T H E S I S

on

A Study of Calcium Compounds for Cream Neutralization.

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1. Introduction.

A large part of the cream which is received from the twenty five thousand Oregon Creamery patrons comes to the factory in a sour condition.

The problem of this sour cream is to make from it a butter of the best possible flavor, aroma, and keeping quality. Butter from medium-acid cream has been repeatedly shown to keep better than that produced from high acid cream. Hence the problem of reducing the excess acidity by a satisfactory method and "neutralizer," before pasteurization.

It is the somewhat over-ripened cream which comes from small or medium sized dairy herds and which makes up a large part of the cream received by Oregon creameries which must be "neutralized". It is very often not possible or economical because of high transportation costs to ship the cream to the creamery more often than from once to three times a week.

Many farmers do not have facilities for properly cooling and storing the cream and the growth of the lactic-acid-producing bacteria is optimum at blood heat, and they do grow well (particularly in milk) at room temperature. Milk comes from the cow at blood heat of from 101.4-102.8 deg. Fah. and often is not cooled below 70 deg. Fah. for several hours during which time lactic acid is produced, and such cream must go on the market as a second grade product.

The committee on the use of alkalis in cream neutralization consisting of Frandsen, Mortensen, Hines, and Bouska, reported at the annual American Dairy Science meetings in Chicago, 1912 (1) that "whenever we have worked out a practical and satisfactory scheme for grading of cream, and when such a system is followed up by the creameries with a common-sense plan for recognizing the difference in grades with a substantial difference in price, much will have been done toward interesting the farmer in the production of a better grade of cream."

"A cream which is absolutely clean in flavor and sweet does not need to be neutralized, and does not require the addition of any foreign substance in order to manufacture, therefrom, by proper ripening afterward, by a first class starter the best butter that it is possible to obtain from such cream."

It is needless to offer proof to a statement that many farmers now have facilities for producing a better quality of cream than they are producing, if a premium were paid for the extra care required.

In the same reference one member of the committee expressed it: "Logically, dirt should be kept out of milk. It is, however, a common practice to remove it by straining or centrifuging. Milk and cream should be produced germ free, but in practice the excessive number of bacteria, are destroyed by pasteurization. Removing the excess acidity

by neutralization is also not ideal, but practically analogous."

The committee states: "The majority of this cream, when its acidity is properly reduced, makes at least a fair butter, which seems to satisfy the trade. If under present conditions this method of treatment were not used it would mean the impossibility of making, profitably, millions of pounds of butter, and would shut off the dairy revenue of countless thousands of western farms."

Some of the defects listed in butter score cards which are due to neutralizers or lack of neutralizer are according to Hunziker (2) sour, curdy and cheesy flavor and aroma, due to butter being made from high-acid cream, oily flavor, due to pasteurizing sour cream at high temperature and cooling it over a surface cooler, metallic flavor may be due to metallic salts in impure lime neutralizers, Neutralization flavor is the presence of an unnatural alkaline condition of the butter.

A survey made by Larrabee and Wilster (3) shows that the greater portion of the butter made in Oregon (1928) was scored 90.0 to 91.0, a smaller portion was scored 89.0 to 90.0, and yet a smaller portion scored 91 and above. Wilster (4) shows that in 1932-33 a greater percentage scored above 92, demonstrating that the quality of butter

has improved.

Cream shipped through a gathering station is usually sour when it reaches the creamery. In the U.S. Dept. of Commerce, fifteenth census of the United States (5) Table I shows that fourteen percent, or two and three fourths million pounds of Oregon's cream is received by creameries through shipping stations. In the United States thirty three percent, or 381.5 million pounds, is received through shipping stations. One can readily see that a large portion of our cream must be neutralized in order that the best possible quality of butter may be manufactured from it.

The average score of Oregon's butter was approximately 90.5 in 1931 (6). The production of butter in Oregon in 1931 was 28,621,252 pounds. (4) "The average price in the San Francisco market, (U.S.D.A. Rep't, 1930) for 90 score was 3,04 cents higher than the average price butter." Raising the score of the lower 50% of the butter to 92 would increase the income of the creameries $435,000".

Wilster, Annual Butter Scoring Report(4) 1933, states "The difference between the price of 90 and 92 score butter at San Francisco, during 1932 was 1.6 cents. Eighty-eight to eighty-nine score butter was not quoted on account of the small demand for this quality butter in that market. Usually the difference between this grade of butter

and that which scores 90 is one cent. Therefore if the prices for these grades ranges from 19.4 to 22 cents a pound, a total of $88,000 more would have been realized for the butter made by the creameries that sent butter to the scorings, if it were sold in accordance with the scores obtained during the last year, (fourth year) than could have been obtained if it had been sold in accordance with the scores obtained during the first year."

Those figures are for the 75 percent of Oregon's butter manufactured by the creameries participating in the monthly scorings.

Frandsen, Mortensen, Hines, and Bouska, The committee on the use of alkalies in cream neutralization report previously referred to , received the following answers to a questionaire sent to leading dairymen and creamerymen. (1), Question 4: To what extent do you believe it possible to improve the quality of butter from bad cream by the use of alkali? was answered as follows by six of those questioned: Ans. (1). "Proper neutralization when used in combination with proper methods of pasteurization and starter, the quality of butter may improve 4 or 5 points." (2). "If really poor cream is used, quality may improve from one to three percent. In clean flavored cream no improvement." (3)."Two points." (4) "Don't know." (5) "At least one grade." (6) "One to three points- the latter only in case of storage." (Just the reverse).

These answers by many of the leading creamerymen in college and practical work help point out the improvement which may be expected when proper "neutralization" is practiced, as long as sour cream is being delivered to the plants.

1. Review of Literature.

Flint (7) in 1858, found that by adding soda water to sour milk he was able to make better butter.

Dean (8) in 1913, found that by adding milk of lime after pasteurization a higher scoring butter was obtained than when adding it before pasteurization. He recommends the use of concentrated lime rather than lime water.

Giltner and Brown (9) 1917, state that "Butter from neutralized cream, although not showing uniformily greater changes, is less desirable for storage."

Frandsen, Mortensen, Hines, and Bouska (1), in 1912 report to the Dairy Instructors Association that lime was considered by many authorities to be more desirable and more widely used than any other cream neutralizer.

O'Callaghan and Ramsay (10) in Australia, in 1918, report that cream neutralized with slaked lime increased in viscosity regardless of its age, acidity or fat content, but decreased in viscosity after pasteurization. Sodium compounds which were used in previous studies decreased the viscosity of the cream.

Mac Iunes (11) 1931 reports success in making butter from high acid cream neutralized with equal parts of lime and sodium bi-carbonate followed by pasteurizing. Both the holding and flash methods were used. With the former, the viscosity of the cream was found to be less than when lime alone was used. With the latter, there was less foaming than when soda was used alone. Sproule and Grimes (12), 1923, favored the butter made from partially neutralized cream.

Jackson (13) 1923, used Ca O, Mg O, Na_2 CO_3 and NaOH as neutralizers in preliminary trials but decided that lime was the most satisfactory neutralizer to use. He found that butter made from neutralized and pasteurized cream had a better flavor than butter made from unneutraliz - ed, raw or pasteurized cream. However, there was very little difference in the keeping quality.

Mortensen of Iowa State College, 1924, (14), states that, "Conclusive proofs have been presented by this station and other stations to the effect that sour neutralized cream of poor quality is improved by neutralization." His results show 1.2 increase in score of the neutralized over the unneutralized cream butter.

Stiritz and Ruehe (15), 1925, made a thorough investigation of various neutralizing agents from several different angles. They concluded that none of the

neutralizers used (limes, soda ash and sodium bicarbonate) could be said to produce the best quality butter. Their conclusions seem to indicate, however, that lime is slightly superior to soda ash and sodium bicarbonate. They did not find any advantage in double neutralization.

Hunziker (2), 1925, has found that on a commercial scale he was unable to make better butter with soda neutralizers than with lime neutralizers.

Walts and Libbert (16) in 1930 in their study of "the effect of various neutralizers on the flavor of butter", studied eleven compounds, 5 lime and 6 soda, with reference to chemical composition, solubility, and alkalinity, time required for reduction of acid in the cream, acidity reduction due to pasturization and influence on the flavor and keeping qualities of butter. They state that: "All of the limes contained considerable impurities chief of which was clay, which was composed chiefly of metallic oxides and silica. The metallic oxides consisted chiefly of iron and aluminum. Iron is a strong catalyst and causes oxidation of the butter fat along with its attendant evils. It also causes metallic flavor in butter when present in sufficient amounts. Therefore, a good lime neutralizer should be as free as possible from metallic oxides." "Lime neutralizers should be low in calcium carbonate as this is less soluble than calcium

hydrate. The carbonate may be due to insufficient burning of the rock lime or to carbonation by exposure to air."

Hunziker (17), in his book "The Butter Industry," states concerning advantages of lime: "Its alkalinity is high, thus minimizing the amount required to reduce a given amount of acid. It can be used in cream of widely varying acidity without danger of frothing and of causing the cream to foam over and out of the vat." Lime has the further advantage of low cost.

Hunziker states also that high grade lime neutralizers properly used, do not injure the flavor of the butter and do, unmistakably, improve its keeping quality.

He lists the disadvantages of lime, as follows: (1) low solubility, the neutralizer mix of suitable strength will, therefore, always contain a large amount of undissolved particles in suspension. (2) Limes often contain calcium carbonates or such other impurities as clay, sand, etc. (3) Lime has a pronounced thickening tendency on the cream. This fault may be partially avoided by neutralizing to .25% instead of lower.

2. <u>Definition of Neutralization</u>.

Hunziker (17), 1927, writes: "In the sense used in the creamery, neutralization refers to the removal of excess acid, reducing the acidity to say approximately

.1% to .3%. The term "Neutralization," therefore, is a misnomer. A correct name for this process would be the "Standardization of cream for acid" or "Acid Reduction of Cream." However, "Neutralization" has become an established trade name. It has become an inherent part of the creameryman's vocabulary so that, in order to avoid confusion, and for the sake of clearness, it seems advisable to retain the trade term "Neutralization" in this discussion."

3. Object of Neutralization

"With the help of proper neutralization, the creamery hopes to and does accomplish the following three principal objects:"

a. To avoid excessive loss of fat that results from churning cream that is pasteurized while excessively sour.

b. To guard against the production of undesirable flavors in cream which may result, when cream that is high in acid is pasteurized at high temperature.

c. To improve the keeping quality of butter made from high-acid cream. This is by far the most important object accomplished by proper neutralization."

4. Importance of correct neutralization.

There are many references to the aid that neutralization of cream has been to the manufacturer of butter, who receives sour cream. But the results of improper neutral-

ization are very disastrous. Over neutralization is destructive to the quality of the butter.

Hunziker (17), declares that: "Generally speaking, the addition of a neutralizer does not improve the flavor of the butter. To be sure, acid reduction assists in preventing the flavor-damaging tendency of the combined action of high acid and high pasturizing temperature. When neutralizing to a point sufficiently low to destroy practically all the acid, the delicate butter flavor usually also suffers, and the resulting butter is flat and tasteless. Such butter has no pronounced flavor; it has no character, unless efforts are made to re-develop flavor and aroma by the proper use of a good starter. It is not advisable to reduce the acidity of the cream lower than is necessary to insure keeping quality in the butter. Alkalies such as must be used as neutralizers of cream have themselves a flavor entirely foreign to the pleasant flavor of good butter. "Too much neutralizer tends to impart to the cream a neutralizer flavor."

Correct neutralization involves five steps:

(a) Adoption of a definite standard of acidity. As was just stated, that standard should be just low enough to insure keeping quality in the butter, Hunziker (17) states, "Results of scientific study and commercial experience have amply demonstrated that, in order to insure

keeping quality and guard against the development in salted butter of fishy flavor and related defects, neutralization of sour cream must be carried far enough so that the acidity of the cream at churning time does not exceed .32%. Twenty-five one hundredths per cent is safe and adequate assuming that it is not intended to ripen the cream." However, if ripening is intended, a somewhat lower acidity may be adopted. Two tenths per cent is used by many.

(b) Correct and accurate test for acidity.

After the limit to which the acidity will be reduced has been decided upon, the amount of acid, calculated as lactic, must be determined.

Hunziker (17) describes the most commonly used test.

Wilster (18), Laboratory tests summer 1932, and Cordes (19) letter to Wilster June 1932, state that CO_2 must be driven off by boiling the sample in order that the true acid to be reduced may be determined. Wilster also made a series of trials with boiled and cold samples, using the common sodium hydroxide titration for acidity with phenolphthalein indicator. In 46 determinations of the difference in titration results when phenolphthalein indicator was used, Wilster (33) found, that when the acidity was reduced from .40%-.60% to .15%, more or less, with $NaHCO_3$, that there was a maximum difference of

.14% between the cold sample titration and the boiled sample titration, showing that carbon dioxide which was expelled by boiling will give a high titration if it isn't driven off.

In cream to which no neutralizer had been added the cold sample titrated a maximum of .05% more acidity than a sample of the same from which the carbon dioxide had been expelled by boiling.

About the same difference between the boiled and cold samples existed after neutralization with calcium compounds.

Cordes(19) states "We have had considerable trouble in some of our plants in getting the proper reduction of acidity by soda ash due, of course, to the CO_2 present in the cream."

The explanation of this higher titratable acidity in the cold sample is that carbon dioxide acts as a weak acid to phenolphthalein. When the carbon dioxide has been driven off by heating the sample, a titration indicates the true acidity. From this correct figure, the amount of neutralizer to add, may be calculated accurately, when the weight of the cream and strength of the neutralizer are known.

5. <u>Adding the Neutralizer</u>.

Protection of the cream against neutralizer flavor

and partially preventing the thickening due to lime "neutralization" depend upon the uniformity of distribution of the alkali. Hunziker (17), states that "a portion of the casein which comes into contact with the neutralizer may be caused to dissolve and disintegrate, a condition which usually yields a disagreeable, bitter flavor. It may also lead to saponification of a portion of the butter fat and the production of a soapy flavor." Haag (20), in a lecture before the members of the Oregon Butter and Ice Cream Makers Convention, Feb. 1932, declared that "The fat and strong base are present at the time of "neutralization" just as they are in the making of insoluble soaps. There is no reason why there would not be some saponification when ordinary factory-ground lime is used as the acid reducing agent."

The burning action of lime on the sugar of cream is easily demonstrated by adding a little lime to sugar solution in a test tube. Lactose content of cream is about 4½% according to Walker, Tisdale and Jones.

In order to avoid such damaging action, the neutralizer must be distributed quickly and completely through out the entire batch of cream.

Hunziker (21) gives the correct dilution of lime and magnesia neutralizers, and the table furnishes readily

available information as to the amount of such a mix to be used to reduce a certain amount of acid in a given volume of cream. The final step in neutralization is testing the cream after pasteurizing and cooling to see if the desired reduction of acidity has taken place, and testing again before churning.

6. Calcium and calcium compounds now used as cream neutralizers:

"Hydrated lime is probably the most commonly used of calcium neutralizers." Handling quicklime is quite inconvenient in a creamery, unless special slaking equipment is installed. Even then, most creamery operators do not properly do the work, writes Hunziker (17).

There are high calcium limes containing five percent or less of magnesium. All-magnesium neutralizers such as magnesium oxide and magnesium carbonate are obtainable after artificial purification which makes them high in price. They are highly satisfactory according to Hunziker (17).

Viscogen is mentioned by Hunziker (17). It is made by slaking one part of lime in three parts of water and adding it to a 30% sucrose solution. After it has settled overnight, syphon off the clear liquid which is called viscogen. The neutralizing strength is low and the cost comparatively high.

Eckel (22), and (23), in his book, "Cements, limes and plasters" 1st and 2nd editions, writes: "Limes as they are produced naturally are rarely free from insoluble materials which usually consist of silicon dioxide and aluminum and iron oxides." These last two are very bad in butter, causing metallic flavor and sometimes tallowiness in storage.

28 units CaO
37 units $Ca(OH)_2$
50 units $CaCO_3$ Have the same neutralizing value.
20.15 units MgO
29.15 units $Mg(OH)_2$
42.15 units $MgCO_3$

Coarsely ground lime, clay, sand, and organic matter is the insoluble material by the Bouska test (24).

Eckel (23) says the limes containing considerable magnesium are more efficient than the other neutralizers, as shown by the comparative amounts required to neutralize a given amount of acidity.

The exact alkalinity of any neutralizer must be known in order to reduce the acid to the desired percentage. It may be measured most conveniently in terms of 100 for a strength equal to that of $Ca(OH)_2$ pure. Then, assuming that all the acid in the cream is lactic acid, it is easy to calculate the amount of the neutralizer necessary to reduce .01% acid in each 100 pounds of cream.

On account of the affinity of lime for casein Hunziker and Hosman (25) and Hunziker (17) have shown that

a correction factor of approximately 20% in addition to the theoretical amount of $Ca(OH)_2$ is necessary.

7. Preparation of Limes for Neutralization of Sour Cream.

Eckel (23) gives the following information concerning the manufacture of lime hydrate: "One hundred pounds of pure quicklime requires 32.1 lbs. of water to completely slake it. One hundred pounds of 90% CaO quicklime requires 28.9 lbs. of water; and grades between 90 and 100, the two given, are calculated accordingly.

It may expand three times in volume if the water is added all at once or about one and seven tenths times if the water is added slowly.

There are three stages in the preparation of good quality hydrated lime:

1. Lump lime must be finely ground.
2. It must be thoroughly mixed with sufficient water.
3. It must be seived to a uniform fine powder.

"The quicklime is obtained in the first place by heating limestone." Holmes (26), in his book "Introductory College Chemistry", says, Limekilns, as a rule, operate at a temperature of 750° Fah.- 900° Fah. Wood fire is better than coal because the flame is longer. Towers 60 feet high and 7 feet wide produce 25 tons of lime daily. There are 6 towers around one gas producer, gas being better than either wood or coal.

On long exposure to the air quicklime becomes air

slacked changing to the hydroxide and the carbonate. The latter is only slightly soluble and is not good for neutralization. For that reason any lime to be used for cream neutralization should be well protected from the air.

II. B. Butter Manufacture.

1. History

Hannay (27) refers to the long period of the use of butter by the human race. Butter has been used by the human race since the earliest days from which records have been left. Ancient Egyptian civilization left many records of dairying. In one of the pictures believed to have been painted 4,000 B.C., the first churning scene is found. The butter is in finished rolls very much like those made on American farms, today. The practice of milking cows and making butter began in Virginia and Massachusetts soon after the establishment of those colonies," States Cameron (28). "By the close of the revolution, dairying was an important American industry, that was constantly growing in importance, fetching in millions to swell the national wealth and filling country graveyards with the worn out wives of farmers. Which is to say that it was an industry that was just as thriving and just as crude as the day in which it first existed."

"The commercial making of butter began at an early date and developed rapidly until in 1894 a billion pounds was being produced annually on the farms alone and some three hundred million pounds in factories." This shows that a large proportion of the total was farm produced less than 40 years ago.

"Much of this butter, probably the greater part of it, was of a very inferior quality being generally made by hit-or--miss, rule-of-thumb methods and without too much regard for principles of sanitation. Its strength lay in its aroma rather than its excellence. Some of it, however, was superlatively good from the earliest times."

Guthrie (29) in his book, The Book Of Butter , declares, "Butter is one of the oldest as well as one of the most universal articles of diet. The Hindoos used it as a food 2000 to 4000 years B.C. It is known that the Scythians and Greeks used butter in 450 B.C. A little later, there is a record of the Persians making and using it. In the early centuries butter was employed in many ways. The Hindoos offered it as a sacrifice in their worship. The Greeks and Romans did not eat it, but they used it as a remedy for injuries to the skin." He mentions its use for doctoring sore eyes, a skin and hair ointment, as a bath in cold regions, ~~doctoring~~

doctoring wounded elephants, lamp oil, medicine, in Spain "for external application only (17th century) and as cooling salve for burns."

"In some countries the possession of butter was considered an indication of wealth. In both Chile and Darel the practice of storing it in the ground still exists." Often it has been stored for over a hundred years.

"In early history butter was used as a food by comparatively few persons. Norway was exporting butter during the thirteenth century. Sweden exported some during the fourteenth century. Late in the seventeenth century record is made of the Irish burying their butter in peat bogs to age it, and hide it from invaders. In the United States butter has been used as a food only. Banding together of producers was the mucleus of the modern creamery company, some of which now make more than thirty million pounds of butter annually."

"The first creamery in the United States was in Campbell Hall, Orange County, New York, in 1856. Soon afterward many creameries were organized, especially in the Central and Western states."

Butter shipping and storage soon developed to such an extent that the problem of producing a high quality product which would keep, came to the foreground. There

was much variation in the experimental procedure of the work at that time, consequently the statements of results are often conflicting.

From the earliest days of buttermaking there has been the problem of over-sour cream. When Flint first used soda in 1858 and found that better butter resulted, he was at the starting point of what is now known in the language of the trade as the practice of "Neutralization."

During the time when most of the butter was allowed to age, as did the Irish by burying it in the peat bogs, the general opinion was that it was wasteful to churn cream of low acidity. That belief prevailed until the latter part of the nineteenth century.

Dean (30) who experimented in making sweet cream butter at Ontario Agr. College, declares, "We must churn sweet cream at low temperatures (45° or below), in order to obtain all the butter. Churning at ordinary temperatures means a great loss of fat in the buttermilk."

Patrick, Leighton, and Bisbee in (31) noted that: (a) the yield of butter from sour cream was usually larger than from sweet; (b) sour cream usually churned more quickly that sweet; (c) the butter from sour cream usually contained less fat and more water than did that from sweet cream; (d) the butter from sour cream usually contained a trifle more casein than did that from sweet.

Ladd (32), in 1889, reported that by churning sweet cream at the same or at a higher temperature than is required for ripened cream, there was a large loss of butterfat, but by lowering the temperature from 68 deg. Fah. for ripened cream to from 50 deg. to 54 deg. Fah. for sweet cream, there was no greater loss in the buttermilk.

3. Efficiency of Pasteurization

Wilster(4) in the fourth year annual report of state butter scoring emphasizes the fact that, "Pasteurization can be more efficient in some creameries. Pasteurization with the lids open and leaving a plug of unpasteurized cream in the pipeline between the vat and the outlet valve are still practiced by some buttermakers. Pasteurization, if properly performed, results in better quality of butter with improved keeping quality.

Pasteurization is well along toward 100% efficient. The result of holding cream in a vat at 145 deg. Fah. for 20 minutes gave, according to Searing (54) a microorganism killing efficiency of 99.864 to 99,994 percent. The larger the number of bacteria in the original sample, the more efficient the bacterial killing effect in the trials by Mortensen, Gaessler, and Cooper (33). Hunziker, Spitzer, Mills and Switzer (34) at the Indiana Agr. Exp. Sta. found that the holding method of pasteurization was more efficient than the flash method of pasteurization

was more efficient than the flash method of pasteurization. It averaged over 99.9 percent of germ killing power on all types of micro organisms. The trials holding 145 deg. Fah. for 10, 15, 20 and 40 showed the advantage of the 20 minute period, though the greatest reduction occurred in forty minutes. White and Campbell (35) compared 116 churnings of sweet cream butter for keeping quality in which some batches of cream were pasteurized at 145 deg. Fah. for 25 minutes, and 145-165 deg. Fah. for 30 minutes. Samples stored at zero deg. Fah. for 7½ to 9 months indicated when scored that keeping quality was not affected by those temperatures of pasteurization of cream. The samples pasteurized at the higher temperatures kept as well as those pasteurized at the lower temperatures.

4. The object of the use of starter.

Starter is a culture of acid, flavor, and aroma producing organisms. Good-grade sweet cream, produced under sanitary conditions, would probably produce butter of good and fairly uniform quality. However, that type of cream makes up a very small portion of the supply of most creameries. Therefore, in order to make a uniform product having what is described as a desirable, sweet, "nutty" flavor and aroma, it is desirable to add such a "starter." Flavor claims nearly half of the total

100 percent score on butter.

By use of a good starter of S. lactis, S citrovorus, and S para-citrovorus in pure culture, sweet cream may be ripened to an acidity which will aid in preventing the development of micro-organisms.

Derby and Hammer (36) at the Iowa Agri. Exp. Sta. maintain that, "surface taint is one of the defects which is caused by a group of organisms which are sensitive to acid, and are, therefore, overcome by the numerous acid producing starter organisms. The value of butter culture in the control of surface taint should be recognized. When such a culture is employed either with or without actual ripening of the cream, the number of butter culture organisms in the fresh butter is comparatively high. The changes in the number of butter culture organisms in the butter are greatly influenced by the salt content, but under any conditions the organisms should be expected to influence the development of the types found capable of producing surface taint, since these belong to a general group of organisms that is sensitive to acid."

Russell and Hastings (37) also report that the development of lactic acid has a protective action against putrefaction.

Eckles (38) mentions an attack of surface taint at

Iowa State College, during a dry period in the summer of 1900. By rejecting poor quality milk, caring for the sanitation of equipment, and use of starter, aided by rainy weather, the trouble disappeared. The contamination was lessened and overcome by the use of the starter organisms. Protein liquifiers are especially blamed for causing surface taint according to Hood and White (39), who made a study of surface tainted butter in Canada.

McKay (40) is of the opinion that, "butter containing no acid will turn rancid."

Grimes (41) has drawn the conclusion that the quality of the cream brought in by the patron determines the quality of the stored butter.

Pasteurized sour cream to which 10 percent starter was added, and pasteurized sweet cream with 10% starter both with out ripening, demonstrated to Hammer and Jensen (42) that the acidity of the butter culture had no significant influence on the quality and keeping quality of butter made from such cream.

Manns (43) Penney (44) and Curtis (45) in 1890 and Robertson (46) in 1891, found that a greater yield of butter was obtained from ripened cream than from sweet cream. Hills (47), in 1893, found that the most complete churning was attained with thick, ripened cream at low

temperatures of churning.

At the present time, the difference in the amount of fat loss in the buttermilk of churnings of sweet and sour cream is negligible.

The investigators during the period between 1890 and 1910 reported on the flavor of the butter manufactured in their experiments in the following terms:

1. In favor of sweet: Ladd 1889 (32) "The flavor of the butter from sweet cream is quite different from that of the butter made from ripened cream."

Manns (43) at the Ill. Agr. Expt. Sta., 1890 stated, "Increase in acidity of cream beyond a certain point brought risk of injury to the quality of butter due to fermentations setting in after that stage in ripening had been reached."

Curtis (45) of the Texas Agr. Expt. Sta. from his work on the quality of butter from sweet and sour cream says, (1890), "Seven split churnings gave scarcely a noticeable difference between the two types of butter. The difference was in favor of the sweet cream butter."

2. In favor of ripened cream butter.

Penney (44) 1890, who compared butter from sour and sweet cream at the Del. Agr. Exp. Sta. declares, "Sweet cream butter lacks the stand-up quality of sour cream butter, and it is considered by creamerymen not to stand shipment, handling, or warm weather as well. In a word,

sweet cream butter is less profitable to make and harder to sell than sour cream butter."

2. In favor of ripened cream butter.

Dean (30) 1891 and 1892 (48) found that his sweet cream butter went off flavor after 3 to 4 weeks storage. It was inferior in quality to the ripened cream butter.

Sweet cream was found to deteriorate less, when kept at 50 deg. Fah. for 3,4,6, and 7 month periods, by (31), Patrick, Leighter and Bisbee, than the ripened cream product.

Beach (49), 1906 compared soured skimmilk and "Pure Culture" Starters in 14 trial split churnings and found the scores of the resulting butter almost identical.

Fishy Flavor: Rogers (50) 1909 of the U.S. Dept. Agri. after working with fishy flavors in butter says, "In the opinion of the writer, fishy flavor is caused by a slow, spontaneous, chemical change to which is favored by the presence of small amounts of oxygen. Fishy flavor may be prevented with certainty by making butter from pasteurized sweet cream."

Sommers (21) In his book called, "Ice Cream Making." 1932 declares that, "A fishy flavor in dairy products is due to the development of trimethylamine by the hydrolysis and oxidation of the lecithin which is always associated with butterfat. The factors which favor

these chemical changes are (1) a high acidity, and (2) iron or copper salts. It is known that acid favors the hydrolysis of lecithin, but it is not definitely established how the iron and copper salts function in producing fishiness. The fact that iron and copper salts are known to be oxidative catalysts in many oxidation reactions suggests that they function here in the same capacity. Thus the explanation would be that the hydrolysis of lecithin favored by the acid reaction, produces choline;and the choline on oxidation yields trimethylamine which has a pronounced fishy odor and flavor."

II. Neutralization of Sour Cream and the Effect on the Quality of Butter Produced Therefrom.

In Ref. (1) the authors state, "A cream which is absolutely clean in flavor and sweet does not need to be neutralized, and does not require the addition of any foreign substance in order to manufacture, therefrom, with the aid of pasteurization and proper ripening, afterward, by a first class starter, the best butter that is possible to obtain from such cream."

It is the cream coming from scattered few-cow farms, the 14% of Oregons cream received through shipping stations, by the creameries, U.S.D. exp. of Com. (5) and

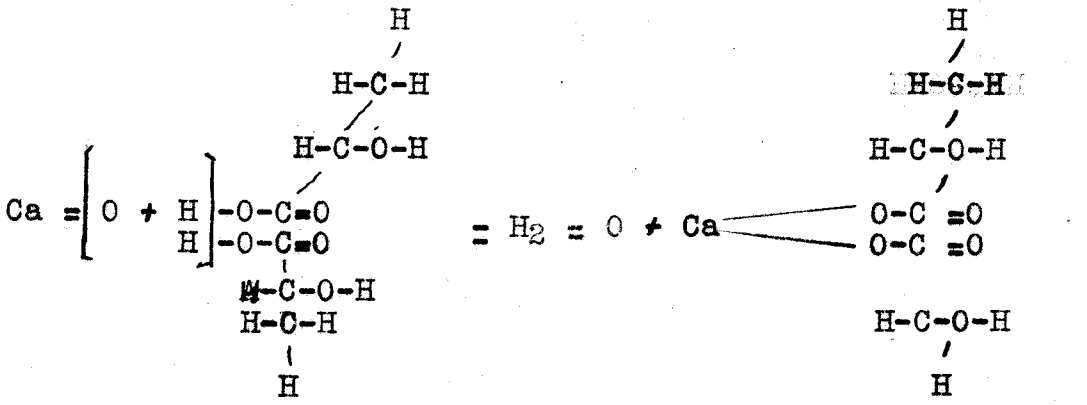
much other, that needs be considered in this part of the discussion of buttermaking.

As has been previously stated in this thesis, neutralization to reduce the acid, the product of bacteria, is not ideal, but practically analogous to straining out dirt from milk and pasteurizing to reduce the excessive bacteria, which should have been kept out in the first place.

Oregon Laws (51) provide that, "The use of lime and soda as neutralizers as is practiced generally in the butter-making process, or the pasteurizing of cream for buttermaking shall not affect the grading of creamery butter."

The chemistry of neutralization as given by Guthrie (29) is as follows: "All cream neutralizing agents in general react the same. The neutralizer combines with the lactic acid in the cream, forming a lactate. Calcium and Magnesium, which are preferred in the United States, are bivalent elements and unite with the lactic acid as follows:"

1 molecule of CaO. + 2 molecules of lactic acid = 1 molecule of water 1 molecule of calcium lactate.



$CaO + CH_3CH(OH).\ COOH = H_2O \quad Ca(C_3H_5O_3)_2.\ 5H_2O$

One pound of CaO will neutralize 3.21 lbs of lactic acid. ($90 \div \frac{56}{2} = 3.21$.) For one pound of lactic acid $\frac{56}{2} \div 90$, or .311 lbs, of CaO will be required."

Calcium has a great affinity for casein and forms compounds having 2.35% CaO and 1.46 percent CaO, called basic or di-calcium caseinate and neutral or mono-calcium caseinate, respectively. Because of this affinity, more calcium is required to neutralize a given amount of acid in cream than is calculated.

Hunziker (17) gives this necessary excess as 20%. Hunziker and Hosman (25) are the original reference. Overman (52) gives the following reason why this extra 20% is necessary: (1) Casein is present in fresh sweet cream in the form of calcium caseinate. (2) Calcium caseinate plus lactic acid formed by bacterial action

= free casein plus calcium lactate plus casein lactate. (3) Lime reduces acidity and favors the combination of free casein plus calcium and the reformation of calcium caseinate.

That lime is the most desirable alkali suitable for the reduction of acidity in cream is the belief of Hunziker (17), Guthrie (29). The questionaire answers by leading creamerymen and educators as mentioned in a answer to question 4 of reference (1), and many others. However, the differences of opinion in favor of lime and soda neutralizers still forms an unsettled issue. Lime has its disadvantages as well as the advantages of low cost, high neutralizing value, suitability over a wide range of acidity without danger of foaming over and out of the vat, and desirable effect on the butter shown by commercial use over a number of years. The quality of the butter from high-acid cream is not as good for storage, and it is likely to have fishy flavor and other storage defects, which can be eliminated, largely, by proper neutralization. Hunziker (17) p. 143 and Sproule and Grimes (12).

Wilster (53), in the annual report of the Oregon State Butter scoring, 1933, (fourth year) says to participants, neutralization of sour cream should receive more consideration by the buttermakers. Proper determination

of the acid in the cream, and careful addition of the well diluted neutralizer are essential in cream neutralization. When sweet and sour cream are mixed, better results are obtained if the sour cream is placed in the vat first and neutralized before the sweet cream is added, than when the two grades of cream are added together without any previous acid reduction of the sour cream.

III. Experimental

A. Purpose of Experiment--The Problem.

The sour cream coming into the creameries to be manufactured into butter has been found by many research workers and practical buttermakers to yield a better flavored and butter keeping quality butter if it is neutralized before it is pasteurized, than if it is churned in the sour condition. However, flavors and odors are found which are caused by the caustic properties of the lime, and which are absent in butter from cream neutralized with sodas which are very soluble and ready to act upon the acid. Long contact of the undissolved particles of lime with the sugar and fat of the cream may cause caramalization and saponification --burning of the sugar, and making soap of the fat, to some extent. Such "scorched" and "burnt by neutralizer" flavors have prompted us to seek to remedy those defects.

The fact has been demonstrated that, when a soluble

alkali such as a ten percent solution of sodium bicarbonate or sodium carbonate is thoroughly and quickly dispersed through cream in such an amount that the acidity will not be lowered too close to the neutral point, no such flavors are noticeable.

"Lime water," according to Hunziker (17) consists of the clear liquid which separates on the surface after the slaked, but undessolved lime has dropped to the bottom. The lime water contains lime hydrate in solution only. From the standpoint of ease of handling, rapidity of action, and accuracy of neutralization, this clear lime water would be most suitable. But lime is only very slightly soluble in water. It is soluble to the extent of about .137% in cold water and of .075% in boiling water. Limestone is much too weak, and the amount required to reduce the acidity from .85 to .25 percent in cream, would be double the volume of the cream.

Milk of lime is made by stirring lime in water to form a suspension. Such a suspension contains lime in solution and undissolved lime in suspension. The strength is not easily controlled because of the setting out of the suspended lime particles.

The purpose of the work herein reported, was that of finding compounds of calcium which would have a greater solubility and ease of complete and rapid dis-

persion when added to cream.

B. Suggestions which were directly helpful from chemical references to the sugar industry. A calcium compound which (1) would contain a relatively high amount of calcium in a form in which there would be no coarse particles to slow up the neutralizing and pass on into the butter, and (2) which could be quickly and easily dispersed through the cream.

The three disadvantages of using lime hydrate which a more soluble form of lime would eliminate or control, have been mentioned i.e. 1. low water solubility 2. Insoluble material present. 3. Increase of viscosity of the cream.

In the "Chemical Abstracts" were found articles written concerning work done with sugar and lime. A sugar solution dissolves more lime than does a water solution. Babcock and Russell (55) have applied this knowledge in their experiments with "Viscogen" to restore the body or viscosity of cream. Hunziker (17) states that its neutralizing strength is too low and the cost too great to be a practical form of lime for cream neutralization on a commercial basis.

Cammeron and Patten (56) found that lime was quite soluble in sucrose solutions and less soluble in glycerol. They were able to get 6.07% $Ca(OH)_2$ to dissolve in a

19.86% sucrose solution.

Van Slyke (57) reports that a sample of "sugar of lime" contained 63.8% sucrose and 11.6% CaO in solution.

Hoglund (58) states that calcium saccharate-technically prepared by the Stephen's process contains generally 100 parts of CaO to 100 parts of sugar, when theoretically it should only have 49.1 parts, The excess is uncombined CaO and $Ca(OH)_2$.

F.E. Combs (59) from his work in preparation of sugar lime states that uniform results can be obtained only by slaking the lime in large quantities, making into a thick paste and having a layer of water over it. Burnt lime changes considerably on storing and batches of milk of lime made from day to day do bot have the same sugar holding power.

Taegner (60) in his article "The estimation of the alkalinity of sugar-lime" writes "The usual method of determining the lime content of tri-calcium saccharate by titrating a weighed quantity of it after mixing with hot water gives incorrect results. When using litmus or phenolphthalein as an indicator, the end point is not constant, for by allowing it to stand, the red color re-appears and more acid is necessary. This comes about by the presence of some unburned lime and carbonates of

metals. He recommends a process similar to that used by Bouska as referred to in reference (24).

The references just mentioned indicated that the solubility of lime could be increased by sugar solutions. It was, therefore, decided to prepare various combinations of lime and sugar and determine their neutralization effect upon sour cream.

2. Source of alkalies and method of storing them.

The lime hydrate was delivered in paper bags holding 100 lbs. each. As they were opened they were emptied into bright, well-tinned 10 gallon cream cans and the lid was pressed on tightly over several thicknesses of parchment paper to keep out the air and moisture.

Other neutralizer materials used, the ground CaO and the prepared calcium sucrate, were kept in covered jars, protected from air and moisture.

CaO, quicklime, was obtained from a fresh supply, in large chunks, ground fine, and stored in glass jars with tight fitting lids until used.

Dr. J.R. Haag of the Agricultural Chemistry Dep. of Oregon State College assisted in the making up of a viscous syrup by stirring a sucrose solution of unknown concentration into a milk of lime suspension. By shaking the container vigorously at short intervals for two hours and 30 minutes, a "glassy", viscous material

resulted. The larger particles of lime, sand, clay and so-forth settled to the bottom after several hours of standing. In later preparations, instead of shaking the container to mix the contents a milk shake mixer was used to keep the milk of lime agitated while the sugar solution was being added. The resulting product was uniformly well mixed and seemed satisfactory so we analyzed it for sugar and $Ca(OH)_2$ content as follows.

The cost of manufacturing, for materials, with lime at $18.00 a ton and sugar at five dollars a hundred pounds, was three cents per pint.

Preparation and Properties of the calcium-sucrose compound. The sucrate mixture was prepared by pouring a solution of three parts of sugar in two parts of water slowly into a vigorously stirred milk of lime suspension of four parts of lime hydrate to five parts of water by weight, that is, approximately 188 parts sucrose, 63 parts lime hydrate, (of a purity of 70% calculated as CaO) and 203 parts of water, when mixed as above directed will reach a standard consistency according to a number of trials.

Analysis of the Complex calcium sucrose mixture.

1. (A) The Bouska method (16) was used to determine the alkalinity. A sample containing 8.69 g. of the highly concentrated lime-sugar complex mixture of calcium

sucrates with probably some suspended calcium hydroxide was weighed into a 250 c.c. glass beaker. The weighing was done on an analytical balance. Eighty c.c., an excess .239 N. HCl, was added and the contents of the beaker was brought to a boil and held at that temperature for five minutes. The excess acid was then titrated with .0955 normal sodium hydroxide. The alkalinity as calcium hydroxide was calculated directly from the titration values. The amount of NaOH used was 13.76 c.c. Approximately 10.1% $Ca(OH)_2$ was a result obtained by the following calculations.

Weight of calcium sucrate complex 8.69 grams.

Amount of N/.239 HCl added 80 c.c.

After boiling for five minutes 13.76 c.c.

of N/.0955 NaOH brought the beaker.content back to neutral to phenophthalein.

CALCULATIONS.

80 c.c. of N/239 HCl is equivalent to 19.12 c.c. N/HCl.

13.76 c.c. of N/.0955 NaOH is equivalent to 1.314 c.c.

19.120--1.314 = 17.806 of N.HCl used against the $Ca(OH)_2$ of the neutralizer.

There is 17.806 c.c. of N. $Ca(OH)_2$ equivalent in the 8.69 grams of the calcium sucrate mixture. In one

gram(17.806 ÷8.69) are 2.05 c.c. of N/1 $Ca(OH)_2$. 2.05 x 1.33 (Sp. Gravity) x 3.7 (Percent of $Ca(OH)_2$ in 100 c.c. of N. sol.) = 10.08% $Ca(OH)_2$ (gr. per 100 c.c.)

The check sample using 4.85 g. of the calcium sucrate mixture from another batch of the same formula gave 10.1% Ca(OH) when calculated by the same method.

The Specific Gravity of the complex sucrate mixture is 1.33 at 20 degrees centigrade. It was determined by weighing a dry 100 c.c. graduated cylinder on an accurate balance, filling it to the 100 c.c. mark with the mixture and re-weighing the cylinder plus the contents. The 100 c.c. content weighed 133 grams. Three trials on three preparations of the same formula showed a check within .4 gram.

An alkalinity determination was made by using 75 c.c. of the calcium sucrate complex mixture in 42 pounds of cream which had been pasteurized to stop acid production by the bacteria. A nine gram sample was weighed out, brought to a boil, cooled, and titrated. The acidity was .31% at this stage when the 75 c.c. were added to the 42 lbs of cream. The temperature was 66 deg Fah. at the time of adding the neutralizer and was held at that temperature for nearly an hour after which time it was cooled to 38° deg. Fah. by placing it in a cold room at that temperature. After 15 hours the cream was warmed

to a churning temperature of 52 deg. Fah. and a boiled nine gram sample titrated for acidity of the 42 pounds of cream. A reduction of .10 percent acidity had been brought about in 42 lbs of cream by 75 c.c. of the neutralizer. as was calculated from previous neutralization trials and addition of 20% to combine as calcium caseinate. 75 c.c. neutralized .1% acid in 42 lbs of cream. 42: 100 = 75:X X = 178.6 c.c. required to neutralize .1% acid, calculated as lactic, in 100 lbs. of cream.

One hundred pounds of cream = approximately 12 gallons 178.6 ÷ 12 = approximately 15 c.c. of calcium sucrate mixture required to neutralize .1% acid in one gallon of cream. To neutralize a 300 gallon vat of cream from .45% acidity to .25% will require 15 X 300 X 2 = 9.000 c.c. of the mixture of sucrates. 9000 c.c. = $\frac{9000}{473.2}$ or approximately 19 pints of the calcium and sugar mixture.

The Pacific Coast Testing Laboratory, Seattle, Wash. reported the analysis of the lime produced about the time that these experiments as used in being Iron and Aluminum Oxides 0.31 percent, Magnesium Oxide 0.25 percent and Calcium Oxide 74.82 percent. Comment is made by the chemist that the Mg O is figured to Mg CO_3, the CO_2 thus used taken from the total CO_2 leaves the CO_2 that is used to combine

with some of the CaO to form $CaCO_3$. The balance of the CaO combines with the H_2O to form $Ca(OH)_2$.

Analysis was made a representative sample of lime hydrate kept in the cream can by weighing out 2.1836 g, adding 8 c.c. of conc. HCl. and making it up to a 250 c.c. volume. Aliquots of 25 c.c. were taken, Five c.c. of $FeCl_3$, were added to each and this solution made basic with NH_4OH. to precipitate Fe and Al in the hydroxide form. The content of the beaker was then filtered, hot, through tared gooch crucibles, washed with hot water. The gooches were dried, then burned to constant weight, and the increase in weight of the tared gooches taken as the weight of Fe_2O_3, and Al_2O_3. To the filtrates was added $(NH_4)_2 C_2O_4$ to precipitate the calcium. The calcium oxalate formed was filtered through gooch crucibles and the gooches ignited at bright red heat to convert the oxalate to oxide. The analytical results thus obtained were for calcium 70% and for iron and aluminum oxides .34 percent.

<u>The sugar content</u> of the mixture of calcium and sucrose was determined by weighing out a sample of 2.1984 grams and a duplicate sample of 2.1309 grams of it into 250 c.c. volumetric flasks allowing the material to digest at room temperature in 5 c.c. of conc. HCl to 10 c.c. distilled water for 27 hours. The acid solution was brought ~~to~~ a very slight acid reac-

tion to litmus by adding stick NaOH. further reduction of the acidity to near the neutral point was accomplished by adding Na_2CO_3. With the solution slightly acid to litmus the volume was made up to 250 c.c. with distilled water. Two 25c.c.aliquot parts of the sample and the duplicate or check sample were pipetted into 250 c.c. beakers, 25c.c. of distilled water added and the solutions heated to boiling. The sugar was reduced by adding Fehlings'solution and boiling it for exactly two minutes. It was then filtered through tared gooch crucibles, the asbestos and Cu_2O were washed with a little ethyl alchoholand ether and the crucibles dried to constant weight in a drying oven used for that purpose. From the weight of Cu_2O read from conversion table in Browne's Sugar Book, published by John Wiley and Sons in 1912 or from the hand book of Chemistry and Physics by Hodgman-Lange published by the Chemical Rubber Publishing Company, Cleveland Ohio, was calculated the weight and percentage of sucrose. The results showed the complex material to be 41 percent sugar. This highly concentrated complex mixture of calcium sucrates with probably some suspended calcium hydroxide was given a serial number 909 by which it will be referred to in this report.

Some slight variations in the amount of lime and

sugar were tried, but were considered less valuable than 909 for cream neutralization.

The results of the experiments made to compare the qualities of 909 with those of the hydrated lime will be shown in the following pages.

4. The cream used in the experiment was delivered by patrons of the Oregon State College Creamery and was soured by its natural flora. Nearly all of it was a good grade of sour cream having acidities as shown on table 4.

5. Adding the neutralizer to the cream

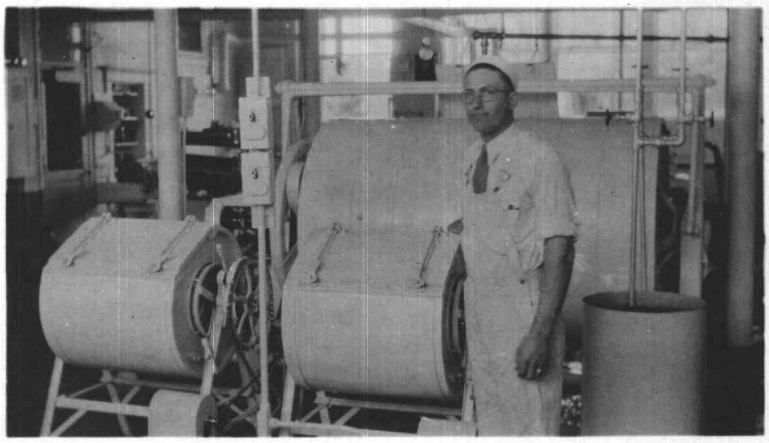
In the experiments, the neutralizers were added in dilute form. Two pounds in a gallon of water, as recommended by Hunziker (17) was followed. The calcium sucrate neutralizer in the #909 form was diluted with an equal volume of warm water. The technically prepared tri-calcium saccharate was added to the cream in a dilution of four pounds to the gallon ratio. In each case the neutralizer was added to the cream from a sprinkler while the cream was being vigorously stirred. The correct point to reduce the acidity to was discussed in part five of the review of literature.

6. Churns used were two which are electrically driven, having a capacity of about 50 gallons of cream. They were used with 15 of the 18 lots of cream that

Plate 1

A comparison of the lack of stability of the lime and water suspension with the stability of the calcium, sucrose, and water mixture.

Two churns used in the experiments



were divided into two parts each in the experiments. A one-gallon, hand operated glass churn was used with three lots of cream from which six churnings were made, when technically prepared tri-calcium saccharate was compared for neutralization with calcium hydrate having the composition reported by the Pacific Coast Testing Laboratory in part 3 of the experimental section of this report.

7. Scoring the butter made in the experiments.

Nearly all of the samples were scored at the Oregon State Monthly Butter Scorings by the judges of butter who score the samples sent in each month to the college for analysis and scoring. The samples were known to the judges by number only. They knew nothing about the manufacturing methods and had no information regarding the butter other than that which they could gather from the numbered samples before them. In most cases there were at least three judges scoring individually. More often there were four judges, all of whom were qualified to do the work. Six lots were scored by number, by the Professor of Dairy Manufacturing and the head butter-maker at Oregon State College. They also scored individually.

The flavor of cream neutralized by the several neutralizers used waw passed on by a number of people at the college who were experienced in scoring milk and dairy products.

Results Obtained.

Comparison of scores of butter made from 12 lots of cream neutralized with #909 and lime hydrate.

lot no	Date of churning	Age (days) when scored	Acidity before past.		Acidity of cream at the churn		Score	
			Hyd.	909	Hyd.	909	Hydrate	#909
1.	3/6/33	42	.44	.44	.18	.16	90.00	91.0
2.	3/15/33	33	.43	.43	.22	.24	90.50	91.0
3.	3/31/33	18	.48	.48	.20	.18	90.50	90.0
4.	4/3/33	15	.53	.53	.20	.24	90.00	91.0
5.	4/12/33	6	.42	.42	.18	.17	90.00	91.5
6.	4/14/33	4	.58	.58	.26	.30	89.00	91.5
7.	4/29/33	10	.56	.56	.25	.25	91.25	91.75
8.	5/1/33	8	.52	.52	.20	.24	90.50	91.25
9.	5/1/33	8	.46	.46	.18	.27	90.25	91.5
10	5/2/33	7	.46	.46	.19	.19	90.25	91.75
11	5/2/33	7	.50	.50	.21	.18	90.50	91.50
12	5/4/33	5	.43	.43	.21	.23	91.50	91.50
Average		12			.207	.221	90.35	91.27
Average diff.						.014		.92

Butter was held at 0° to 8° Fah. until scored.

The sample for the acidity tests were boiled to drive off CO_2.

Comparison of scores of butter made from cream neutralized with the sugar lime neutralizer #909 and calcium hydrate shows that for the 24 churnings an average of .92 of a point in favor of butter made from cream neutralized with #909. The improvement in score ranged from 0 to two and one half points.

TABLE 2

Comparison of scores of butter made from cream neutralized with tri-calcium saccharate, (made by the Stephen's Process as given in Browns Sugar Book (61).) and calcium hydrate.

Sample	Date of churning	Age in days when scored	Score hydrate	saccharate.
1.	4/8/33	10	91.00	91.00
2.	4/12/33	6	91.25	91.75
3.	4/12/33	6	89.00	90.00
4.	4/14/33	4	91.50	90.00
5.	4/17/33	1	90.00	90.00
Average		5	90.55	90.55

A. Comparison of scores of butter made from cream neutralized with tri-calcium saccharate and lime hydrate shows that there was little difference if any in the quality of the butter when these two neutralizers were used to reduce the acidity of the sour cream. The results are shown in table 2.

That conclusion may be justified on the grounds that the insoluble material and of the lime, used to make up the tri calcium saccharate, remains in that product and may carry into the butter the same as when the hydrate of lime is used in its ordinary form.

TABLE 3

Manufacturing data in 18 lots of cream half of which was neutralized with #909 and half with calcium hydrate.

Sample	pounds of cream	test of cream	30 min. period-145° past. temp.	time held cold before churning hours.
1 A	41	33	145	15
1 B	41	33	145	15
2 A	27	31	145	12
2 B	27	31	145	12
3 A	44	35	145	14
3 B	44	35	145	14
4 A	24	38	145	14
4 B	24	38	145	14
5 A	37	32	150	15
5 B	37	32	150	15
6 A	33	40	150	12
6 B	33	40	150	12
7 A	35	28	150	14
7 B	35	28	150	14
8 A	36	46	148	20
8 B	36	46	148	20
9 A	47	24	145	15
9 B	47	24	145	15
10 A	30	33	145	15
10 B	30	33	145	15
11 A	21	31	150	15
11 B	21	31	150	15
12 A	36	29	150	16
12 B	36	29	150	16
13 A	34	34	150	12
13 B	34	34	150	12
14 A	40	30	150	15
14 B	40	30	150	15
15 A	30	33	150	15
15 B	30	33	150	15
16 C	36	29	150	20
16 B	36	29	150	20
17 C	32	31	150	14
17 B	32	31	150	14
18 C	39	33	150	14
18 B	39	33	150	14

A = #909
B = Hydrate
C = technically prepared calcium saccharate.
* = acidity before neutralization and at churn given on table IV.

TABLE 5 (continued)

Manufacturing data in 18 lots of cream half of which was neutralized with #909 and half with calcium hydrate.

(continued) Temp. held cold	churning temp.	Length of churning period	Temp. of buttermilk	Fat in buttermilk.
1 A 38	54	36	58	.20
1 B 38	54	45	58	.35
2 A 38	50	40	56	.40
2 B 38	50	48	56	.20
3 A 46	52	40	58	.25
3 B 46	52	45	58	.25
4 A 38	54	35	60	.60
4 B 38	54	40	60	.45
5 A 44	48	60	54	.30
5 B 44	48	40	54	.15
6 A 38	54	35	58	.10
6 B 38	54	45	58	.10
7 A 38	52	30	58	.20
7 B 38	52	35	58	.20
8 A 38	52	26	58	.25
8 B 38	52	40	58	.20
9 A 40	54	45	60	.15
9 B 40	54	40	60	.15
10 A 38	50	22	58	.20
10 B 38	50	35	58	.10
11 A 38	52	48	56	.25
11 B 38	52	40	56	.20
12 A 38	54	36	57	.10
12 B 38	54	30	57	.10
13 A 38	54	48	59	.15
13 B 38	54	60	59	.10
14 A 38	56	75	62	.10
14 B 38	56	55	62	.12
15 C 38	52	50	58	.18
15 B 38	52	55	58	.20
16 C 38	54	40	60	.10
16 B 38	54	40	60	.11
17 C 38	52	40	58	.14
17 B 38	52	45	58	.20
18 C 38	50	40	56	.10
18 B 38	50	50	56	.30

A = #909
B = Hydrate
C = Technically prepared calcium saccharate.
* = Acidity before neutralization and at churn given on Table IV.

Table III. shows manufacturing data in 18 churnings of nine lots of cream, half of each lot neutralized with calcium sucrate complex mixture and half with calcium hydrate.

The number of pounds of cream in each churning ranged from 27 to 40 pounds which was enough to make the churns operate well.

The test of the cream was from 26.0% to 46.0%. The heavy cream was thinned with whole milk to about 33.0% fat. The pasteurizing temperatures were between 145 and 150 deg. Fah., and the holding period 30 minutes in all cases. The time held cold before churning was from 12 to 20 hours and the average holding temperature 38 deg. Fah.

A churning temperature of 50 to 56 deg. Fah. seemed to control the churning period quite well when the cream was about .25% acidity, and 33-% fat.

The temperature of the buttermilk varied according to the room temperature, from one to two deg. Fah.

Fat loss in the buttermilk varied from 0.10% to 0.60% with an average for all samples of .201%.

TABLE IV.

Original acidity and acidity at the churn, of the cream used in the experimental churnings.

Sample No.	Samples boiled to drive off CO_2	Original		At Churn	
		Hyd.	#909	lime hydrate	909
1		.56	.56	.24	.21
2		.55	.55	.20	.26
3		.43	.43	.23	.21
4		.48	.48	.20	.22
5		.40	.40	.24	.28
6		.50	.50	.20	.20
7		.53	.53	.18	.19
8		.53	.53	.17	.21
9		.45	.45	.21	.18
10		.48	.48	.24	.24
11		.43	.43	.30	.26
12		.49	.49	.31	.30
13		.50	.50	.21	.28
14		.41	.41	.18	.24
15		.49	.49	.22	.22
16		.56	.56	.20	.21
17		.42	..42	.24	.26
Average				.22	.23

Original acidity and acidity at the churn of the cream used in the experimental churnings, shows that the average acidity of the churnings in which #909 was used was .01 higher than in those in which the lime hydrate was used as a neutralizer. That difference is insignificant.

TABLE V. PINTS OF NEUTRALIZER REQUIRED TO NEUTRALIZE ACIDITY TO .25% WITH #909 NEUTRALIZER

Pounds of Cream	Per Cent Acidity in Cream																		Pounds of Cream
	.29	.33	.37	.41	.45	.49	.53	.57	.61	.65	.69	.73	.77	.81	.85	.89	.93	.97	
100	.15	.3	.45	.6	.7	.9	1.05	1.2	1.35	1.5	1.65	1.8	1.9	2.1	2.2	2.4	2.5	2.7	100
200	.3	.6	.9	1.2	1.5	1.18	2.1	2.4	2.7	3.0	3.3	3.6	3.9	4.2	4.5	4.8	5.1	5.4	200
300	.45	.9	1.3	1.8	2.2	2.7	3.1	3.6	4.	4.5	4.9	5.4	5.8	6.3	6.7	7.2	7.6	8.1	300
400	.6	1.2	1.8	2.4	3.	3.6	4.2	4.8	5.4	6.0	6.6	7.2	7.8	8.4	9.	9.6	10.2	10.8	400
500	.75	1.5	2.2	3.0	3.7	4.5	5.2	6.0	6.7	7.5	8.3	9.0	9.7	10.5	11.2	12.	12.7	13.5	500
600	.9	1.8	2.7	3.6	4.5	5.4	6.3	7.2	9.1	9.	9.9	10.8	11.7	12.6	13.5	14.4	15.3	16.2	600
700	1.05	2.1	3.1	4.2	5.2	6.3	7.3	8.4	9.4	10.5	11.5	12.6	13.6	14.7	15.7	16.8	17.8	18.9	700
800	1.2	2.4	3.6	4.8	6.0	7.2	8.4	9.6	10.8	12.	13.2	14.4	15.6	16.8	18.	18.2	20.4	21.6	800
900	1.35	2.7	4.0	5.4	6.7	8.1	9.4	10.8	12.2	13.5	14.8	16.2	17.5	18.9	20.2	21.6	23.0	24.3	900
1000	1.5	3.	4.5	6.0	7.5	9.	10.5	12.0	13.5	15.	16.5	18.	19.5	21.	22.5	24.	25.5	27.	1000
1100	1.6	3.3	4.9	6.6	8.2	9.9	11.5	12.2	14.8	16.5	18.2	19.8	21.4	23.1	24.7	26.4	28.	29.3	1100
1200	1.8	3.6	5.4	7.2	9.	10.8	12.6	13.4	16.2	18.	19.8	21.6	22.4	25.2	27.	28.8	30.6	32.4	1200
1300	1.9	3.9	5.8	7.8	9.7	11.7	13.6	14.6	17.5	19.5	21.5	23.4	25.3	27.3	29.2	31.2	33.2	35.1	1300
1400	2.1	4.2	6.3	8.4	10.5	12.6	14.7	15.8	18.9	21.	23.1	25.2	27.3	29.4	31.5	33.6	35.7	37.8	1400
1500	2.2	4.5	6.7	9.	11.2	13.5	15.7	17.0	20.	22.5	24.7	27.	29.2	31.5	33.7	35.0	38.3	40.5	1500
1600	2.4	4.8	7.2	9.6	12.0	14.4	16.8	18.2	21.3	24.	26.4	28.8	31.2	33.6	36.	38.4	40.8	43.2	1600
1700	2.5	5.1	7.6	10.2	12.7	15.3	17.8	19.4	22.6	25.5	28.	30.6	33.1	35.7	38.2	40.8	43.7	45.9	1700
1800	2.7	5.4	8.1	10.8	13.5	16.2	18.9	20.6	24.	27.	29.7	32.4	35.1	37.8	40.5	51.2	46.2	48.6	1800
1900	2.8	5.7	8.5	11.4	14.2	17.1	19.9	21.8	25.3	28.5	31.4	34.2	37.	39.9	42.7	53.6	48.8	51.3	1900
2000	3.0	6.0	9.0	12.	15.	18.	21.	24.	26.7	30.	33.	36.	39.	42.	45.	48.	51.	54.	2000
2100	3.1	6.3	9.4	12.6	15.7	18.9	22.	25.2	28.	31.5	34.6	37.8	40.9	44.1	47.2	50.4	53.6	56.7	2100
2200	3.3	6.6	9.9	13.2	16.5	19.8	23.1	26.4	29.4	33.	36.3	39.6	42.9	46.2	49.5	52.8	56.1	59.4	2200
2300	3.4	6.9	10.3	13.8	17.2	20.7	24.1	27.6	30.7	39.5	38.	41.4	44.8	48.3	51.7	55.2	58.7	62.1	2300
2400	3.6	7.2	10.8	14.4	18.0	21.6	25.2	28.8	32.1	36.	39.6	43.2	46.8	50.4	54.	57.6	61.2	64.8	2400
2500	3.7	7.5	11.2	15.	18.7	22.5	26.2	30.	33.4	37.5	41.3	45.	48.7	52.5	56.2	60.	63.8	67.5	2500
2600	3.9	7.8	11.7	15.6	19.5	23.4	27.3	31.2	34.8	39.	42.9	46.8	50.7	54.6	58.8	62.4	66.3	70.2	2600
2700	4.0	8.1	12.1	16.2	20.2	24.3	28.3	32.4	36.2	40.5	44.5	48.6	52.7	56.8	60.7	64.8	68.9	72.9	2700
2800	4.2	8.4	12.6	16.8	21.	25.2	29.4	33.6	37.5	42.	46.2	50.4	54.6	58.8	63.	67.2	71.4	75.6	2800
2900	4.4	8.4	13.	17.4	21.7	26.1	29.4	34.8	38.8	43.5	47.8	52.2	56.6	60.9	65.2	69.6	74.	78.3	2900
3000	4.5	9.0	13.5	18.	22.5	27.	30.5	36.	40.2	45.	49.5	54.	58.5	63.	67.5	72.	76.5	81.	3000
3100	4.5	9.3	13.9	18.6	23.2	27.9	31.5	37.2	41.5	46.5	51.2	55.8	60.5	65.1	69.7	74.4	79.1	83.7	3100
3200	4.8	9.6	14.4	19.2	24.	28.8	32.6	38.4	42.9	48.	52.8	57.6	62.4	67.2	72.	76.8	81.6	86.4	3200
3300	4.9	9.9	14.8	19.8	24.7	29.7	33.6	39.5	44.3	49.5	54.5	59.4	64.4	69.3	74.2	79.2	84.2	89.1	3300
3400	5.1	10.2	15.3	20.4	25.2	30.6	34.7	40.8	45.6	51.	56.1	61.2	66.3	71.4	76.5	81.6	86.7	91.8	3400
3500	5.2	10.5	15.7	21.	26.2	31.5	75.7	42.	46.9	52.5	57.8	63.	68.3	73.5	78.7	84.	89.3	94.5	3500
3600	5.4	10.8	16.2	21.6	27.	32.4	36.8	43.2	48.3	54.	59.4	64.8	70.2	75.6	81.	86.4	91.8	97.2	3600
3700	5.5	11.1	16.6	22.2	27.7	33.3	37.8	44.4	49.6	55.5	61.	66.6	72.2	77.7	83.2	88.8	94.4	99.9	3700
3800	5.7	11.4	17.1	22.8	28.5	34.2	38.9	45.6	51.	57.	62.7	68.4	74.1	79.8	85.5	91.2	96.9	101.6	3800
3900	5.8	11.7	17.5	23.2	29.2	35.1	39.9	46.8	52.3	58.5	64.4	70.2	76.1	81.9	87.7	93.6	99.5	104.3	3900
4000	6.0	12.	18.	24.	30.	36.	40.	48.	53.7	60.	66.	72.	78.	84.	90.	96.	101.	108.	4000
4100	6.1	12.3	18.4	24.6	30.7	36.9	41.	49.2	55.	61.5	67.7	73.8	80.	86.1	92.2	98.4	104.6	110.7	4100
4200	6.3	12.6	18.9	25.2	31.5	37.8	42.1	50.4	56.4	63.	69.3	75.6	81.9	88.2	94.5	100.8	106.1	113.4	4200
4300	6.4	12.9	19.3	25.8	32.2	38.7	43.1	51.6	57.7	64.5	70.9	77.4	83.9	90.3	96.7	102.2	109.7	116.1	4300
4400	6.6	13.2	19.8	26.4	33.	39.6	44.2	52.8	59.1	66.	72.6	79.2	85.8	92.4	99.	105.6	111.2	118.8	4400
4500	6.7	13.5	20.2	27.	33.7	40.5	45.2	54.	60.4	67.5	74.3	81.	87.8	94.5	101.2	108.	114.8	121.5	4500
4600	6.9	13.8	20.7	27.6	34.5	41.4	46.3	55.2	61.8	69.	75.9	82.8	89.7	96.6	103.5	110.4	116.3	124.2	4600
4700	7.	14.1	21.1	28.2	35.2	42.3	47.3	56.4	63.2	70.5	77.5	84.6	91.7	98.7	105.7	112.8	119.9	126.9	4700
4800	7.2	14.4	21.6	28.8	36.	43.2	48.4	57.6	64.5	72.	79.2	86.4	93.6	100.8	108.	115.2	121.4	129.6	4800
4900	7.3	14.7	22.	29.4	36.7	44.1	49.4	58.8	65.8	73.5	80.9	88.2	95.6	102.9	110.2	117.6	124.	132.3	4900
5000	7.5	15.	22.5	30.	37.5	45.	50.5	60.	67.2	75.	82.5	90.	97.5	105.	112.5	120.	127.5	135.	5000

V. Summary and Conclusions.

1. There are great quantities of calcium hydrate used for cream neutralization purposes in Oregon as well as in other states. However, calcium hydrate is not entirely satisfactory for that purpose because of its low solubility in water. Cold water will hold in solution. .37% and boiling water .075% calcium hydrate. Lime also has a "burning" effect upon the sugar of the cream. There is a need for a lime neutralizer which contains more lime in solution so that the reaction between the alkali and the acid in the cream will take place very quickly to prevent long contact in the strong alkali state, with the sugar causing burning or caramalization.

2. A calcium sucrate preparation has been made up which contains 10.8 percent of calcium hydrate in solution.

3. Three parts of sugar by weight in two parts of water added slowly into a vigorously stirred milk of lime suspension of four parts of calcium hydrate to five parts of water makes up the preparation.

4. A table has been prepared which shows the exact amount of the calcium sucrate neutralizer to use with various amounts of cream containing various amounts of acidity.

5. The preparation has been used to neutralize sour cream and has been compared with a calcium hydrate in water mixture to show its effect upon the reduction of

acid as calculated and to compare the flavor and odor of the butter made. Butter was made from 36 churnings from 18 lots of cream, 18 churnings of which were neutralized with calcium sucrate in water neutralizer and 18 with calcium hydrate in water. A comparison of the scores showed that there was nearly one point average increase in favor of the butter made from cream neutralized with the sugar-calcium preparation.

6. The importance of introducing an improved lime neutralizer in the manufacturing of butter is readily seen when we consider that approximately one half of Oregon butter is produced from sour neutralized cream, and that an improvement of one point in the score on the basis of .8¢ a pound between scores would increase the income from butter sales $112,000.

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