient homogenization, and improved flavor, body, whipping quality, and melt-down of the finished ice cream (Turnbow, Tracy, and Raffetto, 1947, p. 145). Care must be taken to add only enough alkali to neutralize the developed acidity, and to avoid offsetting the apparent acidity, because overneutralization imparts a bitter or soapy neutralizer flavor to the ice cream (Dahle, 1926; Josephson, 1947).

Dahle and Rivers (1940) pointed out that proper use of neutralizers can control protein destabilization and fat clumping in high-acid mixes. Neutralized mixes are sometimes more susceptible to ice cream shrinkage than are non-neutralized mixes (Frandsen and Nelson, 1950, p. 108).

Drawbridge (1951, pp. 3-11) and Arbuckle (1977, pp. 50-52) have reviewed the use of acid standardizers and other mineral salts in the preparation of ice cream mix. Alkalies, alkaline earths, or carbonates of minerals are capable of reacting with lactic acid to produce salts, and so are suitable neutralizers (Hunziker, 1940, p. 249).

Lime neutralizers (calcium and magnesium compounds) are inexpensive, but relatively insoluble, and thus act slowly in reducing acidity (Hunziker, 1940, p. 249). Because of calcium's affinity for casein, a part of the neutralizing capacity of lime neutralizers is tied up with casein, and is unavailable for reaction with acid (Hunziker, 1940, p. 251). Consequently, more than the calculated amount of lime neutralizer must be used to standardize the acidity, and accurate acidity determinations can be performed only after neutralization has been followed by pasteurization and cooling (Hunziker, 1940, pp. 251-252).

Soda neutralizers (sodium compounds) are more expensive, but completely soluble, and they act immediately on the acid of the mix (Hunziker, 1940, pp. 250-252). They tend to soften casein, and in some cases cause foaming of the mix. They have only about half of the neutralizing strength of the lime compounds.

If used too extensively, both types of acid standardizers can cause flavor problems. In the case of calcium neutralizers, a coarse,

limey flavor and texture results from overneutralization, whereas sodium compounds produce soapy flavors (Hunziker, 1940, pp. 252-253). Flavor problems can sometimes be avoided by double neutralization, in which a portion of the acidity is reduced first with lime, followed by further reduction with sodium compounds.

Turnbow et al. (1947, pp. 145-151) and Hunziker (1940, pp. 253-257) give details on calculating the amount of neutralizer required. The authors recommended mixing the calculated amount of neutralizer with water before adding to the mix.

The literature regarding the effects of acid standardization on mix and ice cream properties has been reviewed by Turnbow et al. (1947, pp. 150-151), Drawbridge (1951, pp. 5-11), and Arbuckle (1977, pp. 50-52). A summary of the information from these three sources follows.

High-acid mixes are often abnormally viscous and more susceptible to fat clumping than low-acid mixes. Smaller ice crystals are formed when the pH is near neutral and acidity around 0.12%. Acidity does not, however, play a role in the crystalline structure of ice cream. There is no consistent relationship between pH, titratable acidity, and lactic acid content of the mix.

Neutralization of high-acid mixes improves flavor, body, texture, and stability of the ice cream, but variable results are reported on the effectiveness of specific neutralizers to correct these problems. Each class of acid standardizers has its own strengths and weaknesses.

Lime neutralizers sometimes contain objectionable insoluble matter. Calcium and magnesium salts interact with casein in the mix, and consequently, a portion of their neutralizing capacity is lost. They

decompose slowly, and if adequate time is not allowed before acidity testing, overneutralization can be the result.

Soda neutralizers have strong wetting effects on casein, which prevents formation of a protective film around the fat globules, and fat clumping can result. Sodium neutralizers promote a smooth, rapid melt-down.

Calcium and magnesium salts are preferred because of their less detrimental effect on flavor and body, but sodium salts promote smaller ice crystals. Overneutralization is to be avoided with both neutralizers because the result can be flat, bitter, or soapy flavors and dull, gray color in the finished ice cream.

Varying results were reported regarding the effects of mix whipping ability. Citrates, phosphates, oxalates, lactates, and other sodium and calcium salts reportedly stabilize protein, and may thus influence viscosity, whipping ability, and fat clumping. In addition, mix properties may be changed by the interaction of the neutralizers with stabilizers and emulsifiers. Some salts used in ice cream today are added for mix improvement, rather than for neutralization.

Sensory Evaluation

General Considerations

Preference testing is one of the most important steps in product development, and serves as a test of the concept as well as a prediction of marketing success (Ellis, 1969). An expert taster relies on memory to subjectively judge samples, according to a set of standards derived from his experience with the product. Product evaluation by a panel, on

the other hand, may be a better system because several individuals make judgments based on clearly defined rating criteria. However, preference tests are limited to a simple evaluation of how well a panel likes a product, and give no information regarding the panel's feelings about specific characteristics of the product, e.g., flavor, color, odor, and texture. Therefore, the method of sensory testing must be chosen carefully to ensure that it can supply the information which is desired.

There are a variety of sensory test methods which are appropriate to new product development, as summarized by Prell (1976). These include single and paired comparisons, scalar scoring, hedonic rating, flavor and texture profiles, food action scale, magnitude estimation, and quantitative descriptive analysis. A survey of 62 major U.S. food companies revealed that the three most often-used sensory testing methods were triangle (66%), hedonic scale scoring (57%), and paired comparison (55%) (Brandt and Arnold, 1977). The triangle test is a difference test, and gives information on panel sensitivity, while the paired comparison may be used as either a difference or a preference test (Brandt and Arnold, 1977; Ellis, 1969). Hedonic scoring is the most widely used preference test, and is applicable to large-scale consumer testing as well (Brandt and Arnold, 1977). The paired comparison preference test is also appropriate in consumer acceptance tests (Prell, 1976).

Preference panelists should not be experienced judges and need only be representative of the target population (Ellis, 1969). The number of panelists should be 16-30 or more, and care should be taken to avoid selection bias [American Society for Testing and Materials (ASTM), 1968, p. 6).

Larmond (1973) gives details on control of the physical test area. Environmental distractions must be kept to a minimum for unbiased judgments by panelists. Physical requirements include the actual booth setup, lighting, sample preparation, sample temperature, size of sample, and order of presentation. The number of samples presented in hedonic tests should be no more than 18 (Prell, 1976).

Questionnaire design must be brief, simple, and unambiguous (ASTM, 1968, p. 20). A rating scale should give the impression of a continuum of degrees of magnitude and should contain at least five categories.

Statistical design should be arranged to utilize each piece of data more than once (Ellis, 1969). A factorial technique allows use of many variables at several levels. Conclusions thus are more generally applicable because each result leads to more than one conclusion.

A survey of major food companies revealed that data analysis was performed both manually and by computer (Brandt and Arnold, 1977). The most-often used statistical tests were Student's t test (55%), analysis of variance (47.5%), and Chi-square (40%).

Theory of Signal Detection

Theory of signal detection (TSD) provides a method for separating an observer's sensory judgment criteria from his ability to perceive differences in stimuli (Angus and Daniel, 1974). In the current work, the primary objective was to determine preference for ice creams. Application of TSD analysis permitted examination of interactions of pH, degree of hydrolysis, and neutralizer in determining preference. The

individual decision criteria for ice cream preference are established as a result of an observer's previous experiences with ice cream (Swartz, 1973). If, for example, an observer consistently rates all samples as being highly desirable, he may have low judgment criteria for preference (Stull et al., 1974). TSD offers a way to distinguish this observer's judgment standards from his actual perceptual decisions. The method also offers a means to compare this judge's decisions with another observer whose judgment standards are unusually high, in which he uses only the bottom end of the rating scale. In contrast, an analysis of variance of mean ratings would not make a distinction between a panelist's observations and his judgment criteria (Angus and Daniel, 1974).

This technique has been very useful in quantifying esthetic preferences for scenic beauty of National Forest landscapes (Daniel and Boster, 1976). TSD has also been applied to food product development, in discriminating and defining rich flavor in ice cream, where no fundamental definition of richness exists (Swartz, 1973; Angus and Daniel, 1974; Stull et al., 1974). In these studies, ice creams containing three levels each of fat, overrun, and vanilla flavor were presented to the panel. Stull and co-workers (1977) applied TSD to test the acceptability of a whey-based, fruit-flavored frozen novelty. In this experiment, preferences were established for pH and sweetener level in whey-based novelties, as well as for the basic preference of whey versus water as the major ingredient. The panelists were six to fourteen years of age.

Swartz (1973, p. 51), using TSD to examine rich flavor in ice cream, found that panelists could discriminate differences in fat levels of two percent. Subject-to-subject variance and run-to-run variance were not significant, indicating that the panel could reproducibly discriminate among variables on the twenty-seven ice creams presented each day (Swartz, 1973, p. 42). Significant interactions between fat-overrun and flavor-overrun on TSD analysis were also found to be insignificant when analyzed by more traditional statistical tests (Swartz, 1973, pp. 42-43). Further, rich-tasting ice cream is a function not only of fat content, but is perceived by the interaction of fat, percent overrun, and amount of vanilla flavoring added (Swartz, 1973, p. 55). The TSD computer program is also presented in Swartz (1973, pp. 72-80).

OBJECTIVES OF THIS STUDY

The overall objective was to develop an acceptable ice cream-type frozen dairy dessert containing fluid cottage cheese whey as the primary ingredient supplying bulk.

The specific objectives of this investigation were:

1. To compare the effect of potassium hydroxide (KOH) and calcium hydroxide [$Ca(OH)_2$] as lactic acid standardizers on the properties of the ice creams.
2. To compare the effects of properties of two whey pH levels, 6.5 and 6.8.
3. To compare the effects on properties of two levels of whey lactose hydrolysis, 50% and 75%.
4. To compare the acceptability of the experimental ice creams with a control ice cream of standard formulation using an untrained observer panel.

MATERIALS AND METHODS

This investigation was conducted in The University of Arizona pilot food science processing plant. Throughout all phases of this study, an attempt was made to utilize equipment which would be normally available in commercial plants. The methods used here could be easily adapted for feasibility and practicality under commercial plant conditions.

Whey Treatment

Choice of Variables

Each of the eight experimental ice creams contained fluid cottage cheese whey which had been treated in three ways: 1) lactic acid neutralization with either KOH or $Ca(OH)_2$, 2) adjustment of pH to 6.5 or 6.8, and 3) hydrolysis of lactose to either 50% or 75% of completion. A ninth ice cream, the control, was a standard formulation containing whole milk instead of whey.

Acid Standardizers. A review of the literature and pre-testing had indicated that there were advantages and disadvantages inherent with both lime and soda-type neutralizers, and that there was no "best" neutralizer to use. The commercial acid standardizers used by Drawbridge (1951, p. 14), which were double salts and buffered salts intended for use in ice cream, were no longer available.

Flavor, chemical, and physical acceptability of NaOH, KOH, $Mg(OH)_2$, $Ca(OH)_2$, and $NaHCO_3$ were examined in small batch pre-testing, as well as double neutralization with $Ca(OH)_2$ and KOH. Because of its inhibitory effect on lactase enzyme, NaOH was not acceptable. Based on efficiency of use and taste tests, it was decided that KOH and $Ca(OH)_2$ gave the best results.

The $Ca(OH)_2$ used was analytical grade reagent (Baker), 97.0% pure, with 0.024% insoluble matter, while the analytical grade KOH (Baker) was 86.0% pure and contained 0.002% insoluble matter.

KOH was added as a 30% aqueous solution. Because $Ca(OH)_2$ is only slightly soluble in water, this neutralizer was used as a thoroughly mixed, 30% slurry.

Whey pH. In pre-testing, neutralized whey pH levels between 6.4 and 7.0 were considered. With both acid standardizers, at pH 6.4 the whey still had a slightly sour, "whey taint" flavor, which tended to a salty, neutralizer flavor at pH 7.0. In view of these results, and the fact that normal milk pH is 6.5-6.7 (Johnson, 1974, p. 4), a decision was made to use whey pH levels of 6.5 and 6.8.

Extent of Lactose Hydrolysis. Because of the predominance of lactose in the solids of whey, the danger of sandiness in the whey ice creams was considered. β-D-galactosidase (lactase) is commercially available in highly purified form, and is very useful in reducing the danger of lactose crystallization in ice cream and other dairy products. Consequently, in this study, Maxilact 40,000 lactase (GB Fermentation Industries, Des Plaines, Illinois), a finely divided powder produced

from Saccharomyces lactis, was used to reduce whey lactose. According to a Technical Bulletin (GB Fermentation Industries), Maxilact is purified to be free of zymase and protease. It hydrolyzes the β-D-galactosidic linkage of lactose, with an activity of 40,000 ONPG units/g. Its optimum activity is at pH 6.5-7.0. Its activity is high at both 4 C (39.2 F) and 30 C (86 F), but the enzyme is inactivated at about 40 C (104 F). Potassium and ammonium ions enhance its activity, while sodium ion and heavy metals are inhibitory. In milk, 99+% hydrolysis is achieved at 4 C (39.2 F) for 24 hr using 225 mg enzyme per quart. At 30 C (86 F), 75% conversion is achieved with 75 mg/qt in 2 hr. Pasteurization before hydrolysis is unnecessary, but due to inhibitory activity in raw milk, slighlty lower conversions will result.

Preliminary small-scale experiments had shown that near-complete conversion of lactose to glucose and galactose in KOH-neutralized cottage cheese whey could be achieved with 600 mg/l Maxilact in 2-4 hr at 33 C (91.4 F). With $Ca(OH)_2$, 1200 mg/l was required to reproducibly give 99+% hydrolysis at pH 6.8-7.0. However, with 140 gal of whey in a large vat, hydrolysis was more efficient than expected, and probably less enzyme and a shorter holding period would have been adequate. In this study, therefore, incubation at 33 C (91.4 F) for 4 hr at pH 7.0 resulted in 98+% hydrolysis, using 600 and 1200 mg/l of enzyme for KOH- and $Ca(OH)_2$-neutralized wheys, respectively.

The two levels of hydrolysis used in the experimental mixes were arbitrarily chosen to be approximately 50% and 75% conversion to glucose and galactose.

Whey Supply and Pre-Treatment

Fresh, fluid cottage cheese whey was supplied by a commercial plant (Shamrock Foods Co., Phoenix, Arizona), and was shipped by refrigerated truck in six-gallon, plastic-lined, cardboard milk containers. Cartons were held at 33 ± 1 F (0.6 ± 0.6 C) in a walk-in cooler until used. Whey was 7-10 days old when processing began.

Approximately 140 gal (1190 lb; 540 kg) of cold whey was mixed in a 150-gallon, water-jacketed milk vat for 2 min. The agitator was then turned off and the vat was left undisturbed for 2 hr to allow the fine curd particles to settle to the bottom. A sanitary stainless steel pipe (38 mm O.D.) was introduced down the inside of the vat almost to the bottom, a siphon started, and the gravity-clarified whey was decanted into 10-gallon milk cans. An alternative method would have been to open the valve at the bottom of the vat to flush the fines out the bottom. Approximately 41 lb of casein fines, concentrated in a small amount of whey, were removed from 1190 lb of whey.

Neutralization and Hydrolysis of Whey

Neutralization and hydrolysis steps were carried out in the water-jacketed vats. Two vats, one of 150-gallon capacity for $Ca(OH)_2$, and the other of 50-gallon size for KOH, were used. The weight of clarified whey treated in each case was 340 lb (40 gal; 154 kg).

The temperature of the whey was adjusted to room temperature (25 C; 77 F) by the addition of steam to the water surrounding the vat. The pH electrode was introduced directly into the vat for constant monitoring of pH. With slow agitation, measured amounts of neutralizer

solution were added to the whey until the pH was steady at 7.0. Special care was required to avoid overneutralization when $Ca(OH)_2$ was used because of its low solubility and slow neutralizer action.

The whey lactose was hydrolyzed immediately after neutralization. Temperature of the whey was increased to 33 C (91.4 F) by adding steam to the vat water jacket. The calculated amount of enzyme was added [i.e., 600 mg/l for KOH and 1200 mg/l for $Ca(OH)_2$], and incubation at 33 C was continued for 4 hr.

During pre-testing, some difficulty was encountered in hydrating the low-density, fine-particle enzyme without lumps, since maximum enzyme efficiency was achieved only with a smooth suspension of the enzyme in whey. Three techniques were found to produce a smooth suspension: 1) adding a small amount of whey to a few grams of lactase powder, stirring to make a paste, and then diluting further; 2) adding the enzyme to a glass tissue grinder (VWR Scientific) and grinding out the lumps; and 3) adding the enzyme to a blender jar half-full of whey, and blending at low speed only long enough to disperse the enzyme without heat-denaturing it (a few seconds). The latter procedure was most satisfactory for use in this study because of the relatively large amount of enzyme (91 or 182 g) to be hydrated. To avoid the possibility of denaturation, the enzyme was added to neutralized whey at the incubation temperature.

At the completion of the incubation period, the whey was heated to 70 C (158 F) to inactivate the enzyme. Other workers have pasteurized after hydrolysis to kill any microorganisms which might have grown during the incubation period. However, in this work, the whey was to be

pasteurized within a few days in the mixes, so less rigorous heating was used to avoid cooked flavors and whey protein denaturation. Following enzyme inactivation, whey was cooled immediately to 48 C (118 F) by adding chilled water to the vat water jacket.

Adjusting Whey to Desired Hydrolysis and pH Levels

At the completion of hydrolysis, percent hydrolysis of lactose was determined colorimetrically. Concentration of lactose was determined on neutralized whey before hydrolysis by the method of Teles et al. (1978), and analysis of glucose liberated following hydrolysis was performed with the Glucostat Reagent Set (Worthington Diagnostics, Freehold, New Jersey). Percent hydrolysis was calculated by dividing the amount of glucose actually liberated by the amount of glucose expected from complete hydrolysis of lactose, assuming that 1 mg/ml lactose would liberate 0.5 mg/ml glucose.

Once the initial degree of hydrolysis in the whey was determined, plain acid whey was blended with hydrolyzed whey to give the desired final hydrolysis level for each of the eight mixes. Calculation of the proportions of plain and hydrolyzed whey required to give 50 or 75% hydrolysis was calculated using the Pearson square (Arbuckle, 1977, pp. 138-140). Final percent hydrolysis was verified using the Glucostat analysis and the calculations detailed above.

The addition of unneutralized acid whey to the neutralized, hydrolyzed whey reduced the pH below the desired levels of 6.5 or 6.8. Thus, on the day the ice cream mixes were made, whey for each mix was adjusted to the final level with measured amounts of 30% KOH or 30%

$Ca(OH)_2$. The amount of neutralizer added, in mg/l, was calculated for each mix, based on recorded weights of whey and 30% alkali solutions.

Preparation of Mixes

Each whey ice cream had the same composition, except for the variable array in the whey ingredient. The identity of the eight mixes, based on the three variables of whey treatment, is listed in Table 3. It can be seen that the variables are arranged in a 2x2x2 factorial experimental design. Mix I was a control of standard formulation. In this mix, whole milk replaced the neutralized, hydrolyzed whey as a bulk or volume source.

Table 3. Identification of whey variable array in ice cream mixes.

Mix	Neutralizer	pH	Lactose Hydrolysis (%)
A	KOH	6.5	50
B	KOH	6.5	75
C	KOH	6.8	50
D	KOH	6.8	75
E	$Ca(OH)_2$	6.5	50
F	$Ca(OH)_2$	6.5	75
G	$Ca(OH)_2$	6.8	50
H	$Ca(OH)_2$	6.8	75
I	Control	--	--

Ninety-two pounds (approximately 10 gal) of each mix was made, using the following formula: 10% fat, 11% MSNF, 9.7% granulated sugar (sucrose), 6.2% corn sugar (dextrose), and 0.35% stabilizer-emulsifier (0.31% in control). The ingredients used, in addition to the treated whey (3.30% fat whole milk in the control) were: fresh cream (41.74% fat); spray-dried, low heat, Grade A, nonfat dry milk (United Dairymen of Arizona, Tempe, Arizona); granulated sugar (Amstar, San Francisco, California); corn sugar (Staleydex Brand 333 Dextrose, A. E. Staley, Decatur, Illinois); and stabilizer-emulsifier (Ultra-Hi, Germantown Manufacturing Co., Broomall, Pennsylvania). Ultra-Hi contains cellulose gum, carrageenen, guar gum, locust gum, mono- and diglycerides, and polysorbate 80. The two ice cream formulations, in pounds and in percent by weight, are given in Table 4.

Although the hydrolyzed whey was noticeably sweeter than the milk used in the control mix, to insure ingredient uniformity, no reduction in the amount of sweeteners was made in the whey formulas.

During neutralization, a white precipitate of fine, soft particles formed, which was assumed to be whey protein. Previous trials had shown that this coagulum, after homogenization, was smoothly reincorporated into the mix; consequently, it was not removed from the whey. A similar precipitate was removed by decanting or centrifugation by Loewenstein et al. (1975).

In assembling the mix ingredients, the appropriate amount of whey or milk was mixed with about two-thirds of the cream needed for each mix in a 10-gal milk can with a sanitary fitting in the side near the bottom. The dry ingredients were added slowly to the can and were

Table 4. Formulas for whey and control mixes in lb per 92-lb batch, and in percent by weight of total composition.

Ingredient	lb	%
Whey Formulas		
Cream (41.74% fat, 5.51% MSNF)	22.43	24.39
Fluid whey (.07% fat, 6.38% MSNF)	48.81	53.06
Nonfat dry milk (0% fat, 95% MSNF)	5.80	6.31
Sugar	8.92	9.70
Corn sugar	5.70	6.20
Stabilizer-emulsifier	.32	.35
	91.98	100.01
Control Formula		
Cream (41.74% fat, 5.51% MSNF)	17.41	18.92
Milk (3.30% fat, 9.43% MSNF)	55.05	59.83
NFDM (0% fat, 95% MSNF)	4.64	5.04
Sugar	8.92	9.69
Corn sugar	5.70	6.19
Stabilizer-emulsifier	.29	3.1
	92.01	99.98

mixed and recirculated with the aid of a one-horsepower sanitary milk pump. The remaining cream was then added. This procedure was necessary to avoid excess foaming of the whey mixes.

Each mix was then transferred to a 10-gal, stainless steel milk can and heated to 160 F (71.1 C) in a starter-sterilizer water bath. Mixes were homogenized at 160 F (71.1 C) with a Manton-Gaulin, two-stage homogenizer (2000 and 500 lb/sq in. pressure on first and second stages, respectively).

Due to the small volume of each mix, the high-temperature, short-time pasteurizer unfortunately could not be used. Consequently, all mixes were vat-pasteurized in 10-gal milk cans placed in the starter-sterilizer at 165 F (73.8 C) for 30 min. Some difficulty was encountered in holding the pasteurization temperature steady, however, and temperatures as high as 172 F (78 C) were noted at times.

After pasteurization, each can of mix was immediately transferred to a 32-gal, plastic trash can filled with crushed ice. Mixes were either constantly agitated mechanically with a Lightening mixer, or were stirred periodically by hand with a stainless steel stirring rod, for approximately 1.5 hr. When the temperature of each mix reached 50 F (10 C), the can was transferred to a walk-in cooler (33 ± 1 F; 0.6 ± 0.6 C) and stored one week until freezing was scheduled.

Freezing Ice Cream

Before freezing, flavoring and coloring ingredients were added to each mix according to the manufacturer's instructions. A vanilla-vanillin flavoring (Petran Golden-Van No. 180-V, Petran Products,

Milwaukee, Wisconsin) was used which was four-fold vanilla with 1 oz vanillin added to each fold of vanilla. Annatto cheese color (Hansens Standard of the World Cheese Color, Chr. Hansen's Laboratory, Inc., Milwaukee, Wisconsin) was the coloring material used.

Mixes were frozen with a commercial Cherry-Burrell continuous-type freezer (85 gal/hr capacity), using ammonia as the refrigerant.

Percent overrun was checked periodically and adjusted to 90% as necessary. The calculation by weight method of Arbuckle (1977, pp. 194-196) was used to follow overrun in each mix.

Ice creams were drawn from the freezer into either one-gallon or half-gallon paper ice cream containers, or into pre-labeled 3.5-oz lidded plastic containers (Sweetheart Cup Corp., Chicago, Illinois). Samples were placed in the hardening room (-22 ± 2 F; -30 ± 1.1 C) within 2 min of freezing.

Temperature and appearance of each ice cream as it came from the freezer was recorded.

Samples in plastic cups were stored in the hardening room for approximately one month until sensory evaluation could be scheduled. Half-gallon and gallon cartons to be examined for storage stability and melt-down were transferred to a commercial-type freezer (0 ± 2 F; -17.8 ± 1.1 C) after two days in the hardening room.

Sensory Evaluation

Panel and Test Procedure

Acceptability of the ice creams was evaluated by an untrained panel of twenty University faculty, staff, and graduate and undergraduate

students. Panelists represented eleven departments in three colleges. There were about equal numbers of males and females, varying in age from about 20 to 55. The observers had no prior knowledge of the variables used in the ice creams. Information on tobacco use and frequency of ice cream consumption was volunteered by each panelist. The sensory test procedure was approved by the University Human Subjects Committee.

A 10-point (9 to 0) hedonic rating scale was used to evaluate preference for the nine ice creams. In a comparison that will be reported elsewhere, the panelists also rank-ordered the ice creams. Only the rating results will be reported here, but the use of two sensory evaluation methods is reflected in the experimental design.

To accommodate the availability of space, panelists were divided into two groups, and met at either 10-11 a.m. or 11-12 a.m. on each of four successive days. Rating and ranking procedures were alternated for each group during the four days, and on any given day, the two groups performed different scoring procedures. Ratings were replicated three times on each of two days.

Panelists were seated at individual booths, containing a score sheet (with sample numbers pre-coded on the sheets), pencil, spoon, napkin, glass of water, mouth rinse container, and scoring guidelines. They were given oral instructions regarding scoring methods, and general information regarding the nature of the study. The panel was advised of the validity of first impressions in making sensory judgments, and given the option of mouth rinsing between samples.

Figure 1 shows the score sheets used in ratings. Figure 2 is a facsimile of the rating guidelines posted in each booth.

Observation #_____

RATINGS SCORE SHEET

SAMPLE NO.									
PREFERENCE RATING									

Panelist Name_____ Date_____

Figure 1. Score sheet used by panelists in the rating procedure. -- Observation number, sample, number, and date were all pre-coded.

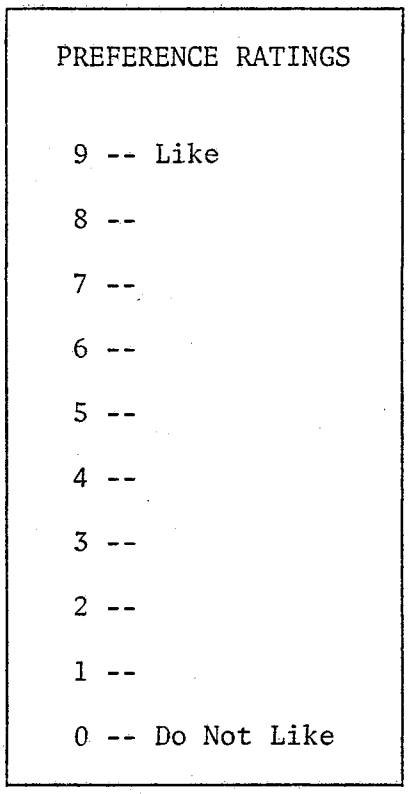

Figure 2. Rating guidelines used by panelists in scoring ice creams. -- These guidelines were posted in each booth.

Presentation of Samples

Sample cups were labeled with three-digit random numbers. A total of 27 ice creams was presented on each day of the ratings; i.e., nine different ice creams presented three times. On each day of testing, every ice cream was assigned a different random number for each replication. A table of random numbers was used to randomize the order of sample presentation as well. No practice or "warm-up" samples were given.

Samples were tempered at 0 ± 2 F (-17.8 ± 1.1 C) for approximately twenty hours before serving. Proctors presented each set of samples at a rate of about one per minute for nine minutes. A 3-4 min break was given between each set of nine samples for distribution of new score sheets, but communication between panelists during the break was not allowed.

Computational Procedures

Data from panel testing were compiled and coded onto computer cards. Analysis of data was by signal detection theory (Swartz, 1973; Angus and Daniel, 1974; Stull et al., 1974; Daniel and Boster, 1976). A factorial design analysis of variance was performed by computer as well.

Chemical and Physical Tests

Tests on Ingredients and Mixes

pH. A Fisher model 220 pH meter with combination electrode (Fisher Scientific Co., Pittsburgh, Pennsylvania) was used in all pH determinations. The instrument was standardized at room temperature

(25 C; 77 F) with either pH 4.0 or 7.0 standard buffer solution (Matheson, Coleman and Bell, Norwood, Ohio). Determination of pH on mixes was made after aging 24 hr.

Titratable Acidity. Acidity of wheys and mixes was determined in duplicate by titration with 0.1 N NaOH in the method described by Goss (1953, pp. 170-183), to the phenolphthalein end point (pH 8.3). Standardization of the 0.1 N NaOH was by the potassium hydrogen phthalate method (American Association of Analytical Chemists, 1975, Sec. 50.032-50.035). Titratable acidity tests on mixes were made after aging 24 hr.

Fat and Solids. Fat in mixes, whey, milk, and cream were determined in duplicate by the Mojonnier modification of ether extraction, as described by Goss (1953, pp. 297-325). Total solids were also measured with the Mojonnier milk tester (Mojonnier Brothers, Chicago, Illinois).

Protein. Protein in whey, milk, and ice cream mix was determined in duplicate by Kjeldahl. The Kjeldahl determination was a modification, for fluid samples, of the American Association of Analytical Chemists (AOAC) procedure (AOAC, 1975, Sec. 16.253). In the modification, 5 g ice cream mix or 10 g whey or milk were added to Kjeldahl flasks, taking care to avoid deposit of sample inside flask necks. Sample weights were determined either by the use of Mojonnier weighing pipettes and weighing tray (Mojonnier Brothers, Chicago, Illinois), or by difference, using a watch glass-covered beaker of sample. Boiling stones, a few crystals of catalyst, and 5 ml concentrated H_2SO_4 were

added to each flask, including flasks containing 5 or 10 ml distilled water for blanks. Flasks were heated at a very low temperature (3, on a scale of 1 to 7) until nearly dry (5-40 min, depending upon type of sample). After cooling, the remainder of the catalyst and H_2SO_4 was added and the procedure continued as in a normal determination.

Whey ice cream mixes analyzed in this study tended to foam on distillation, so heat was held very low (3, on a scale of 1 to 7) in the beginning and then gradually increased. The factor 6.38 was used to convert percent nitrogen to percent crude protein.

Because of the proposed ice cream standards changes, which would have specified minimum protein levels, interest has increased in finding an easier method than Kjeldahl to monitor ice cream protein content as a quality control procedure. It appears that dye binding can be an attractive alternative to the more hazardous and time-consuming Kjeldahl method (Kristoffersen and Miller, 1976; Bruhn, 1978; Kroger, Katz, and Weaver, 1978). Time did not permit comparison of these two analyses in the current study, however.

Lactose. The procedure of Teles et al. (1978) was used to determine lactose on duplicate samples of whey before hydrolysis. A 1:50 dilution of whey was made, and a standard curve between 0.2 to 1.0 mg/ml lactose was prepared. Color intensity was measured at 520 nm with a Bausch and Lomb model 20 spectrophotometer (Bausch and Lomb, Rochester, New York).

Glucose. The Macro Method described in the Glucostat Reagent Set (Worthington Diagnostics, Freehold, New Jersey) was used to analyze

glucose liberated by hydrolysis of lactose on duplicate samples of whey. A 1:25 dilution of whey, and a standard curve between 0.2 and 0.8 mg/ml glucose was used in the analysis. Color intensity was measured at 420 nm with the spectrophotometer described previously.

Relative Viscosity of Mixes. The pipette procedure described by Arbuckle (1977, p. 371) was used to measure the relative viscosities of triplicate mix samples. In this method, ice cream mix was drawn into a 25-ml transfer pipette supported by a buret clamp, attached to a ring stand. The time required for the sample to flow to the 25-ml mark was recorded to the nearest 0.1 sec.

Relative Protein Stability of Mixes. The ethanol precipitation method of Drawbridge (1951, p. 20) was used to determine the relative stability of the protein in duplicate mix samples. In this method, 1 ml of distilled water and 1 ml of ice cream mix (added with a Macro-Set Transfer Pipetting System, Oxford Laboratories, Foster City, California) were mixed in a 25-ml Erlenmeyer flask. The mixture was titrated with 95% ethyl alcohol until the first sign of a definite coagulum appeared. Results were recorded to the nearest 0.05 ml.

Analysis of Calcium and Potassium. Determination of Ca and K on whey, milk, and mix was done by a commercial laboratory using atomic absorption spectrophotometry (Perkin-Elmer 303, Perkin-Elmer, Norwalk, Connecticut) of dry-ashed samples.

Finished Ice Cream Tests

Scoring by Expert Panel. A panel of three experienced ice cream judges examined half-gallon cartons of each ice cream after one week, and then approximately once a month for five months. Scores for flavor, body and texture, and color were recorded using the American Dairy Science Association Dairy Products Judging guidelines (American Dairy Science Association, Champaign, Illinois). Additional half-gallons were examined at the same time for shrinkage. Samples for scoring and shrinkage were held in a commercial-type freezer at 0 ± 2 F (-17.8 ± 1.1 C) for the entire five-month scoring period.

Melt-Down. Melt-down appearance was described qualitatively after three months of storage in a commercial-type freezer at 0 ± 2 F (-17.8 ± 1.1 C). In addition, two quantitative methods of describing melt-down were employed on duplicate samples after three months in the commercial freezer. Melt-down samples (50 ± 6 g) were dipped from gallon cartons at 2 ± 2 F (-16.7 ± 1.1 C), using a standard commercial ice cream dipper.

In the first procedure, described by Drawbridge (1951, p. 21), the percentage melt-down in 45 min at room temperature (23 C; 73.4 F) was determined on duplicate samples. In this method, 75-mm funnels topped with 1/4-inch mesh wire screens were set up over tared 250-ml beakers. A uniformly dipped, weighed sample of ice cream was placed on the screen, and allowed to melt for 45 min. The melted ice cream was then weighed and percent melt-down calculated.

In the second procedure, described by Nickerson and Pangborn (1961), volume of melt-down was measured at five-minute intervals on duplicate samples held at room temperature (23 C; 73.4 F). Funnels and screens were set up over 50-ml graduated cylinders. Uniformly dipped ice cream was placed on the screens, and the volume of melted ice cream was recorded every five minutes for 45 min.

Additional Calculations

Some data were calculated on the basis of formulas given by Arbuckle (1977). These included: percentage overrun (p. 195), lb/gal of mix (p. 201), lb/gal of ice cream (p. 203), and lb/gal of food solids (p. 198).

In addition, the theoretical acidity of the mixes was calculated by multiplying the percent MSNF by 0.018 (Frandsen and Nelson, 1950, p. 107).

RESULTS AND DISCUSSION

Chemical and Physical Analysis of Whey

In this section, data are reported regarding neutralization and lactose hydrolysis of the cottage cheese whey. Results of this study are compared to results from similar studies, and improvements in the methods are suggested.

Each of the final wheys used in the eight experimental mixes contained a different combination of variables. The variables were: 1) neutralization of acid whey with either $Ca(OH)_2$ or KOH, 2) neutralization to either pH 6.5 or 6.8, and 3) hydrolysis of lactose to either 50% or 75% of complete conversion to glucose and galactose. Choice of neutralizer differentiated the two basic groups of wheys used in this study, because of the considerable differences in properties of the two alkalis.

Basic Composition and Neutralization

Table 5 gives the composition of fresh, untreated cottage cheese whey, as well as the changes in composition resulting from neutralization with KOH or $Ca(OH)_2$. Finally, the composition of the two batches of whey after hydrolysis of lactose are reported.

The table shows that, to reach the optimum pH for lactase enzyme activity (7.0), the acidity was reduced to an abnormally low level, especially with $Ca(OH)_2$ as the neutralizer. One indication of a

Table 5. Composition of fresh cottage cheese whey before and after neutralization of acidity and hydrolysis of lactose.

Analysis	Untreated Whey	Whey Following Acid Standardization		Whey Following Lactose Hydrolysis	
		$Ca(OH)_2$	KOH	$Ca(OH)_2$	KOH
pH	4.40	6.95	7.05	6.85	7.05
Titratable acidity, as lactic acid (%)	.551	.039	.065	.045	.067
Amount neutralizer added (ppm)	--	2234	3669	--	--
Amount Ca^{++} or K^{+} added (ppm)	--	1175	2554	--	--
Ca (ppm)	1056	--	--	--	--
K (ppm)	1233	--	--	--	--
Fat (%)	.07	.07	.06	.06	.04
Total solids (%)	6.45	6.72	6.73	6.92	6.84
MSNF (%)	6.38	6.66	6.67	6.86	6.80
Moisture (%)	93.48	93.28	93.27	93.08	93.16
Protein (%)	.84	--	--	--	--
Lactose (mg/ml)	45.31	45.13	44.68	--	--
Glucose (mg/ml)	.34	--	--	--	--

desirable level of acidity in the neutralized whey would be to compare it with the titratable acidity of sweet whey (0.12%) (Webb, 1970a, p. 105).

In the current study, acidity of whey was reduced by gradual addition of neutralizer solution, while constantly monitoring pH. In commercial practice, it might be preferable to calculate the amount of alkali required to neutralize the desired amount of lactic acid in the whey. The neutralizer required for a given volume of whey could then be dissolved or suspended in water and quickly added to the whey without the need for constant pH determinations. Another alternative would be the addition of a neutralizer solution from a continuous-flow metering device. In neutralization, KOH is a more predictable neutralizer than $Ca(OH)_2$, because more than the calculated amount of the latter alkali is required to give the expected neutralizing strength (Hunziker, 1940, pp. 251-252). Within a given plant, however, the acidity of the whey supply is usually relatively constant, and only a few trials with the neutralizer to be used would give an indication, within an acceptable range of final acidities, of the amount of neutralizer required for a given amount of whey.

The amount of neutralizer required to standardize the acidity to pH 7 is shown, in parts per million (mg/l). This calculation is based on the amount of 30% solution added to a specific amount of whey. The amount of Ca^{++} and K^{+} added was calculated from the percentage contribution of each ion to the molecular weight of the alkali. It can be seen that $Ca(OH)_2$ was the stronger neutralizer, as would be expected by calculation (Hunziker, 1940, pp. 252-254).

As previously mentioned, neutralization caused a white precipitate to form, which was assumed to be whey protein, and this soft coagulum was not removed. Weetall et al. (1974) noted a white precipitate upon neutralization of acid whey ultrafiltrate, which was found to be calcium phosphate. Potter and Williams (1949) found that heat-coagulated whey protein retarded whipping in whey sherbets. The excellent whipping properties characteristic of whey is associated with portions of whey not coagulated by heat.

Table 5 shows that there was a slight increase in total solids following both neutralization and lactose hydrolysis. This was probably due to the addition of inorganic matter in the neutralizers. The source of the small amount of glucose in the untreated whey is presumably the result of lactose hydrolysis by residual lactase-secreting microorganisms in the whey. Tobias (1970) and Bouvy (1974) recommended pasteurizing whey after removing it from the cheese vat to kill any remaining cheese starter organisms. In the current work, this was not done, and perhaps the glucose in the untreated whey resulted from the lactase activity of Streptococcus lactis in the starter culture.

Table 6 shows compositional data of the final wheys, milk, and cream, which are related to the neutralization procedure. The pH and titratable acidity measurements were taken 48 hr after neutralization, so that $Ca(OH)_2$ should have been completely dissociated. The titratable acidity measurements again point out the difficulty of avoiding over-neutralization when using $Ca(OH)_2$, because of its slow action.

An indication of the amount of neutralizer added to the final wheys is seen by examining the concentration of Ca and K, as measured by

Table 6. Composition, relating to neutralization, of fluid whey, milk, and cream used in ice cream mixes.

Mix Ingredient	pH	Titratable Acidity, as Lactic Acid (%)	Ca (ppm)	K (ppm)
Whey for mixes:				
A	6.52	.152	1026	2843
B	6.50	.158	1110	3057
C	6.82	.104	877	2544
D	6.80	.108	1038	3075
E	6.59	.073	1096	1263
F	6.52	.081	1776	1280
G	6.86	.046	1327	794
H	6.80	.056	1096	1263
Milk for Mix I	6.70	.153	1044	1233
Cream	6.69	.090	--	--

atomic absorption. Wheys A-D and E-H were treated with K and Ca, respectively. The levels of Ca and K in the untreated whey are given in Table 5. The mean increase in Ca and K levels in the final wheys, as compared to the untreated whey (Tables 5 and 6), is 9.6% and 38.8%, respectively.

Table 7 shows the similarity of the final wheys with regard to fat, solids, moisture, and protein levels. The composition of the final wheys is contrasted to the milk used in the control mix. Cream of identical composition was used in all nine ice creams. There are no data on protein in the cream because an attempt to analyze it by the Kjeldahl modification outlined in the Materials and Methods was unsuccessful in this high-fat product. Excessive spattering of fat during acid digestion was responsible for loss of nitrogen-containing material in the cream.

Hydrolysis of Lactose

As detailed in the Materials and Methods, whey neutralized with either $Ca(OH)_2$ or KOH was hydrolyzed to an optimum level with Saccharomyces lactis lactase (Maxilact) enzyme. The percent conversion to lactose to glucose and galactose was then determined using lactose and glucose assay.

Table 8 shows details on determination of degree of hydrolysis. The concentration of glucose expected was assumed to be one-half the concentration of lactose. The small amount of glucose present in the whey before hydrolysis was treated as a blank, and subtracted from the expected glucose. Following these calculations in the table, it can be

Table 7. Basic composition of fluid whey, milk, and cream used in ice cream mixes.

Mix Ingredient	Fat (%)	Total Solids (%)	Solids-Not-Fat (%)	Moisture (%)	Protein (%)
Whey for mixes:					
A	.07	6.53	6.46	93.47	.87
B	.07	6.65	6.58	93.35	.88
C	.10	6.73	6.63	93.27	.87
D	.06	6.64	6.58	93.36	.85
E	.07	6.74	6.67	93.26	.86
F	.06	6.75	6.69	93.25	.90
G	.05	6.64	6.59	93.36	.88
H	.06	6.69	6.63	93.31	.90
Milk for Mix I	3.30	12.72	9.42	87.28	3.35
Cream	41.74	47.25	5.51	52.75	--

seen that 99.4 and 97.8% hydrolysis was achieved in $Ca(OH)_2$- and KOH-neutralized wheys, respectively.

Table 8. Calculation of degree of lactose hydrolysis in whey neutralized with either $Ca(OH)_2$ or KOH.

Analysis	Whey Neutralized with $Ca(OH)_2$	Whey Neutralized with KOH
	------- mg/ml -------	
Lactose, before hydrolysis	45.13	44.68
Glucose, before hydrolysis (blank)	.23	.34
Glucose, after hydrolysis	22.20	21.52
Expected glucose (calculated value)	22.57	22.34
Expected glucose (blank)	22.34	22.00
Degree of lactose hydrolysis (%)	99.4	97.8

Figure 3 illustrates the use of the Pearson square to determine the proportions of hydrolyzed and untreated wheys to be mixed to give the desired hydrolysis level. The example is the calculation used in producing KOH-neutralized whey at 75% conversion of lactose.

Table 9 lists the number of pounds of each ingredient used in this study to give 60 lb of either 50 or 75% hydrolysis, when either $Ca(OH)_2$ or KOH served as the neutralizer. Following subsequent standardization to pH 6.5 or 6.8 with either KOH or $Ca(OH)_2$, the eight 60-lb batches of whey were ready to be used in the various ice cream mix formulations.

PROBLEM: How much hydrolyzed whey of 97.8% conversion and how much untreated whey must be mixed to give 60 lb of whey whose lactose is 75% hydrolyzed?

SOLUTION: Have 97.8% hydrolyzed whey and 0% untreated whey. Want 60 lb whey at 75% hydrolysis level.

Hydrolyzed	97.8	75.0 parts	76.7%	46.02 lb	hydrolyzed
Untreated	0	22.8 parts	23.3%	13.98 lb	untreated
		97.8 parts	100.0%	60.00 lb	

46.02 lb of 97.8% hydrolyzed whey mixed with 13.98 lb of untreated whey give 60 lb of 75.0% hydrolyzed whey.

EXPLANATION:

Step 1. Draw a rectangle with two diagonals. In the center, place the desired hydrolysis level, and at the left-hand corners place the hydrolysis levels of the starting ingredients.

Step 2. At the right-hand corners place the differences, subtracting diagonally, disregarding signs.

Step 3. Add the differences, to give the total number of parts of each ingredient.

Step 4. Calculate the proportion of each ingredient in percentage, based on the number of parts.

Step 5. Using the percentage of each ingredient and the desired number of pounds of whey, calculate how many pounds of each ingredient to use. All of the figures at the top of the square refer to the 97.8% hydrolyzed whey, while the bottom figures pertain to untreated whey.

Figure 3. Use of Pearson square to determine the amounts of hydrolyzed (97.8% conversion) and untreated (0% conversion) wheys that must be combined to give 60 lb of whey at 75% lactose conversion. -- After Arbuckle (1977, pp. 138-140).

Table 9. Number of pounds each of hydrolyzed (ca. 100%) and untreated (0%) wheys mixed to give 60 lb of 50 or 75% hydrolyzed whey.

Resulting Whey	Hydrolyzed Whey Used (lb)	Untreated Whey Used (lb)
50% hydrolysis, KOH neutralized	30.7[a]	29.3
75% hydrolysis, KOH neutralized	46.0[a]	14.0
50% hydrolysis, $Ca(OH)_2$ neutralized	30.2[b]	29.8
75% hydrolysis, $Ca(OH)_2$ neutralized	45.3[b]	14.7

[a]97.8% of lactose hydrolyzed.

[b]99.4% of lactose hydrolyzed.

Table 10 shows the results of experimental verification of the final hydrolysis level in the final wheys. It can be seen that the desired levels of 50 and 75% of complete hydrolysis were not achieved. Wheys A through D (KOH) ranged from 1.7 to 4.0% below the desired level, while wheys E through H [$Ca(OH)_2$] were 5.1 to 8.0% below expected levels.

Unfortunately, time did not permit verification of the final hydrolysis levels before the whey was used in the mixes. Consequently, the problem of lower-than-expected conversion was not discovered in time to correct the calculations for the weights of whey to be used. However, since thelevels of hydrolysis were chosen arbitrarily, it was felt that any levels would be acceptable as long as they were reasonably consistent between the two neutralizers.

Table 10. Experimental verification of percent of lactose hydrolyzed in fluid whey used in ice cream mixes.

Final Whey	Desired Parameters			Actual Degree of Hydrolysis (%)
	Neutralizer	pH	% Lactose Hydrolysis	
A	KOH	6.5	50	47.7
B	KOH	6.5	75	71.6
C	KOH	6.8	50	48.3
D	KOH	6.8	75	71.0
E	$Ca(OH)_2$	6.5	50	44.9
F	$Ca(OH)_2$	6.5	75	67.6
G	$Ca(OH)_2$	6.8	50	44.3
H	$Ca(OH)_2$	6.8	75	67.0

Therefore, an important consideration with regard to lactose hydrolysis was the comparison between the two neutralizers at a given pH. Table 11 shows that the $Ca(OH)_2$ percent hydrolysis at either pH was consistently only 4% below the corresponding level in the KOH-neutralized whey, with the exception of the 2.8% difference at pH 6.5, 50% desired hydrolysis. Any difference between the two neutralizers in sweetness, freezing point, or other ice cream property was probably insignificant at these low levels of difference.

Table 11. Comparison of degree of lactose hydrolysis achieved at a given pH with either KOH or $Ca(OH)_2$ neutralization.

pH and Desired Hydrolysis Levels	Actual Degree of Hydrolysis Achieved (%)		
	KOH	$Ca(OH)_2$	Difference
pH 6.5, 75%	71.6	67.6	4.0
pH 6.8, 75%	71.0	67.0	4.0
pH 6.5, 50%	47.7	44.9	2.8
pH 6.8, 50%	48.3	44.3	4.0

The reason for the discrepancy between the expected and the actual levels of hydrolysis is not clear. Presumably, microbial and chemical changes could have occurred in the whey, since it was at an optimum pH and contained readily metabolizable glucose. It probably would have been preferable to pasteurize the whey after hydrolysis at 33 C, to minimize microbial activity.

Five days after the original determination of degree of lactose hydrolysis, the analysis was repeated with quite different results. The revised hydrolysis levels were 84.80 and 88.40%, respectively, for $Ca(OH)_2$- and KOH-neutralized whey. If new Pearson square calculations are performed to determine the proportions of untreated and hydrolyzed wheys, the percent difference from the amounts actually used is very similar to the difference in percent conversion the second time compared to the first.

Vujicic, Lin, and Nickerson (1977) explained a similar discrepancy in predicted degree of hydrolysis as being due to the formation of oligosaccharides. Perhaps a similar explanation could be applied here.

Unfortunately, these conflicting results could not be repeated because of the shortage of Glucostat Reagent. Because of the anticipated replacement of this product on the market by Statzyme, the laboratory supply ran out and did not permit replication of these results a third time.

Conclusions Regarding Neutralization and Hydrolysis.

Most of the data in this study indicate that KOH is better than $Ca(OH)_2$ for acid standardization. This conclusion is based on chemical, physical, and sensory considerations. In addition, its water solubility makes KOH easier to use. Its immediate dissociation in whey makes it more predictable in its effect, and reduces the danger of overneutralization. KOH is more expensive than $Ca(OH)_2$, however, and in concentrated solutions is corrosive.

In terms of ingredient cost, the most expensive treatment used in this study was that of hydrolysis with lactase enzyme. As detailed in the Materials and Methods, Maxilact 40,000 yeast lactase was used in soluble form; when hydrolysis was completed, the enzyme was irreversibly heat-denatured.

On the basis of price information supplied by the manufacturer, the average enzyme cost for each 92-lb batch of ice cream was $4.50, or about 45¢ per gallon. If the KOH-neutralized mixes alone are considered, the hydrolysis cost if $3.07, or 30¢ per gallon.

A less-purified grade of Maxilact is available, which has the same activity but contains small amounts of proteases. This less-expensive grade of enzyme might have been acceptable for use in this study, because the protein content of whey is so small, and because the whey is used as an ingredient in ice cream mixes where any off-flavor may not have been detectable. In this case, the average cost per mix would have been 75¢. If only the KOH-neutralized mixes are considered, the cost would have been only 50¢ per batch, or 5¢ per gallon of ice cream.

Several authors (Reithel and Kim, 1966; Hill and Huber, 1971; Giacin et al., 1974) have reported on the detrimental effects of divalent cations (e.g., Ca^{++} and Mg^{++}) on activity of bacterial and fungal lactases. In addition, there are poorly defined interrelationships between monovalent ions (e.g., K^{+} and Na^{+}) and divalent ions present in the substrate which may cause activation or inhibition of enzyme activity (hill and Huber, 1971). Guy and Bingham (1978) recently reported that Ca^{++} ions reduced lactase activity by 14% in skim milk. Consequently, it

would not be unreasonable to suspect that improved efficiency of hydrolysis could be achieved in whey which had not been neutralized. This would suggest that the use of Aspergillus niger lactase, which has an optimum pH of 4.5, would have been a better choice in the current work, because it could be used in acid whey without neutralization. With improved efficiency, enzyme cost could be reduced.

Efficiency of hydrolysis may also be related to competitive inhibition by galactose, especially at higher levels of conversion (Woychik and Wondolowski, 1973). The microbial quality of the whey is important in determining lactase efficiency as well. Microbial quality, in turn, may be influenced by the temperature of hydrolysis and whether pasteurization has preceded hydrolysis (Woychik and Wondolowski, 1973; Wierzbicki and Kosikowski, 1973c). The optimum temperature for A. niger lactase is 55 C (131 F), while Saccharomyces lactis lactase may be used at 4 C (39.2 F). Both of these temperatures would have been preferable to the 33 C (89.6 F) used in this study, from the standpoint of discouraging microbial activity. In addition, heat treatment may inactivate inhibitory compounds in whey, to enhance hydrolysis with lactase (Wierzbicki and Kosikowski, 1973c). Guy and Bingham (1978) commented that pasteurization of whey before hydrolysis is essential to eliminate starter organisms which could reduce pH to below optimum levels for lactase.

The ultimate in efficient use of lactase would have been to immobilize it and re-use it. This is, of course, possible because enzymes are simply chemical catalysts, and are not used up or destroyed during use. There are many detailed articles on the subject of lactase

immobilization, and very good results have been demonstrated (Wierzbicki, Edwards, and Kosikowski, 1973; Wierzbicki et al., 1974; Woychik and Wondolowski, 1973; Weetall et al., 1974; Giacin et al., 1974). A. niger lactase is apparently more suitable for immobilization than are the lactases from other sources (Woychik and Wondolowski, 1973).

Shukla (1975) mentions two possible disadvantages of using lactase-hydrolyzed dairy products in foods. The first problem is with oligosaccharide formation, in which small amounts of indigestible sugars are produced. As a result, in the intestine, flatulence and bloating may result in sensitive individuals. The second possible problem is with high intake of galactose in persons suffering from congenital galactosemia, which is caused by a transferase deficiency. Consequently, galactose-containing foods are contraindicated for galactosemics.

Chemical and Physical Analyses of Ice Cream Mixes

In this section, data are reported regarding the composition and properties of ice cream mixes before freezing. These analyses are important because many of the legal requirements outlined in the Ice Cream Standards or Identity are based on mix properties.

Basic Composition

Table 12 details the composition parameters which relate to legal requirements. Levels of fat, total solids, and protein are of interest with regard to these standards.

Fat. The percent fat in the mixes ranged from 10.03 to 10.53, probably because of slight variations in weighing ingredients. All of

Table 12. Basic composition of ice cream mixes.

Mix	Fat (%)	Total Solids (%)	SNF[1] (%)	MSNF[2] (%)	Moisture (%)	Protein (%)	Wt./Gal. (lb.)
A	10.46	38.14	27.68	11.43	61.86	2.80	9.23
B	10.53	38.41	27.88	11.63	61.69	2.96	9.20
C	10.44	39.20	28.76	12.51	60.80	3.17	9.21
D	10.43	38.15	27.72	11.47	61.85	3.13	9.20
E	10.22	39.22	29.00	12.75	60.78	2.87	9.25
F	10.03	38.28	28.25	12.00	61.72	3.22	9.22
G	10.12	38.59	28.47	12.22	61.41	2.99	9.23
H	10.29	38.29	28.00	11.75	61.71	2.95	9.21
I	10.19	38.82	28.63	12.42	61.18	4.07	9.17

[1](Percent Total Solids) - (Percent Fat).

[2]Estimated by subtracting non-milk solids (sweeteners, stabilizer-emulsifier) from solids-not-fat (i.e., for Mixes A-H, non-milk solids = 16.25%; for Mix I, non-milk solids = 16.21%).

the mixes were above the legal minimum of 10.0% fat. Any differences in perceived richness among the nine ice creams due to these variations were probably insignificant (Swartz, 1973, p. 51).

Solids. Total solids were determined by Mojonnier desiccation of a sample of mix. Solids-not-fat (SNF), which include solids of non-milk origin (those of sugar, corn sugar, and stabilizer-emulsifier), were calculated by subtracting the determined values for percent fat from the ones for percent total solids. Milk solids-not-fat (MSNF), in turn, were estimated by subtracting the theoretical percentage of non-milk solids in the mix formula from the SNF. The percent MSNF is only an estimate because there is no laboratory test to differentiate the solids of milk ingredients (protein, minerals, lactose, glucose, and galactose) from those of non-milk origin. This calculation serves only as a check on the mix formulation in which 11% MSNF was desired. It can be seen that all of the mixes were slightly above the MSNF level set by the mix composition formula.

Protein. The whey mixes contained less protein than the control, but they were all within the legal minimum of 2.7% set by the proposed standards of identity. The whey mixes could have been supplemented with even more protein simply by increasing the non-fat dry milk by 1 or 2%.

In a study by Kristoffersen and Miller (1976), fifteen brands of commercially available ice cream were checked for protein content. The range for the samples in February, April, and June of 1974 was 2.49 to 4.38%, with a mean of 3.41% protein. They concluded that protein content

of ice cream was determined by company policy and was related to the amount of whey solids substitution for non-fat dry milk.

Loewenstein (1975) reported on an experimental formulation in which hydrolyzed whey (50%) was used to supply 100% of the MSNF. The protein content in this mix was 1.82%. When whey concentrate replaced 50% of the MSNF, the protein content of the mix was 3.11%.

As Table 12 shows, the range in protein content for the whey mixes in this study was 2.80-3.17%, with a mean of 3.01%. The whey mices contained 53% of that component by weight (Table 4), and they contained an average of 11.97% MSNF (Table 12). The final wheys contained an average of 6.61% SNF (Table 7). Therefore, the whey contributed an average of 3.50% of the total MSNF, for an average replacement of 29.2% of the MSNF in the whey mixes. The whey mixes, with an average of 29.2% replacement of the MSNF by fluid whey solids, contained an average of 3.01% protein.

The possibility of reduced protein content in frozen desserts containing whey has been a matter of concern in the controversy over changes in ice cream regulations. In contrast to the present study, practically all of the previous work has involved replacement of up to 25% of the MSNF by whey powder or concentrate. In the present work, however, the bulk of the non-fat solids is supplied by nonfat dry milk, and fluid whey replaces the fluid milk of the standard formulation. Table 4 illustrates this difference between the control and whey ice creams.

This point has two important implications related to the standards controversy. The first is one of economics. As mentioned

previously, the primary objection of the milk producers to the standards was one of losing some of their dry milk markets. But, as Table 4 clearly shows, the whey ice creams (A-H) utilized even more dry milk than did the control (I). On the other hand, for the ice cream manufacturer producing these whey desserts, the ever-increasing cost of dry milk would have to be offset by savings elsewhere, if prices for the whey product are to be competitive.

The second point is that of replacing protein. In this work, liquid acid whey was found to contain approximately 0.9% protein, compared to 3.35% for fluid whole milk (Table 7). Dry sweet whey, the product generally used in ice cream, contains 13.0% protein (Glass and Hedrick, 1977), compared to NFDM at 34.86% protein (Feeley et al., 1972). If one assumes that 25% of the NFDM in the control (Mix I) is replaced by dry whey, the mix protein would be reduced by nearly 15%, from 4.07% to 3.48%. This latter figure agrees with the value obtained experimentally (3.14%) by Kristoffersen and Miller (1976).

In contrast, the average protein content of the whey ice creams is 3.01%, and calculations show a reduction of only 0.5% protein from the 25% whey substituted produce when liquid whey completely replaces milk in the formula. Due to the dessert nature of ice cream, it is questionable whether this slightly reduced protein content would be considered critical in practical situations (Kristoffersen and Miller, 1976).

These calculations are based on assumptions, of course, but they do illustrate that the reduction in protein through use of fluid whey is not as drastic as might be expected.

The cost of using fluid whey in place of milk in frozen desserts would reflect the cost of enzyme and neutralizer, possible additional labor and equipment, and more extensive use of dry milk. However, for a cottage cheese manufacturer who is assessed sewage treatment charges for acid whey disposal, liquid whey ice creams could offer an attractive alternative. Because of the increased sweetness of hydrolyzed lactose, sweetener costs could be reduced as well.

Additional studies could be undertaken to determine whether lactase enzyme is actually required to prevent sandiness in the whey ice creams, and if so, what extent of hydrolysis is needed. It is not inconceivable that heat-shock experiments would prove that these ice creams are resistnat to sandiness without lactose hydrolysis for an acceptably long storage period. If not, lactase immobilization could make hydrolysis profitable.

The social cost of polluting the environment with unused acid whey should not be overlooked. Whey utilization is one of the great benefits of using these formulations. The esthetic benefit could be a major factor in determining if this method is feasible.

The environmental quality issue in the past has been important enough to warrant petitions to allow the use of acid whey in ice cream (Arbuckle and Singh, 1971; Igoe et al., 1973). The most recent petition has been from the International Association of Ice Cream Manufacturers (IAICM) in response to FDA's 1978 withdrawal of the proposed changes in ice cream standards (IAICM, 1978).

Cottage cheese whey has excellent keeping qualities in the non-neutralized state. The pH of 4.5 is low enough to prevent growth of

most microorganisms indefinitely, and it can be frozen with no curdling or other deterioration of properties. The high acidity could conceivably present problems with corrosion of non-acid-resistant materials, however.

Composition as Related to Neutralization

Table 13 gives data for the mixes as related to the neutralization of acidity.

Normal mix acidity and pH are a function of MSNF content; as MSNF increases, the acidity increases, while the pH decreases (Arbuckle, 1977, p. 50). The normal acidity of a mix can be calculated by multiplying the percent MSNF by the empirical factor 0.018 (Frandsen and Nelson, 1950, p. 107). Normal mix pH is about 6.3 (Arbuckle, 1977, p. 50); the normal pH of milk is 6.5-6.7, while the acidity is normally 0.15-0.18%, calculated as lactic acid (Johnson, 1974, p. 4).

The factor 0.018 has been determined experimentally for milk-based mixes, and represents a graphical function of the relationship of MSNF to titratable acidity. Arbuckle (1977, p. 50) shows this function in the form of a table. The whey used in the experimental mixes contained about the same amount of fat, lactose, and minerals as skim milk, but the percent protein was about 0.9%, compared to 3.5% for skim milk (Table 1). Consequently, it is probably not completely correct to use the factor 0.018 to calculate the normal acidity of the whey-based mixes, but a detailed study would have been required to determine the appropriate factor for whey-based mixes. Nevertheless, data are shown in

Table 13. Composition, as related to neutralization, of whey (A-H) and control (I) mixes. -- Mixes A-D and E-H were neutralized with KOH and $Ca(OH)_2$, respectively.

Mix	pH	Titratable Acidity, as Lactic Acid (%)	Theoretical Acidity (%)	Relative Protein Stability (ml)	Relative Viscosity (sec/25 ml)	Ca (ppm)	K (ppm)
A	6.31	.209	.206	.85	24.3	1408	3021
B	6.31	.213	.209	.88	24.6	1314	2685
C	6.42	.187	.225	.90	25.0	1119	2355
D	6.45	.189	.206	.90	24.6	1343	3219
E	6.31	.204	.230	.63	29.8	1841	2069
F	6.29	.209	.216	.53	27.6	1942	2100
G	6.37	.184	.220	.60	29.0	2144	2235
H	6.31	.204	.212	.53	34.9	1870	1965
I	6.50	.182	.224	1.58	22.7	1320	1815

Table 13 for theoretical acidity, calculated by multiplying MSNF by 0.018, to serve as a rough estimate of the expected acidity.

The table shows that the mixes containing whey which had been standardized to pH 6.5 (A, B, E, F) demonstrated a mix pH close to 6.3, while the mixes made with whey at pH 6.8 had a higher pH. The high pH of the control mix is unexpected, because the milk contained in it was of normal pH and acidity (Table 6).

The KOH mixes containing whey at pH 6.5 (A, B) had nearly theoretical acidities. The pH 6.5 $Ca(OH)_2$ mixes were more unpredictable, and no definitive statement can be made about acidity.

From the data, it would appear that the mixes containing whey at pH 6.8 (C, D, G, H) were slightly overneutralized, while the mixes with pH 6.5 whey (A, B, E, F) were more nearly normal.

An inspection of the titratable acidities of several mixes at the same pH reveals some inconsistencies. Likewise, there are exceptions to the trend of increasing pH with decreasing acidity. This study confirms the conclusion of previous studies (Dahle, Budge, and Keith, 1930; Gould and Potter, 1948; Drawbridge, 1951, pp. 24-28; Loewenstein et al., 1975) that titratable acidity of an ice cream mix cannot be precisely predicted by pH.

Relative Protein Stability. In the alcohol test, protein stability is directly related to the number of milliliters of alcohol used; i.e., the more alcohol required to coagulate the protein, the more stable the protein. These data (Table 13) show that the mixes neutralized with $Ca(OH)_2$ (E-H) were slightly less stable than those neutralized

with KOH (A-D). All of the acid-standardized mixes were less stable than the control (I).

Drawbridge (1951, p. 32) found that the relative stability of the mix protein was inversely related to the mix acidity. The addition of Ca and Mg acid standardizers reduced the protein stability more than did the other neutralizers. Dahle and Rivers (1940) found NaOH neutralization to be less detrimental to protein stability than $Ca(OH)_2$.

Drawbridge (1951, pp. 51-53) explained the decrease in protein stability with the addition of neutralizers as an effect on the particles' electric charge. Divalent ions from neutralizers, or hydrogen ions from developed acidity, decrease the negative charge on the protein molecules enough to cause the proteins to clump together and precipitate out. A similar effect on fat clumping will tend to increase the viscosity of neutralized mixes, and contribute to a curdled melt-down.

Dahle and Rivers (1940) noted that sodium ions were less detrimental than calcium ions in destabilizing protein and in increasing viscosity, but the opposite effect was noted on the stablity of melt-down.

The increase in protein stability of neutralized mixes compared to high-acid mixes is mainly due to the fact that the pH is increased above the isoelectric point of the protein (Dahle and Rivers, 1940). This beneficial effect is partially offset by the destabilizing effects of the neutralizing ions, however.

Relative Viscosity. In the relative viscosity test, the viscosity is directly related to the time required for the mix to flow

through the pipette. The data in Table 13 show that the whey mixes were slightly more viscous than the control, and that mixes E-H, neutralized with $Ca(OH)_2$, were slightly more viscous than mixes A-D, which were neutralized with KOH. The exceptionally high viscosity of mix H was first noted in cooling the mix after pasteurization. Tables 6 and 7 reveal nothing unusual about the composition of the whey used in this mix; the only readily apparent explanation for the high viscosity is a possible small weighing error in the amount of stabilizer-emulsifier added.

Arbuckle (1977, pp. 52-53) states that mix viscosity may influence smooth body, resistance to melting and rate of whipping in the freezer. Desirable viscosity can be produced only if the mix has a balanced composition with regard to fat, salts, and stabilizer. Temperature, total solids content, pasteurization, homogenization, and aging are important as well.

Drawbridge (1951, pp. 30-31) noted an increase in mix viscosity as the acidity increased, and when Ca and Mg neutralizers were used. The holding method of pasteurization (160 F for 30 min) produced more viscous mixes than did the high temperature-short time period.

Loewenstein et al. (1975) noted increases in viscosity in mixes containing KOH-neutralized cottage cheese whey concentrates, especially after 24 hr aging. The relatively large amount of stabilizer-emulsifier (0.32%) probably was more significant than the use of whey ingredients in producing viscous mixes.

The amount of stabilizer-emulsifier required by the whey formulations has been determined by trial-and-error in the present study, and

no in-depth investigation of other combinations of stabilizing ingredients was attempted. In a trial batch of whey ice cream, 0.5% stabilizer-emulsifier was used, and produced an overstabilized mix which was extremely viscous and difficult to whip. In contrast, in a batch of whey ice cream made after the completion of this work (see Appendix), a stabilizer level of 0.31%, equal to the amount in the control, was found to be adequate. Consequently, the 0.35% level in these mixes is probably too high, and some problems in properties of the whey ice creams, including viscosity, may be partially explained by overstabilization. Studies could easily be done to determine the optimum blend of stabilizing and emulsifying ingredients for these products as well, but such work was beyond the scope of the present project.

Calcium and Potassium Content. Table 13 lists the calcium and potassium contents of the mixes, as determined by atomic absorption spectrophotometry. The amount of these ions in the mixes is not well-correlated with the analysis of the treated wheys (Table 6).

Properties of Ice Cream

In this section, data are reported regarding properties of the finished ice cream. Freezing characteristics are important quality factors in frozen desserts, and also relate to legal requirements. The stability of ice cream in storage is of considerable concern in determining shelf life. Acceptability of new products by the consumer is the primary goal of any product development work, and may be predicted by taste panel observations.

Freezing Properties and Legal Status

Table 14 gives data regarding mix freezing properties and shows the result of computations on overrun and weight per gallon of the finished ice cream.

Drawing Temperature and Appearance. In order to produce small ice crystals and a fine-textured ice cream, the mix must be rapidly frozen to a low temperature (Arbuckle, 1977, p. 240). In a continuous freezer, freezing time should be about 24 sec, and drawing temperature about 21-22 F. Mix characteristics which affect freezing time include composition, freezing point, mix processing methods, and acidity of ingredients (Arbuckle, 1977, p. 241). Mechanical characteristics of freezer operation are also reflected in freezing time and temperature. The drawing temperatures listed in Table 14 are consistent with adequate freezer operation.

The KOH-neutralized whey ice creams (A-D) had a more desirable, firm, dry appearance as drawn from the freezer than did the control (I). The $Ca(OH)_2$-neutralized ice creams (E-H) were noticeably gassy in appearance, although relatively dry. This gassy consistency was not encountered in any trial batch of ice cream containing non-hydrolyzed whey, and its occurrence was unexpected. Perhaps the reduced freezing point of the mix as a result of lactose hydrolysis, coupled with the divalent Ca^{++} ions, produced less than desirable freezing conditions. The gas pockets were barely noticeable in the containers after hardening.

Table 14. Freezing properties and computations relating to legal status of whey (A-H) and control (I) ice cream.

Ice Cream	Temperature from Freezer °C	Temperature from Freezer °F	Appearance from Freezer	Overrun (%)	Wt/Gal Ice Cream (lb)	Wt/Gal Food Solids (lb)
A	-6.8	19.8	Firm, dry, smooth	95	4.73	1.80
B	-7.8	18.0	Firm, dry, smooth	95	4.72	1.81
C	-6.7	20.0	Firm, dry, smooth	95	4.72	1.85
D	-6.8	19.8	Firm, dry, smooth	93	4.77	1.82
E	-6.7	20.0	Pronounced gassiness, buttery, slightly less dry than A-D	92	4.82	1.89
F	-6.5	20.3	Definite gassiness, slightly less dry than A-D	99	4.63	1.77
G	-6.8	19.8	Definite gassiness, slightly less dry than A-D	95	4.73	1.83
H	-6.6	20.1	Definite gassiness, slightly less dry than A-D	96	4.70	1.80
I	-5.6	22.0	Slightly soggy and wet, smooth	86	4.75	1.84

The very desirable, firm, dry body and texture of the whey ice creams may be related to the properties of the whey proteins. Ice creams containing whey solids have superior body and texture because of excellent water-binding and emulsification properties of the whey proteins (Alesch, 1958). Tobias (1970) and Rosenberger and Nielsen (1955) also mentioned the dry consistency of whey ice creams, but gave no explanation for this phenomenon.

McDonough and co-workers (1974) noted that whey proteins can trap water in a gel-like, filamentous network, even when heat-denatured. Perhaps this, then, is a partial explanation for the dry appearance of the whey ice creams as compared to the non-whey control.

Overrun. Calculated percent overrun shows some variation from the desired 90% in each ice cream. In constructing a table of ice cream weights required to give various amounts of overrun, an average weight of the nine mixes was used in the calculations. Because of small differences in the actual weights of the nine ice creams, use of an average value for the mix weight was not strictly correct, and variations in overrun were probably related to this approximation. Also, because of differences in mix viscosity, the freezer air control valve was frequently adjusted to give 90% overrun, and the ice cream weight recorded for each batch may not have reflected the fully adjusted overrun.

Weight per Gallon. Both the present and proposed standards of identity for ice cream specify minimum weight and food solids per gallon of ice cream of 4.5 and 1.6 lb/gal, respectively. Weight per gallon is an indirect measure of the amount of air incorporation, and so is

important not only in meeting legal requirements, but in maintaining uniform quality and profits (Arbuckle, 1977, p. 41). Table 14 shows that all of the ice creams met the legal requirement for these criteria.

Quality Characteristics of Finished Ice Cream

Melt-Down. When melted, ice cream should look like the original mix, with no whey separation, foaminess, or curdiness (Arbuckle, 1977, pp. 330-331). Curdy melt-down and whey leakage are due to protein destabilization, and can be avoided, in partm by preserving the natural salt balance of the mix and by use of the proper stabilizer. Slow melting usually indicates overstabilization.

Table 15 shows that the appearance of the melted whey ice creams was less desirable when the control, because of a tendency toward curdiness and whey-off. Ice creams E-H, which had been treated with $Ca(OH)_2$, had a slightly better appearance than the KOH-neutralized ice creams A-D. Prevention of overneutralization and overstabilization, and use of a better combination of stabilizing and emulsifying ingredients, might have eliminated this problem.

As shown in Table 15, the volume of melt-down recorded in 5-minute intervals was similar for all of the ice creams. Whey ice creams E-H, neutralized with $Ca(OH)_2$, melted slightly faster than A-D, in which KOH was the whey neutralizer. This trend is verified by the data on percent melted ice cream in 45 min. The control ice cream (I) melted at an intermediate rate, as compared to the whey ice creams.

Dahle and Rivers (1940) recorded undesirable melting properties in ice creams standardized with both $Ca(OH)_2$ and NaOH. Neutralized ice

Table 15. Melt-down characteristics of ice cream at room temperature (23 C).

Ice Cream	Volume Melted Ice Cream in 5-Min Intervals (ml/50±6g)									Melt-Down in 45 Min (%)	Melt-Down Appearance
	5	10	15	20	25	30	35	40	45		
A	0	<1	<1	2	5	9	14.5	21.5	28	54.0	Slight whey-off, slightly curdy
B	<1	<1	<1	2.5	5	9	14.5	20	25.5	59.9	Very little whey-off or curdiness
C	0	0	0	<1	3	6.5	12.5	20	29	47.7	Slight whey-off, slightly curdy
D	0	0	<1	1.5	4	8.5	15	23	31	57.8	Slight whey-off, slightly curdy
E	0	0	<1	3	7	12	18	29	38	75.8	Fairly uniform
F	0	<1	<1	2	6	12	20	30	39	76.6	Slight whey-off
G	0	0	<1	2	5	11	18.5	28	37	73.6	Slight whey-off
H	0	<1	<1	2	5	10.5	20	29	38.5	76.0	Very little whey-off
I	0	0	<1	2	5	10.5	18	26	34	65.3	Uniform

creams wheyed off and had an increased tendency toward protein destabilization upon melting.

Loewenstein and associates (1975) noted variable results with regard to melting resistance when KOH-neutralized whey concentrates were used to replace portions of NFDM in ice cream. In some mixes, melting resistance was improved by the addition of whey concentrates, while in others melting was faster. Melt-down appearance was not recorded. It should be noted that precipitated protein in the neutralized, concentrated whey was removed before incorporating the concentrate in mixes.

Sensory Quality and Storage Stability. Tables 16 and 17 show results of ice cream evaluation by an expert panel, at one-month intervals, for flavor, body and texture, color, and shrinkage. The American Dairy Science Association Product Judging Scorecard was used for scoring, in which the perfect scores for flavor, body and texture, and color were 10, 5, and 5, respectively. Melt-down was checked only once during the storage interval for the ice creams held at 0 ± 2 F (-17.8 ± 1.1 C).

The most common flavor defects were cooked and caramelized flavors, almost certainly caused by overheating the mixes in the vat pasteurization method. In addition, the mixes containing $Ca(OH)_2$-neutralized whey consistently had a chalky, "fuzzy" mouthfeel, which was especially objectionable in ice cream E. A sweeter taste was noticed and recorded occasionally, probably due to the greater degree of hydrolysis and resulting increase in relative sweetness in some samples.

Table 16. Expert panel criticisms and amount of shrinkage in cartons of ice cream examined at monthly intervals for five months.

Ice Cream	Flavor		Body and Texture		Color	Shrinkage	
	1 Week	4 Weeks	1 Week	4 Weeks		1 Week	4 Weeks
A	Sl. caramel	Sl. caramel	No criticism	No criticism	No criticism	--	None
B	Sl. caramel Sl. sweeter	Sl. caramel Sl. sweeter	Sl. weak	Sl. weak Sl. coarse	No criticism	--	None
C	Sl. caramel	Sl. caramel	No criticism	Sl. coarse	No criticism	--	1 mm edges None top
D	Sl. caramel	Sl. caramel	No criticism	Sl. coarse	No criticism	--	None
E	Caramelized Sl. chalky	Caramelized Chalky Lacks flavor	Crumbly	Coarse Buttery Sl. crumbly	More intense No criticism	--	<1 mm edges None top
F	Sl. caramel Sl. chalky	Caramelized Chalky Lacks flavor	No criticism	Coarse Buttery Sl. crumbly Sl. weak	More intense No criticism	--	<1 mm edges None top
G	Caramelized Sl. chalky	Sl. caramel	Sl. coarse	Sl. coarse	More intense No criticism	--	<1 mm edges None top
H	Sl. caramel Sl. sweeter	Caramelized Sl. sweeter	Gummy Sl. coarse Sl. buttery	Buttery Icy	More intense No criticism	--	<1 mm edges None top
I	Sl. caramel	Sl. cooked	Sl. coarse	No criticism	No criticism	--	None

Table 16, Continued.

Ice Cream	Flavor		Body and Texture		Color	Shrinkage	
	9 Weeks	14 Weeks	9 Weeks	14 Weeks		9 Weeks	14 Weeks
A	Sl. cooked Sl. caramel	Sl. caramel Sl. cooked	Sl. coarse	Sl. sandy? Sl. coarse	No criticism	None edges Slight top	1 mm edges 3 mm top
B	Sl. caramel Sl. cooked	Sl. cooked Sl. sweeter	Sl. coarse	Sl. coarse	No criticism	1 mm edges Slight top	2 mm edges 3 mm top
C	Sl. caramel	Sl. cooked Sl. caramel	Sl. coarse	Sl. coarse	No criticism	3 mm edges Slight top	3 mm edges 6 mm top
D	Sl. caramel	Sl. cooked Sl. caramel	Sl. weak	Sl. weak Sl. coarse	No criticism	Slight edges None top	1 mm edges 3 mm top
E	Sl. caramel Chalky	Sl. caramel Sl. cooked Chalky	Sl. coarse Gummy	Buttery Coarse Sl. weak	More intense No criticism	2 mm edges Slight top	3 mm edges 7 mm edges
F	Sl. caramel Sl. chalky	Sl. caramel Sl. cooked Chalky	Weak	Buttery Coarse Sl. weak	More intense No criticism	4 mm edges Pronounced top	4 mm edges 15 mm top
G	Sl. caramel Chalky	Sl. caramel Sl. cooked	Weak Buttery Sl. coarse	Sl. buttery Sl. weak Sl. coarse	More intense No criticism	1 mm edges Slight top	3 mm edges 6 mm top
H	Caramelized Chalky	Sl. caramel Sl. cooked	Icy Weak	Coarse Weak, Buttery	More intense No criticism	1 mm edges Slight top	2 mm edges 4 mm top
I	Sl. cooked	Sl. cooked	Sl. coarse	Sl. coarse	No criticism	1 mm edges None top	2 mm edges 3 mm top

Table 16, Continued.

Ice Cream	Flavor		Body and Texture		Color	Shrinkage	
	17 Weeks	22 Weeks	17 Weeks	22 Weeks		17 Weeks	22 Weeks
A	Sl. cooked Sl. caramel	Sl. caramel Sl. cooked	Sl. coarse	Coarse	No criticism	2 mm edges 4 mm top	2 mm edges 8 mm top
B	Sl. caramel Chalky	Sl. caramel Sl. cooked	Coarse	Coarse	No criticism	3 mm edges 14 mm top	3 mm edges 18 mm top
C	Sl. caramel	Sl. caramel Sl. cooked	Sl. coarse	Sl. sandy Sl. coarse	No criticism	4 mm edges 15 mm top	4 mm edges 22 mm top
D	Sl. caramel Chalky	Sl. caramel Sl. cooked	Sl. coarse	Coarse	No criticism	2 mm edges 8 mm top	5 mm edges 9 mm top
E	Sl. caramel Chalky	Chalky	Buttery Coarse	Sandy Coarse	More intense No criticism	4 mm edges 15 mm top	5 mm edges 24 mm top
F	Sl. chalky Sl. caramel	Chalky	Buttery Weak V. coarse	Sandy	More intense No criticism	4 mm edges 32 mm top	6 mm edges 45 mm top
G	Sl. caramel Sl. chalky	Chalky	Buttery Coarse	Sandy	More intense No criticism	4 mm edges 13 mm top	5 mm edges 20 mm top
H	Chalky Sl. caramel	Chalky	Sl. sandy? Sl. weak	Sandy	More intense No criticism	4 mm edges 15 mm top	6 mm edges 30 mm top
I	Sl. cooked	Sl. cooked	Sl. coarse	Coarse	No criticism	2 mm edges 7 mm top	4 mm edges 10 mm top

Table 17. Flavor, body and texture, and color scores of ice cream examined at monthly intervals by expert panel using American Dairy Science Association Project Judging Scorecard.

Ice Cream	Flavor Scores (Week after Freezing)						Body and Texture Scores (Week after Freezing)						Color Scores All Evaluations
	1	4	9	14	17	22	1	4	9	14	17	22	
A	9	9	8.5	9	9	9	5	5	4.5	4	4	3.5	5
B	9	9	8.5	9.5	8.5	9	4.5	4	4.5	4.5	3	3.5	5
C	9	9	8.5	9	9	9	5	4.5	5	4.5	4	3	5
D	9	9	8.5	9	8.5	9	5	4	4	4	3.5	3.5	5
E	8.5	8	8	8	7	8	3.5	3	3	3	2.5	2	5
F	9	8	8.5	8	7.5	8	5	3	3.5	3	2.5	2	5
G	8.5	8.5	8	8	7.5	8	4.5	4.5	3	3.5	2.5	2	5
H	9	8	8	8	7.5	8	3.5	2.5	2.5	2.5	2	2	5
I	9	9.5	9	9.5	9	9	4.5	5	4	4.5	4	3	5

The increase in relative sweetness as a result of lactose hydrolysis has been quantified by using taste panels. Bouvy (1974) reported that 80% conversion to glucose and galactose was equated to 7 g/l sucrose in yogurt. In other words, 80% conversion was equal to 0.7% sucrose. Guy et al. (1974) found a linear relationship between percent hydrolysis and percent sucrose, where 30% and 90% conversion equaled 0.3% and 0.9% sucrose, respectively.

The most commonly encountered body and texture problem was coarseness, which developed into an icy criticism later in the storage period in some samples. The ice cream containing whey neutralized with KOH had a noticeably smoother body than the control ice cream. This quality may have been related to the superior firm and dry appearance of these samples at the freezer.

A slight degree of sandiness was noted in sample H after 17 weeks of storage. After 22 weeks (5 months), all of the $Ca(OH)_2$-neutralized ice creams (E-H) were sandy, although the crystals were relatively small. One of the KOH-neutralized samples (C) was slightly sandy as well. The control ice cream (I) and KOH samples A, B, and D were coarse, but not sandy.

Color remained normal throughout the storage period in all samples. Mixes E-H [$Ca(OH)_2$] were noticeably more intensely yellow than the other samples, and tended to appear slightly brownish later in the storage period. This color was not considered objectionable, however, and the samples were not downgraded on this basis.

Variable results have been previously reported on sensory acceptability of whey-containing ice creams and sherbets.

Potter and Williams (1949) reported that, when good quality whey was used in sherbets, the flavor was clean and refreshing. The body and texture were extremely smooth.

Drawbridge (1951, pp. 43-44) noted that use of calcium oxide as an acid standardizer produced a pronounced neutralizer flavor in ice cream, the intensity of which increased upon storage. These samples had desirable body and texture, but poor melting quality. The ice cream melted in chunks, curdled, and wheyed off. No shrinkage was noted in four weeks of storage in a commercial-type freezer.

Arbuckle and Singh (1971) reported very favorable storage stability in terms of flavor, body and texture, and appearance for acid whey-containing frozen desserts. Problems with off-flavors during storage were avoided by the use of acid-modifying salts.

Igoe et al. (1973) found that use of low levels of KOH-neutralized acid and sweet wheys produced ice cream with sensory qualities nearly identical to regular ice cream.

Guy et al. (1974) found that ice creams containing lactose-reduced sweet whey and KOH-neutralized acid whey were more stable than the control, to flavor and texture changes during storage.

Loewenstein and co-workers (1975) noted reduced flavor and body-texture scores for ice creams containing KOH-neutralized acid whey concentrates as compared to the control, at a level of more than 20% replacement of non-fat dry milk.

Arnold et al. (1976) found that sweet whey-containing ice creams held at -10 F did not change significantly in terms of texture over 12 weeks of storage. At +10 F, statistically significant reduction in

texture scores occurred at 4 weeks, probably due to high, fluctuating temperatures, larger ice crystal formation, and lactose crystallization. At both temperatures, flavor scores were significantly lower at 8-12 weeks than initially.

The smooth texture of the whey ice creams could be related to freezing point depression as a result of lactose hydrolysis, and altered ratios of protein to lactose and minerals. The freezing points of the whey mixes are probably lower than that of the standard formulation because of the differences in amounts of minerals, lactose, glucose, and galactose.

A lower freezing point means that less water is frozen at a given temperature (Cosgrove, 1974; Brown and Gibson, 1955). These mixes thus have a better texture because of less extensive freezing of the mix water (Brown and Gibson, 1955). However, since less water is frozen at the freezer, more is frozen upon hardening, and this could lead to coarser texture and less heat-shock resistance (Cosgrove, 1974).

The use of corn syrup sweetener solids and effective stabilizer-emulsifier combinations have been shown to contribute to desirable body and texture, and to storage stability (Igoe et al., 1973). Calcium and sodium acid standardizers had no effect on the rate of lactose crystallization in stored ice cream (Whittier, 1933). Body and texture of stored ice cream is influenced by 1) freezing temperature, 2) storage time, 3) storage temperature, and 4) severity of heat shocking, in descending order of importance (Frazeur and Harrington, 1968).

Shrinkage. The whey ice creams were subject to shrinkage, as shown in Table 16. Arbuckle (1977, pp. 333-334) discussed the inconsistent nature of this defect. Among the possible causes, the ones most likely at fault in the present study include neutralization of mix ingredients, excessively smooth texture (implying small ice crystals and air cells) as a result of the emulsifiers used, partially destabilized protein, and weak body as a result of excessive overrun.

Table 16 shows that shrinkage was first noted in all samples after either 4 or 9 weeks of storage at 0 ± 2 F (-17.8 ± 1.1 C). Arbuckle's suggested causes for this defect can be applied to sample F. The table shows that this sample underwent the largest amount of shrinkage, and was often criticized as having a weak body. Table 13 shows that sample F had low relative protein stability as well. Table 14 indicates that the highest overrun of the nine ice creams was recorded for sample F.

It should be noted that these storage studies were more lengthy than would likely be encountered in commercial practice. Reid and Shaffer (1947) reported, in a survey of manufacturers, that ice cream is normally held in the hardening room from 12 hr to 4 days, and is usually purchased within a week. Although the ice creams examined in the present study were held in a commercial-type freezer, they were not subjected to heat shocking typical of retail and home handling.

Sensory Evaluation of Ice Creams

The nine ice cream samples were examined by twenty untrained panelists (nine female and eleven male) to determine their preferences.

Only 10% of the panelists reported tobacco use. Their reported frequency of ice cream consumption was: 25% regularly and 75% occasionally.

Rating scores were made in triplicate on each of two days, and results were analyzed using theory of signal detection (TSD) methodology, as detailed in Materials and Methods. Through use of TSD, an observer's decision for one sample can be evaluated in relation to his conclusions for other samples, and this gives information about his judgment criteria (Angus and Daniel, 1974; Stull et al., 1974). Thus, the effect of differences between groups of people in terms of their perception and individual scoring criteria can be "normalized" by the statistics. TSD also allows examination of numerous samples at one sitting, whereas more traditional methods would require many more sample presentations to yield comparable information.

In the TSD analysis for the current study, the standard by which all the ice creams were compared was the control formulation, Mix I. Based on all the rating responses for each mix, computations were performed to relate each ice cream to the standard, which was definitely preferred by the panel. The distance metric (d_m) is a measure of the degree of discrimination and preference between the control and experimental samples (Angus and Daniel, 1974; Stull et al., 1974). The d_m values in this case are negative numbers; the higher the d_m, the higher the preference for that sample. In absolute terms, a higher d_m indicates better ability of the observer to distinguish between the control and the experimental sample (Angus and Daniel, 1974; Stull et al., 1974).

Table 18 shows the preferences for the eight whey ice creams as a function of pH, percent hydrolysis, and neutralizer. The d_m values in this table indicate that the most preferred ice cream contained whey which was neutralized to pH 6.8 with KOH, and contained 50% hydrolyzed lactose (Mix C). All four of the KOH-standardized mixes (A-D) were preferred to the $Ca(OH)_2$-neutralized samples (E-H). The least preferred ice cream was sample E, containing whey neutralized with $Ca(OH)_2$ to pH 6.5 and 50% lactose hydrolysis.

Because TSD computations were used, it is possible to segregate the effects of pH, neutralizer, and hydrolysis, and to correlate any of these with replication, day, or observer interactions. Thus, a factorial design analysis of variance was used to analyze significance of interactions.

Table 18. Mean d_m ratings of whey ice creams for different neutralizers, whey pH, and lactose hydrolysis levels.

		Lactose Hydrolysis (%)	
	pH	50	75
KOH	6.5	-43	-43
	6.8	-20	-29
$Ca(OH)_2$	6.5	-117	-64
	6.8	-54	-102

The ANOVA results indicated a highly significant ($P < .001$) three-way interaction between hydrolysis level, pH, and neutralizer. When days of panel testing were included with these three parameters, there was no statistically significant interaction. However, there was a low level of significance ($P < .025$) when replications were correlated with neutralizer, pH, and hydrolysis level.

Conclusions to be drawn from this information are more easily examined with the aid of Figures 4 and 5. The higher numerical d_m values for KOH as compared to those for $Ca(OH)_2$ show that KOH was the preferred neutralizer. With KOH, pH 6.8 was slightly preferred to 6.5, regardless of the degree of hydrolysis (Figure 4). At pH 6.5, level of hydrolysis has no effect on preference.

With $Ca(OH)_2$ as the neutralizer, the picture is a little more complex, as the effects of pH and degree of hydrolysis are mutually important. At pH 6.5, 75% hydrolysis was preferred, while at pH 6.8, preference was for the lower hydrolysis level. Figure 5 clearly shows this cross-over of preference as a function of degree of hydrolysis.

The significant interactions of neutralizer, hydrolysis, and pH with replications is easily examined by calculating the mean of all observations on replications 1, 2, and 3. These are, respectively, 6.29, 5.90, and 6.04, which suggests the possibility of fatigue, boredom, or satiety on the part of the panelists as the number of samples tasted increased.

It probably would have been preferable to replace the three replications of the nine ice creams in a single day with a less tedious

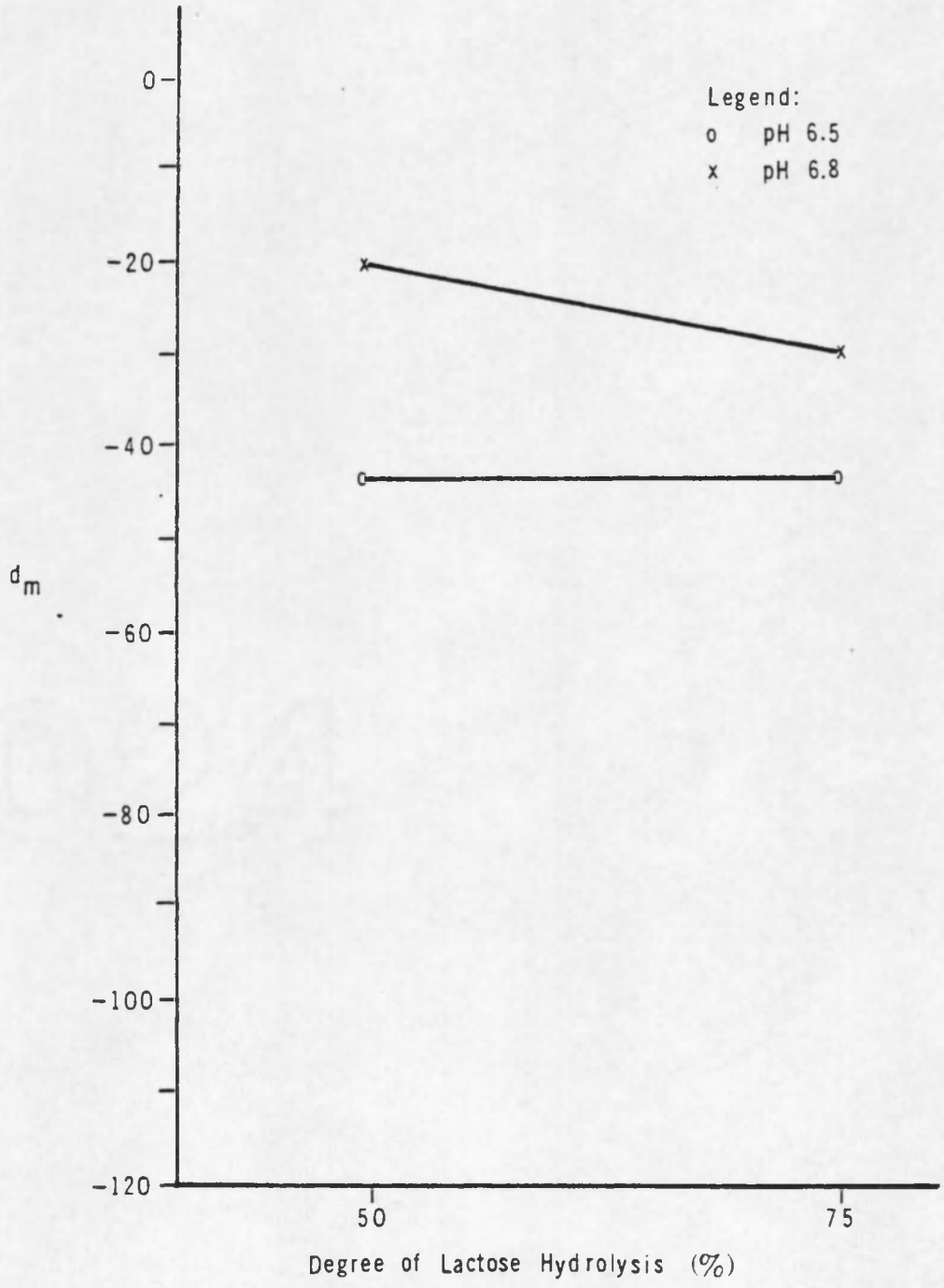

Figure 4. Results of TSD analysis (d_m values) of ice creams containing whey neutralized with KOH, as a function of pH and degree of hydrolysis.

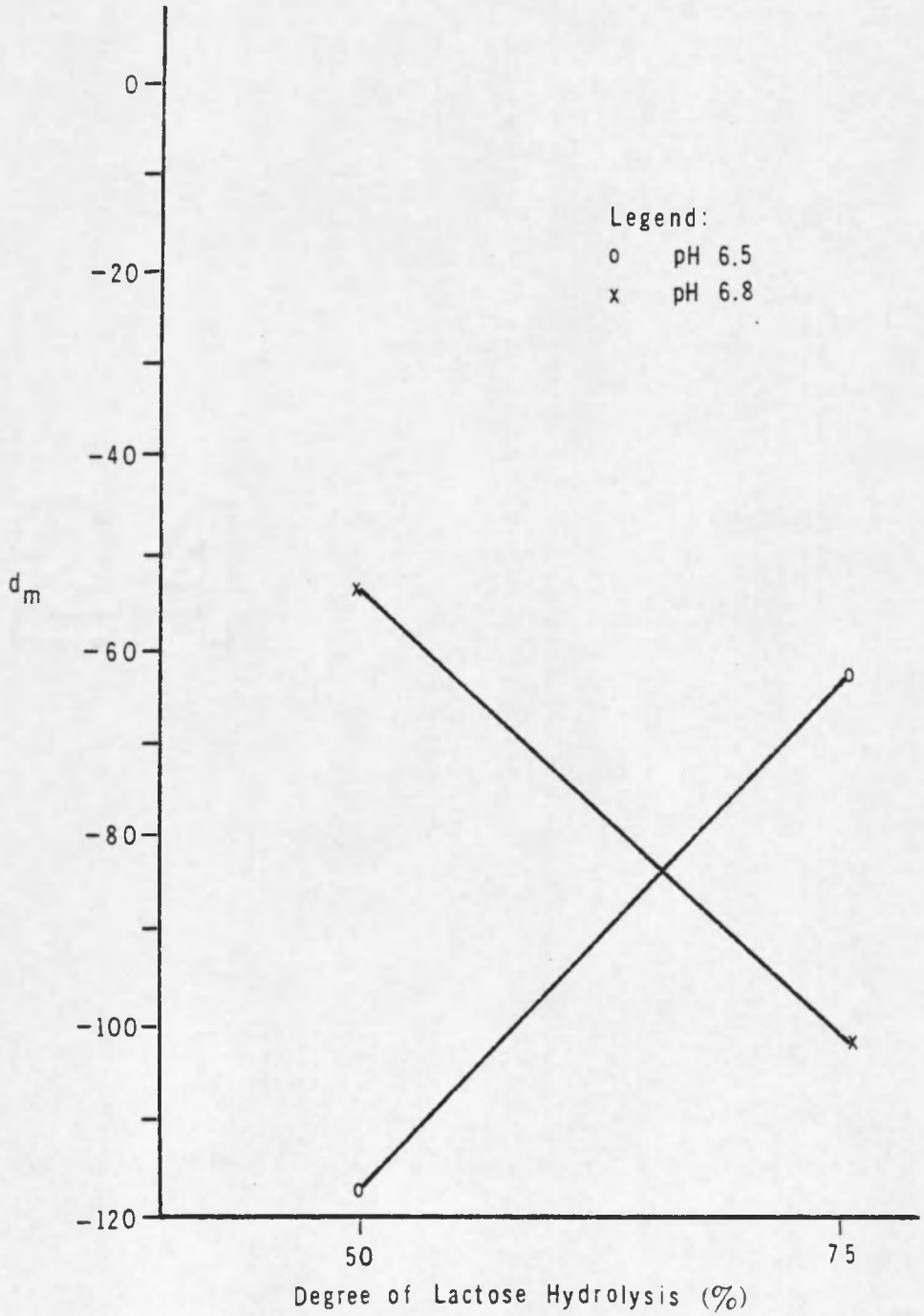

Figure 5. Results of TSD analysis (d_m values) for ice creams containing whey neutralized with $Ca(OH)_2$, as a function of pH and degree of hydrolysis.

experimental design. This procedure was chosen because of the simultaneous use of ranking criteria in another comparison (see Materials and Methods). Probably, a single rating series performed on each of three days would have been a better plan than the one actually used, from the standpoint of reproducibility and enjoyment by the panel.

Because the panelists were required to taste and swallow 27 samples of a rich dessert, their accuracy of replication might have been expected to be less than optimum. However, neither day-to-day nor replication-to-replication vairance was statistically significant, corroborating the work of Stull et al. (1974), in which a naive panel reproducibly discriminated 27 different ice creams.

TSD is an extremely valuable tool in sensory testing because more meaning can be interpreted from the results than with traditional methods. Without this computational method, little more than a raw mean and standard deviation could have been calculated for each ice cream, plus an analysis of variance among the means. With TSD, however, the computations minimize individual perceptual biases and give more reliable results.

CONCLUSIONS

General Conclusions

The goal of this investigation was to develop and test the acceptability of an ice cream-type frozen dairy dessert which contains fluid cottage cheese whey as the major bulk-supplying ingredient. It was hoped that the procedures could be profitably used by firms producing a relatively small amount of whey.

Based on chemical, physical, and sensory considerations, the products which results from this study could be considered acceptable. On a pilot-plant scale with a limited number of trials, it appeared that no unusual processing problems should arise. Relatively minor problems with shrinkage and melt-down, for example, could probably be corrected with refinements in stabilizer-emulsifier selection and use.

Commercial applicability of the products described in this thesis are, as yet, questionable because of 1) the present regulatory status of whey, neutralization, and lactose use in ice cream; and 2) the economic feasibility of using lactase enzyme preparations of various sources and forms.

Specific Conclusions

1. All whey ice creams were within legal requirements for ingredients, fat, weight per gallon, food solids per gallon, and protein, based on the proposed new standards of identity. They met

all of the guidelines in the current (1960) standards (fat, MSNF, total solids, total solids per gallon, and weight per gallon), except for the use of neutralized, hydrolyzed cottage cheese whey.

2. KOH is a better choice as a lactic acid neutralizer than is $Ca(OH)_2$, based on nearly every chemical, physical, and sensory consideration, ease of use, and predictability of action.
3. Based on evaluation of taste, a whey pH of 6.8 is slightly better than 6.5 when KOH is the neutralizer. With $Ca(OH)_2$, the preferred whey pH is dependent upon degree of lactose hydrolysis.
4. Extent of lactose hydrolysis plays little role in acceptability of KOH-neutralized mixes, judging from taste panel data. In $Ca(OH)_2$-neutralized mixes, on the other hand, a higher level of hydrolysis is preferred at the lower whey pH.
5. Ice creams made with fluid whey contain only slightly less protein than conventional ice creams.
6. Cooked and caramelized flavors might be corrected by using high temperature-short time, rather than batch pasteurization.
7. The amount of added sweetener could be reduced because of the increase in sweetness following lactose hydrolysis.
8. Undesirable melt-downs and problems with shrinkage in the whey ice creams might be corrected by adjusting the amount and types of stabilizing and emulsifying ingredients.
9. The whey ice creams were free of sandiness for four months in a commercial-type freezer (0 ± 2 F; -17.8 ± 1.1 C).

10. The high cost of lactase enzyme would probably demand the use of an immobilized enzyme system in order for this method to be economically feasible.

APPENDIX

CONSUMER PREFERENCE TEST

Introduction

Following the completion of the research required for this thesis, the opportunity arose to test the acceptability of the whey ice cream utilizing a large consumer panel. Not only would such a test result in more data, but it would provide an opportunity to make improvements in formulations and procedures, in a significantly larger batch (40 gallons of ice cream) than was possible before. In particular, slight modifications were made in sweetener and stabilizer-emulsifier levels in the experimental mix, and the batch was large enough to use the high temperature-short time pasteurizer.

Materials and Methods

Preparation of Ice Cream

The most acceptable whey formulation in the previous study had proven to be Mix C, containing whey neutralized to pH 6.8 with KOH, and with 50% of the whey lactose hydrolyzed. In the whey mix for the consumer test, the percent stabilizer-emulsifier was reduced to 0.31%, compared to 0.35% used previously. In addition, the sucrose level was reduced by 0.5% to take into account the increase in sweetness from glucose-galactose as a result of 50% lactose hydrolysis (Guy et al.,

1974). The control mix formula was exactly as before. The whey and control mix formulations are given in Tables A1 and A2.

Fresh, fluid cottage cheese whey was allowed to settle overnight in a 150-gal, ice water-jacketed vat to separate the casein fines. The next morning, the clarified whey was siphoned from the top of the vat, as before. Twenty-four gallons (204 lb; 92.5 kg) of clarified whey were then neutralized to pH 6.8 with 30% KOH solution (4,216 ppm KOH, or 2,934 ppm K^+, required). In the previous work, the whey had been neutralized to pH 7.0 rather than 6.8.

The neutralized whey was heated to 33 C (91.4 F) and 600 mg/l Maxilact lactase was added. Degree of hydrolysis was followed organoleptically, due to the non-availability of Glucostat Reagent. In this technique, when the sweetness of the whey was judged to be equal to that of a 0.5% sucrose-in-whey solution, the whey lactose was assumed to be 50% hydrolyzed (Guy et al., 1974). No verification of this judgment was made experimentally. Hydrolysis required about 1 1/4 hour.

The whey was then heated to 180 F (82 C) to inactivate the enzyme. Unfortunately, the desired temperature of inactivation (160 F; 71 C) was accidentally exceeded. The whey was cooled by adding ice water to the vat jacket. Cooled whey was immediately incorporated into the ice cream mix.

Both mixes were homogenized at 160 F (71.1 C) with a Cherry-Burrell, two-stage homogenizer (2,000 and 500 lb/sq in. pressure on first and second stages, respectively). They were then pasteurized in a Creamery Package, high temperature-short time pasteurizer (185 F for

Table A1. Comparison of formulations used in calculating whey and control mixes.

Formula	Whey Mix (%)	Control Mix (%)
Fat	10.0	10.0
MSNF	11.0	11.0
Sugar	9.2	9.7
Corn sugar	6.2	6.2
Stabilizer-emulsifier	0.31	0.31

Table A2. Ingredients used in whey and control mixes, expressed as lb per 368 lb batch, and as percent by weight of total composition.

	Whey Mix		Control Mix	
Ingredients	lb	%	lb	%
Cream	102.2	27.8	69.9	18.9
Milk	--	--	220.2	59.8
Whey	184.0	50.0	--	--
NFDM	24.0	6.5	18.6	5.1
Sugar	33.9	9.2	35.7	9.7
Corn sugar	22.8	6.2	22.8	6.2
Stabilizer-emulsifier	1.14	0.31	1.14	0.31
	368.04	100.01	368.04	100.01

25 sec) and immediately cooled below 50 F. The mixes were stored at 33 ± 1 F (0.6 ± 0.6 C) for five days until frozen.

On the day of freezing, two-fold pure vanilla flavoring (Golden-Van No. 180, Petran Products, Milwaukee, Wisconsin) was added according to manufacturer's directions. Only half the recommended amount of cheese coloring (Hansens Standard of the World, Chr. Hansen's Laboratory, Milwaukee, Wisconsin) was used in the whey mix because it was obvious during its addition that the color would be too dark if the full amount were added. This difference in color absorption by the KOH-neutralized whey mix had not been encountered previously. An attempt to match the color in the control mix was unsuccessful, and, as a result, the control was slightly more intensely yellow when frozen. This color difference was barely noticeable, especially under the lighting conditions at the test location.

The ice creams were frozen as before, to a desired overrun of 90%. The appearance of the whey ice cream as drawn from the freezer was again more dry and firm than the control. Samples were drawn into pre-labeled, 3 1/2 oz lidded plastic cups, as before.

Samples were held in the hardening room (-22 ± 2 F; -30 ± 1.1 C) for three weeks until used in the panel test.

Sensory Evaluation

Sensory evaluation was conducted in a large Tucson shopping mall which was completely enclosed and air-conditioned. The preference test exhibit was one of many on display at the Arizona Food Fair, a community

nutrition information program, held in conjunction with National Nutrition Week.

Panelists consisted of shoppers and persons attending the Food Fair. They were instructed as to the purpose of the test by a large sign which read: "University of Arizona; Nutrition and Food Science Department; Frozen Dairy Dessert Test; Taste two vanilla ice creams made with different recipes."

Samples were transported from the hardening room to the shopping mall via dry ice-cooled ice chests. Sample cups were then held in a portable freezer (6 ± 12 F; -14 ± 6.7 C) until served. Accurate control of sample serving temperatures was difficult under the testing conditions.

Panelists were given sample cups containing the two ice creams, a score sheet (Figure A1), and a spoon. They were orally instructed to indicate on the score sheet the sample which was preferred. Because of the large number of persons taking the test at any one time, panelists usually tasted the ice creams on nearby benches. Consequently, little control could be maintained in assuring that consumers made independent choices.

Nearly all of the panelists were adults. About one thousand pairs of ice cream samples were distributed in less than four hours. The preference test was conducted either at late-morning or mid-afternoon sessions on each of two successive days. After participating in the test, panelists were given an information/explanation sheet (Figure A2).

ICE CREAM PREFERENCE TEST

Please circle the symbol which corresponds to the ice cream you like best.

Δ

Comments:

Figure A1. Score sheet used by panelists in consumer preference test.

FROZEN DESSERT PREFERENCE TEST

One of the desserts you have tasted is made from a common recipe. The other sample is made by using an experimental recipe developed in the Food Science program at the University of Arizona. It contains a milk product not normally used in ice creams under current standards.

It is expected that the results of this research will be submitted to a technical journal for publication. Thank you for participating in this test.

Figure A2. Information sheet given to consumer panelists following their participation in the preference test.

Results and Discussion

Chemical and Physical Tests and Scoring

Results of limited chemical physical analyses on the two ice cream mixes are shown in Table A3. This table also includes expert panel flavor, body-texture, and color scores, as judged after ten weeks of storage at 0 ± 2 F (-17.8 ± 1.1 C). The American Dairy Science Association Scorecard was used as before, where perfect scores for these three characteristics were, respectively, 10, 5, and 5.

After ten weeks, the whey ice cream was slightly more coarse in texture than the control. The control ice cream had a slightly more chewy body. The flavor of the control was criticized as being slightly cooked, while the whey ice cream was slightly caramelized. The caramelization had not been noticeable one week after freezing, however, and probably resulted from overheating the hydrolyzed whey during enzyme inactivation. Apparently, the high temperature-short time pasteurization method prevented pronounced caramelization in the whey and control ice creams, a prevalent defect in the previous study.

Melt-down was observed only subjectively. The control melted uniformly, while the whey ice cream wheyed off slightly. The separation in the whey formula was less pronounced than previously, however.

Preference Test Results

A total of 732 score sheets were returned. The tally of preferences was as follows: 370 consumers preferred the whey ice cream; 343 preferred the control ice cream, and 19 were unable to make a decision.

Table A3. Basic composition and expert panel scores for whey and control ice creams used in consumer preference test.

Ice Cream Characteristic	Whey Ice Cream	Control Ice Cream
pH	6.65	6.6
Titratable acidity, as lactic acid (%)	.184	.189
Fat (%)	9.80	10.06
Total solids (%)	36.51	36.61
Moisture (%)	63.50	63.40
SNF (%)	26.34	26.55
MSNF (%)	10.63	10.84
Flavor score	9	9
Body-texture score	5	5
Color score	5	5

The t test was used to judge the significance of the results (ASTM, 1968, p. 52). This calculation resulted in a t value of 0.272, which was not statistically significant.

Therefore, the results of this consumer test indicated that the panel had no clear preference for either the whey or the control ice cream, and any difference in preference between the two samples could have been entirely due to chance.

A tally was made of all comments received on both ice creams. Although some consumers commented that they could not distinguish the two samples, the majority of comments indicated that there was a perceptible difference between the whey and control ice creams.

Accepting the comments at face value, there were only two observations that were significantly different between the two groups. Twenty-three observers felt that the whey ice cream was "more creamy," while only six thought the control sample was more creamy. Similarly, seventeen comments referred to "better flavor" in the control ice cream, compared to seven similar comments for the whey sample. These comments correspond well with the opinions of the expert panel.

No interpretation of the terminology used by the observers was made in tallying these comments. It is recognized that quite different conclusions could be drawn if the comments were re-categorized using conventional terminology. For example, "more creamy" could be interpreted as meaning "smoother" or "better texture." Likewise, "better flavor" might refer to differences in sweetness, freshness, or amount of vanilla flavoring.

Conclusions

The goal of the experiment reported in this appendix was to make formulation and processing improvements on an ice cream containing fluid cottage cheese whey neutralized to pH 6.8 with KOH, and with 50% of the whey lactose hydrolyzed. Its acceptability was then tested against a control ice cream of standard formulation, using a consumer panel.

The following conclusions were reached:

1. The whey ice cream was evaluated by an expert panel as being nearly identical to the control in flavor, body-texture, and color.
2. Caramelized flavors were virtually eliminated by use of high temperature-short time pasteurization.
3. The amount of stabilizer-emulsifier in a whey mix probably need not be in excess of the amount added to a standard mix.
4. The amount of sweetening ingredients in the formula can be reduced in mixes containing hydrolyzed lactose, according to guidelines by Guy et al. (1974).
5. A consumer taste panel of 732 persons liked the standard and whey ice creams equally well. The whey ice cream was often described as being "more creamy," while the standard product was characterized as having "better flavor."

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