DESTABILIZED FAT IN HOMOGENIZED MILK

By

Bobby J. Demott

A THESIS

Submitted to the School of Graduate Studies of Michigan
State College of Agriculture and Applied Science
in partial fulfillment of the requirements
for the degree of

DOCTOR OF PHILOSOPHY

Department of Dairy

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AN ABSTRACT

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Approved G. Malcolm Trout

Extensive studies on a defect of homogenized milk, characterized by a fat ring which is accentuated by longer-than-usual storage periods, showed that the defect resulted largely from inefficient homogenization. Analysis revealed that the ring material is composed largely of de-emulsified fat. Heat shocking of fresh homogenized milk hastened fat-ring formation, but had no effect on milk bottled over 72 hours. The reliability of fat tests was lowered when a fat ring was present.

Curd tension, surface tension, heat stability and relative viscosity of the homogenized milk gave no indication as to its suseptibility to fat-ring formation. Likewise evaporation had little effect on the development of the ring. However, the higher the pasteurization temperature followed by subsequent cooling to a specific temperature, or the lower the temperature of homogenization following pasteurization at a specific temperature, the greater was the degree of fat rising on milk after homogenization.

High homogenization pressures or rehomogenization prevented fat-ring formation.

Studies on the effect of 19 different foreign fats homogenized into skimmilk showed that lanolin tended to be the most "seraphobic" of the fats studied, in that heavy cream plugs were obtained when this product was used as the sole source of fat. When tributyrin was homogenized into skimmilk no ring developed, but the product tasted extremely bitter.

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INTRODUCTION

Since the advent of every-other-day and three-times-per-week milk delivery to the home and five- or six-day plant operation, many serious problems have come into view in regard to the keeping and physical qualities of milk. Not the least of these problems is the phenomenon of a "fat ring" appearing at the surface, but attached to the sides of a bottle of milk after some of the milk has been poured from the bottle. This defect is particularly objectionable in homogenized milk. Many milk bottling plants throughout the United States have been troubled with this defect and have sought corrective measures. Many of the factors associated with fat-ring formation are not known, thus a study of the cause of this defect seems timely and worth-while.

REVIEW OF LITERATURE

Viscosity

Viscosity is often defined simply as "the resistance of a liquid to flow". However, a more complete definition was given by Glasstone (1946) as "the force in dynes that must be exerted between two parallel layers 1 square cm. in area and 1 cm. apart, in order to maintain a velocity of streaming of 1 cm. per second of one layer past the other". The viscosity is expressed in dynes per square cm. and is called a poise unit. A centipoise is one one-hundreth of a poise and water is often taken as a standard; it having a viscosity of 1.009 cp. at 20°C. (68°F.).

The first viscosity measurements on dairy products were made by Soxhlet in 1876 who found that the viscosity of whole milk increased faster than did that of water when the temperature was lowered. He attributed this change in relative viscosity of milk to the casein.

Kobler (1908) found that the viscosity of milk was due both to the fat and protein content and that this value was lowered by skimming or dilution with water. For both of these adulterations the decrease in viscosity was equal to the sum of the decrease due to each.

Pasteurization has been found by several workers (Steiner, 1901a; Taylor, 1913; Dahlberg and Hening, 1925; Woll, 1895; and Babcock and Russell 1896a, 1896b) to decrease the viscosity of whole milk, but if heated above the pasteurization temperature the viscosity was increased

(Steiner, 1901a and Taylor, 1913); this latter effect being attributed to the coagulation of albumin. However, Jensen (1912) found that the heating of milk or cream above the pasteurizing temperature did not increase its viscosity. Babcock and Russell (1896b) noticed the large drop in viscosity in cream due to pasteurization and advocated the addition of viscogen, a lime sucrate, to cream to restore its viscosity.

A relationship between total solids and viscosity was formulated by Kooper (1914) who found the viscosity number according to his standards, multiplied this number times 0.1384 and obtained the total solids for that sample of milk. He could detect as little as 5 percent added water to the milk and could also detect skimming of the sample. However, Steiner (1901b) concluded that viscosity was not due to total solids content, but was a function of the fat and solids-not-fat, and further stated that this decrease in viscosity by pasteurization was not due to the coagulation of albumin alone. Tapernoux and Vuillaume (1934a) stated that the viscosity of whole milk was between 0.0211 and 0.0264 c.g.s. units and that of skimmilk between 0.0183 and 0.0199 c.g.s. units at 15°C. (60.8°F.). These authors stated further (1934b) that skimmilk has a lower viscosity than whole milk and that the viscosity of whole milk increases more during cooling than does that of skimmilk.

The viscosity changes due to homogenization of milk or cream was first studied by Buglia (1908) who reported an increase due to this process. Bishop and Murphy (1911) reported a 20 percent slower flow rate on homogenized milk, but homogenization of skimmilk had no effect on its viscosity. Wiegner (1914) and Quagliariello (1917) also found that the viscosity of whole milk was increased by homogenization. Evenson and Ferris (1924) found that homogenization at 3500 pounds pressure per square inch increased the viscosity of both milk and cream. Homogenization pressures of 1200 pounds increased the viscosity of cream considerably, but increased that of milk only slightly. Homogenization with a single-stage valve produced increasing viscosities with increases in pressure, according to work by Mortensen et al. (1927) who also showed that the second stage lowered the viscosity compared to the single stage; that viscosity increased as clumping increased and finally, that the second stage reduced clumping of fat globules.

Bateman and Sharp (1928a) found that homogenization produced a large increase in resistance to flow in cream of 34.8 percent fat. They stated further (1928b) that agitation produced no decrease in viscosity of skimmilk or of homogenized milk; that homogenization increased the viscosity of whole milk, but not of skimmilk; and that viscosity was not a measure of total solids content. Trout, Halloran and Gould (1935) found that as the pressure of homogenization increased the viscosity decreased. These workers used single stage and pressures of 500, 1500 and 2500 pounds per square inch. Using a single-stage valve, 3000-pounds pressure, Whitaker and Hilker (1937) homogenized 20-percent cream at temperatures of 80 to 145°F. and found that the product was more viscous than when homogenized above those temperatures. Homogenization of pasteurized milk at 80°F. and 2500 pounds pressure produced a viscous product, according to Moore and Trout (1947). Caffyn (1951) found that the viscosity of homogenized milk would decrease with increases in temperature up to about 60°C.

(140^{o}F.). If heated above this temperature the viscosity increased with increasing temperature.

Doan and Minster (1933) discussed the role of fat clumping in viscosity of homogenized milk. They found that homogenization increased the viscosity of milk, but that with milk of less than 6-percent fat, clumping of fat globules was not an important factor in the viscosity increase due to homogenization.

As was brought out in Stokes' law (Sommer, 1952), viscosity is an important factor in the creaming process. Lower viscosities contribute to faster creaming, consequently have a bearing on fat-ring formation.

Surface tension

Molecules of liquids at temperatures below boiling have more attraction for one another than they do for molecules of another kind. This gives rise to a tension resembling a stretched rubber sheet over the surface of a liquid. This tension is known as surface tension. Glasstone (1946) defined surface tension as "the force in dynes acting in the surface at right angles to any line of 1 cm. length". Molecules of different liquids exert different surface tensions and this property is often used to characterize a liquid and explain some of its properties. Water, possessing a surface tension of 72.8 dynes per cm., is often used as a standard.

Among the early workers on the surface tension of milk was Burri and Nussbaumer (1909) who found that surface tension was lowered upon aging of the milk and that it was lowered abruptly when milk was cooled.

This decrease in surface tension due to cooling was permanent in character and was used to determine if milk had been previously cooled to a definite temperature. Heating of the milk to 50°C. (122°F.) for 30 minutes caused the depression almost to disappear. These authors associated this phenomenon of lowered surface tension of milk upon cooling with the solidification of fat. Bauer (1911) confirmed these results as did Quagliariello (1917), but the latter put forth the opinion that the glycerides of higher fatty acids become solid and liberate short-chain fatty acids which affect the surface tension. This worker further stated that homogenized milk had a lower surface tension than non-homogenized milk, and that homogenized milk was not affected by the cold temperatures in regard to the lowering of surface tension.

Kobler (1908) found that the surface tension of milk was increased with dilution, skimming and coagulation of casein.

Behrendt (1922) found that the fat had no essential influence upon the surface tension of milk and that milk with a reduced protein content had a considerably higher surface tension than normal milk. He stated that stalagmometric properties of milk were dependent upon the protein and dissolved organic substances particularly fatty acids. However, Dahlberg and Hening (1925) found that the surface tension of milk and cream decreased with increasing fat percentage, and it usually decreased with aging. Pasteurization usually increased the surface tension. Aging would not reduce this increased surface tension to normal.

Reid and Moseley (1926) and Reid and Garrison (1929b) have studied the effect of processing ice cream mix upon the surface tension of the product, and found that as the fat globules were broken up, more protein was taken from the plasma to coat the fat globules, thus increasing the surface tension of the mixture. The latter workers found that the surface tension increased with increasing pressures of homogenization and that the highest surface tension was found on samples containing the highest percentage of solids-not-fat.

Doan and Minster (1933) found that homogenization of milk preheated to a low temperature caused the surface tension to be decreased if the fat content was not over 5 to 7 percent. If over this level the processing increased the surface tension at first, but upon holding, it became progressively less. These workers found further that if milk was heated over 130°F., homogenization increased the surface tension if this value was determined within a few hours. If heated over 150°F. the surface tension was increased permanently. The greatest degree of fat clumping occurred when milk was heated to 145°F. and homogenized at 100°F. or lower. When heated to 180°F. and homogenized at this temperature, there was no clumping in samples containing up to 10 percent fat. The surface tension appeared to be high in samples having considerable fat clumping. Two-stage homogenization reduced surface tension compared to single-stage homogenization.

Webb (1933) found that with higher homogenization pressures and higher fat contents a greater surface tension resulted. Doan (1933) found the critical temperature for a 30-minute holding period for the increase in surface tension due to homogenization to be 129°F., and stated further that lipase was apparently found primarily in the milk plasma rather than associated with the fat.

Trout, Halloran and Gould (1935) found that homogenization of raw milk at 90°F. reduced the surface tension but when the milk was first pasteurized then homogenized at 145°F. slight increases in surface tension were noted, but these were quite small and probably negligible. The surface tension of milk was stated by Kopaczewski (1936) to be about 53 dynes per cm. and that dilution had little effect on the capillarity constant of milk. The surface tension of skimmilk was only slightly higher than that of whole milk, and aging produced a distinct increase in surface tension of milk. Agitation of milk was found to cause an increase in surface tension. These findings were somewhat contradicted by Belle (1936) who found that average surface tension of cow's milk to be 50.4 dynes and that the surface tension of fresh milk decreased about 3 dynes in 2 to 3 hours. Cardoso and Wancolle (1939) stated that the surface tension of cow's milk varied between 49.4 and 53.3 dynes per cm. as measured by the duNouy tensiometer. Boiling or shaking decreased the surface tension slightly, whereas slow pasteurization increased it slightly. Addition of water or removal of cream did not alter it. Surface tension could not be used in the control of milk so far as added water was concerned.

Shaking of raw milk while the fat was in the liquid state produced lipolysis which continued after the milk had been cooled, according to Krukovsky and Sharp (1938). Krukovsky and Herrington (1939) concluded that the rate of lipolysis seemed dependent upon the crystalline state of fat, therefore, upon previous temperature history. According to Herrington and Krukovsky (1939) milk has two lipases--one being inhibited by formalin, the other not. This observation was supported by Gould (1941) who found that lipolysis in normal milk could be stopped by formalin. However, lipolysis induced by homogenization could not be stopped by this chemical.

The agglutinin theory of creaming was supported by Sharp and Krukovsky (1939) when they found that if the agglutinin was adsorbed on to the fat globule the surface tension would be lowered. However, if it were in the plasma portion the surface tension of milk would be increased.

Tarassuk and Smith (1939) demonstrated that as milk became rancid the surface tension gradually decreased from a normal to 50 to 52 dynes per cm. to 37 to 38 dynes per cm. This was supported by Hlynka and Hood (1942) who found a correlation of 0.23 between lipase activity as measured by surface tension and odor of milk. Dunkley (1951) also found surface tension on rancid samples usually below 45 dynes and those over 46 dynes per cm. seldom were rancid. He also showed that the higher the fat in the sample the lower the surface tension.

Hetrick and Tracy (1948) found a decrease in surface tension from 44 to 44.7 dynes to 35 to 37.1 dynes per cm. due to the development of rancidity in milk, and that the surface tension of milk was not materially changed by heating. Tarassuk and Smith (1939) explain this decrease in surface tension accompanying rancidity as being due to the free fatty acids present and was of interest in the growth of _S. lactis_ because of the fact that this organism would not grow in a medium which had a surface tension of less than 35 dynes per cm. These authors (1940) found that as this organism grew in a medium of low surface tension, that the surface tension increased to approximately that of normal milk. However, Costilow and Speck (1951) showed that rancidity in milk, as shown by a lowered surface tension of about 10 dynes per cm., inhibited the growth of S. lactis, S. zymogens and L. casei, but that this inhibition was not caused by the slight reduction in pH or the reduced surface tension. They explained the growth inhibition as being due to some compound in rancid milk not yet determined. Tarrasuk (1939) stated further that rancidity could be detected sooner by measurement of surface tension than by organoleptic methods.

Krukovsky and Sharp (1940) showed that the lipolytic activity of resurfaced fat globules increased as the temperature increased, but the rate of lipolysis of fat globules with the original normal surface increased as the temperature was lowered.

The temperature necessary to inactivate lipolysis in homogenized milk was found by Gould (1940) to be over 145°F. The range of 135 to 145°F. inhibited, but did not entirely prevent lipolysis accelerated by homogenization. A complete lack of lipolytic activity was noted by Krukovsky and Herrington (1942) when milk was heated to 140°F. for 35 minutes. Hetrick and Tracy (1948) found that a temperature of 137°F. for 30 minutes was sufficient to prevent lipolysis.

The question of specificity of milk lipase was studied by Gould (1942) who found that it was a non-specific fat splitting enzyme and would produce lipolysis on a wide variety of fatty substances. He also found that homogenization would hasten lipolysis by pancreatic extract.

The effect of ammonia on the development of rancidity and surface tension lowering was studied by Castell (1942) who found that exposure of milk to an ammonia atmosphere or addition of ammonia to milk were effective in rancidity development and surface tension lowering in milk held at 5°C. (41°F.).

Gould (1944) found that the free fatty acids of butterfat obtained by churning were not responsible for the typical rancid flavor of dairy products. Fats secured from rancid milk with acid degrees as high as 11.5 did not possess a rancid flavor nor produce it when homogenized into pasteurized skimmilk, yet raw milk homogenized at 700 pounds pressure was usually rancid when the acid degree was 1.5 to 2.0. Gould and Johnson (1944) stated that the free fatty acids obtained by churning homogenized raw milk were apparently of the water-insoluble, fat-soluble type and were not responsible for the typical rancid flavor of dairy products. Further work (Gould 1947) was made, but it could not be shown that one fatty acid was involved in lipolysis more than another.

The effect of increasing amounts of a calcium caseinate solution added to milk was shown by El-Rafey and Richardson (1944) to decrease both the surface tension of milk and its foaming ability. The same effect was noted with additions of lactoglobulin or lactalbumin. Later workers (Richardson and El-Rafey, 1948) found that the stability of foam reached its minimum at 5-percent fat and at a temperature of 30 to 35°C. (86 to 95°F.).

Aschaffenburg (1946) isolated a sigma proteose which he found to be surface-active. He believed that it was the factor which reduced the surface tension of milk. In milk samples diluted with water, he found that the surface tension would not rise until the samples contained about

0.18 percent fat. At this point the surface tension increased sharply. But this was not true in undiluted samples. The addition of skimmilk raised the surface tension of milk. The heat coagulable proteins had little or no influence on the surface conditions of milk.

Whitnah, Conrad and Cook (1949) worked under the assumption that the freshly formed surface of homogenized milk should not show a surface tension greatly different than that of water. Their results, however, showed, that any great change in the surface tension of homogenized milk relative to water must have taken place before the surface age of 0.0003 seconds. The vibrating-jet method was used for these determinations.

No results have been reported in the literature attempting to correlate the lowered surface tension of milk due to the presence of fatty acids to the development of fat ring on homogenized milk.

Curd tension

A large amount of work has been done on the curd tension of milk since Hill (1923, 1928, 1931) recognized that milk of different cows varies in the hardness of its curd. This worker found that the softer the curd the more easily digested was the milk. Soft-curd milk was found to agree digestively with babies who otherwise could not retain cow's milk in their stomachs. Hill (1923) found that skimmilk had a higher curd tension than the whole milk from which it came, thus leading to his conclusion (1928) that butterfat softened curd. He found further (1928) that pasteurization had little effect upon curd tension but that boiling would soften the curd. This is supported by Doan and Welch (1934) who found that heating lowered the curd tension of milk.

Johnson and Weisberg (1932) found that homogenization would produce soft-curd milk and obtained a patent on this process. This was confirmed by Halloran (1932), Weisberg, Johnson and McCollum (1933), Theophilus, Hansen and Spencer (1934), Doan and Welch (1934), Whitaker and Hilker (1937), Tretsven (1939), Krauss, Sutton, Burgwald, Washburn and Bethke (1941), Doan and Mykleby (1943), Judkins (1943) and Spur (1948). Doan and Mykleby (1943) found that the curd tension dropped sharply upon homogenization, even at low pressures, but increasing pressures caused only a small additional reduction which became negligible when pressures between "medium" and "high" were used (approximately 2500 pounds with plug-type valves). These workers also found that milk sufficiently homogenized to reduce its curd tension to a practical minimum was found to have a Farrall index of 12 or less. Judkins (1943) also reported that homogenization could be quite inefficient before the curd tension was affected.

Weisberg, Johnson and McCollum (1933) found that higher concentrations of calcium and phosphorus were present in hard-curd milk. Fat, casein and calcium phosphate, by means of their concentrations and manner of dispersion, seemed to control the curd character.

The higher the casein content of the milk the harder the curd of that milk. This fact was supported by Weisberg _et al_. (1933), Doan and Welch (1934), and Riddell, Caufield and Whitnah (1936); the latter workers finding a correlation of 0.76 plus or minus 0.04 between curd tension and total protein content of milk.

Doan and Welch (1934) believed that, since milk of hard curd character has a higher casein content, it was retained in the stomach longer for digestion and this retention was due to the higher casein content, not because of the fact that the curd was harder.

Agitation of milk below churning temperatures, according to Palmer and Tarassuk (1936), did not cause the milk to have a reduced curd tension. Butterfat globules did not adsorb any substances from skimmilk which reduced the curd tension of milk plasma.

Sonic vibration can reduce the curd tension of milk and this effect was explained by Chambers (1936) as being due to increased fat dispersion.

The action of rennet in the curdling of milk was explained by Sohngen, Weiringa and Pasveer (1937) as being due to the adsorption of the enzyme on casein particles to precipitate the casein. The more swollen the casein particle the slower the action of the rennet.

Berggren (1938) found that 2 percent gelatin added to milk produced soft curd and showed that at a pH of 5.7 to 5.9, milk showed the greatest curd tension.

Palmer and Tarassuk (1940) stated that "a normal rennet clot may be completely prevented by emulsifying a small amount of diglycol laurate into raw milk at room temperature, aging the emulsion in the cold and adding rennet at 35°C." (95°F.). Under these conditions the surface tension and the pH were lowered when the curd tension was decreased. This, they explained, was due to the liberation of lauric acid by lipase. Pasteurized milk exhibited no such lowering of the above properties.

Tarassuk and Richardson (1941) found that the addition of lauric, myristic or palmitic acid to milk inhibited rennet coagulation if held at a low temperature, but when heated to the melting point of the fat acid, the milk again possessed its normal properties.

Palmer and Hankinson (1941) found that the addition of capric, lauric, or oleic acid inhibited the rennet clotting of milk after aging, but this inhibition was overcome somewhat by heating the milk or by the addition of calcium chloride. The adverse effects of oleic acid occurred without aging and were not overcome completely by the same heat treatment.

One of the latest studies on the nutritive value of low-curd-tension milk has been made by Hadary, Sommer and Gonce (1942, 1943) who found no relationship between the gastric emptying time and the curd tension of milk and milk products.

Curd tension, as explained above, is decreased by homogenization of the milk. No work has been reported to date, attempting to correlate the curd tension with degree of fat rising on homogenized milk, or with the character of the fat present in the milk.

Heat coagulation

Sommer and Hart (1919, 1922, 1926) advanced the theory of salt balance upon the heat-coagulation time of milk, concluding that casein apparently required a definite optimum calcium content for its maximum stability. This calcium content of casein is largely controlled by the amount of magnesium, citrates and phosphates present in the milk. These workers also found no relation between titratable acidity and heat coagulation. They noted that the addition of water increased coagulation time.

Rogers, Deysher and Evans (1921) found that the heat stability of evaporated milk could not be determined by the stability of the fresh product and that the salt balance was not as important in evaporated milk as it was in fresh milk.

Studying homogenization pressures on sweet cream, Webb, and Holm (1928) found that increases in pressures decreased heat stability, that increases in fat content decreased heat stability, and that increases in viscosities in preheated homogenized cream was accompanied by a decrease in heat stability.

Doan (1929) found that the proteins of dairy fluids were destabilized by homogenization when fat was present and this effect increased with fat concentration and with the efficiency of homogenization. The hydrogen-ion concentration was increased, but the loss in protein stability was only partially due to this reaction. The fat clumping tendency paralleled the loss of stability of the proteins, but failed to do so when such loss was due to added or developed acidity. He signified there was some indication that the amount of calcium ions or amount of soluble calcium was a prime factor in both fat clumping and protein stability. Later (1931) he stated that fat clumping was probably the most important factor affecting stability of homogenized cream toward heat. Mechanical destruction of clumps increased stability and dual homogenization was found to reduce viscosity, but to increase heat stability. Heat stability was found to increase in some cases by the addition of solids-not-fat but in excess of a certain amount decreased heat stability.

Webb (1931) showed that homogenization of cream decreased its stability toward coagulation by heat and rennet. Double homogenization with the second stage at about 500 pounds per square inch was beneficial in increasing heat stability of creams containing over 15-percent butterfat. If the second stage were over 2000 pounds per square inch the heat stability was decreased when compared to single-stage treatment.

Concentrated skimmilk exhibited a lower heat stability than the fresh product according to Webb and Holm (1932) who found that this stability could be predicted by the heat stability of the uncondensed product. They stated further that the salt balance was important especially in products of low solids-not-fat concentrations.

Holm, Webb and Deysher (1932) could find no correlation between heat coagulation time and the salt balance as determined by analysis. They noted no relationship between heat stability of the fresh product and that of the evaporated milk.

An increase in protein stability, as measured by the alcohol number, due to dual homogenization as compared to single-stage homogenization was noted by Doan and Minster (1933). This effect was more pronounced as the fat content increased.

Trout, Halloran and Gould (1935) found that the protein stability, as measured by the alcohol number, was decreased by homogenization whether the milk was raw or pasteurized, but that the pasteurized-homogenized milk was more stable than the raw-homogenized product. As the pressure of homogenization was increased the stability decreased, but was less pronounced in pasteurized milk than in raw homogenized milk.

Cole and Tarassuk (1946) showed that a fairly straight-line relationship existed between coagulation temperature and the logarithm of the coagulation time.

No information is in the literature relative to the decreased heat stability of homogenized milk in relation to homogenization efficiency, or fat-ring formation on the milk.

Heating of homogenized milk

A vast amount of work has been done on the effect of heat on certain properties of milk. That by Gould (1951) was especially noteworthy. However, no studies related directly to the effect of heat on fat rising on homogenized milk have been reported in the literature.

Recirculation of milk through the homogenizer

The procedure of passing milk through a homogenizer more than once is not a practical problem, but of academic interest only. Trout and Sheid (1941) passed raw milk through a homogenizer five times at a temperature of 40^{o}F. using 5000 pounds pressure and found that this procedure failed: a) to disperse the fat; b) to increase the titratable acidity; or c) to alter the flavor after 96 hours of storage. These workers found a 17^{o}F. increase in temperature due to one passage of milk through the homogenizer.

Halloran and Trout (1932) recorded as much as 8^{o}F. increase when milk was homogenized at 145^{o}F. at a pressure of 3500 pounds. Mohler (1938) found, in commercial operation, a 6-to 8^{o}F.-increase in temperature due to passage of milk through a homogenizer at 3500 pounds pressure.

A temperature rise of 10.8°F. could be expected at 3500 pounds pressure if all the energy of the homogenizer went to heating the milk, according to calculations by Wenrich (1946).

A temperature rise of the milk due to homogenization was noted by Roadhouse and Irwin (1950), the rise in temperature being in direct relationship to the pressure applied. These authors further gave a formula for calculating the temperature rise of a liquid due to the pressure applied. Herrington's calculations (1948) showed that the heat energy equivalent to the work done in homogenizing one pound of milk at 5000 pounds pressure was 14.3 BTU, enough to raise the temperature about 15.6°F.

Pasteurization and homogenization temperatures

Homogenization temperatures have been studied a great deal and were summarized by Trout (1950) who stated that so long as the fat was in a liquid state and the enzyme lipase inactivated, a satisfactory product could be produced.

Filled milks

A large amount of work has been done on the nutritional value of butterfat as compared to other fats, but Hilditch (1940) summed up all this and stated that glycerides of all varities were digested equally well so long as the glyceride was in a liquid state at body temperature. He stated further that certain unsaturated fatty acids, especially linoleic acid appeared to be essential to health but apparently were not synthesized by, at all events, certain animals.

"Milks" containing a part or all of their fat in the form of fats other than butterfat are being manufactured for human consumption, but no scientific literature is available at this writing on the subject.

EXPERIMENTAL PROCEDURE

Homogenization index

The homogenization index as given by the United States Public Health Service Milk Ordinance and Code (1953) and used in this study was calculated as follows:

$$\frac{\text{Fat test on top 100 ml. of milk from a quart bottle} - \text{Percentage fat in the remainder}}{\text{Percentage fat in the top 100 ml.}}$$

X 100 = Homogenization index.

The homogenization index was determined on 196 quart bottles of homogenized milk obtained from four different sources and stored at 40°F. Daily, two bottles of milk were heat shocked. They were brought out of the refrigerated storages and allowed to remain at room temperature for 30 minutes after which they were returned to the 40°F. cooler. The following day, the top 100 ml. and the remaining portions from these two samples plus two control samples were analyzed for fat using the Gerber procedure, as outlined by Roadhouse and Henderson (1941). The Gerber procedure gave clearer fat columns on homogenized milk fat tests than did modified Babcock procedures.

The top 100 ml. from one of the heat-shocked samples and from one of the control samples were taken in such a manner as to permit the inclusion of the fat-ring material in the milk of the lower portion of the bottle, such as might be done by careless technicians.

The presence or absence of fat-ring formation on bottled homogenized milk was noted as the homogenization index studies were being made.

Analysis of fat-ring material

The fat-ring material was analyzed for fat by the Mojonnier method using the procedure outlined by Mojonnier and Troy (1925) for cream testing over 25-percent butterfat. The total solids content was determined by the Mojonnier test, whereas the nitrogen determination was made by means of the Kjeldahl tester as outlined by Menefee and Overman (1940). The fat-ring material was obtained from three different sources of homogenized milk stored for one week at 40°F.

Creation of fat ring

Eighty gallons of milk were heated to 190°F. and held at this temperature for 30 minutes in a 300-gallon pasteurizer. Forty gallons were cooled immediately to 40°F. and stored at this temperature for 24 hours. The other 40 gallons were divided into seven lots and each lot homogenized immediately at a definite temperature, after which the samples were cooled to 60°F., bottled and stored at 40°F. for one week. The homogenization pressure used was 3000 + 500 pounds (3000 pounds on the first stage, 500 on the second) and the temperatures were 190, 170, 150, 130, 110, 90 and 70°F. After 24 hours at 40°F. the first half of the 80-gallon sample was warmed to these same homogenization temperatures and homogenized at the same pressures after which they were cooled to 60°F., bottled and stored for one week at 40°F. After one week at 40°F. all samples were examined for fat-ring formation and the fat-ring material taken from the surface of the milk with a spatula and analyzed for fat, total solids and ash content. As a control, a pasteurized nonhomogenized sample was used.

The intensity and nature of the fat ring on the bottles of homogenized milk were examined by means of the unaided eye. The degree of fat rising was expressed as follows:

+	slight fat ring
++	distinct fat ring
+++	prominent fat ring
++++	cream plug.

<u>Study of the effect of evaporation on ring formation</u>

To study the influence of evaporation on fat-ring formation, various concentrations of gelatin solutions were put into test tubes, weighed, and allowed to remain at room temperature for five days. The surface of the gelatin was exposed to the air in the laboratory for the entire five-day period. The amount of evaporation and presence of ring formation was noted daily.

To check further the effect of evaporation upon ring formation, 11 test tubes were filled with milk of various compositions. The addition of nonfat dry milk solids or water provided the means of varying the composition of the milk. These open tubes, containing approximately 10 grams of milk, were stored uncovered at $40^{\circ}F$. in a household refrigerator except when they were warmed to room temperature for weighing. The samples were weighed daily for five days, the weight loss recorded and the presence of fat ring noted.

Study of the effect of addition of washing powder to homogenized milk upon fat-ring formation

A one-percent solution of "Blue Ribbon" washing powder, a common, surface-active detergent in use in the dairy industry, was added in various amounts to homogenized milk. After storage at 40°F. for one week the samples were examined for fat ring.

Study of the effect of heating homogenized milk upon some of its physical properties

Two portions of 3.7-percent milk, pasteurized by the holding process and homogenized at 2500 pounds per square inch at 130°F., was obtained directly from a 600-gallon-per-hour homogenizer. One portion was heated to 165°F. and held for one-half hour at that temperature. The heated milk was then cooled to 60°F., sampled, bottled and held for one week at 40°F. when it was then examined for the presence of fat ring. The control portion was treated in a like manner with the exception of the heat treatment.

The heat stability of the samples was determined by sealing one ml. of the milk in eight mm. pyrex tubes and immersing them in an oil bath tempered and held at 120°C. (248°F.). The tubes were rotated occasionally while in the oil bath. The lapse of time between the insertion of the tube into the oil bath and the first sign of coagulation or precipitation was considered as the heat stability of that sample.

Curd tensions were made according to a method outlined by the American Dairy Science Association (1941) with the exception that the reading was taken after the knife penetrated the surface of the curd and became stabilized. A Submarine Signal Company curd tensiometer was used to record the curd tension.

Surface tension measurements were made on the samples by use of a du Nouy surface tension apparatus.

<u>Study of the effect of composition upon some of the physical properties of homogenized milk</u>

Twelve quarts of homogenized milk obtained from the College Creamery were treated by substituting various amounts of milk with water, thus lowering the total solids concentration. The samples were stored for one week at 40°F. and then examined for fat ring.

In effect, the above was repeated, but using this time only three quarts of milk. The first was used as a control; to the second was added nonfat dry milk solids to bring the solids-not-fat to nine percent, and to the third was added water to adjust the solids-not-fat to eight percent. These samples were stored for one week at 40°F. after which they were examined for the presence of a fat ring.

The ratio of total solids to water in milk, as affects the physical properties of the milk, was studied using the methods previously described for the determination of surface tension, curd tension, heat stability and the presence of fat ring.

Milk pasteurized by the holding process was obtained from the College Creamery before it was cooled, brought to the laboratory and divided into five portions. The first portion was homogenized with no adulterant added. The other four portions had various quantities of water, cream or nonfat dry milk solids added before they were homogenized.

All samples were homogenized at 2000 + 500 pounds pressure per square inch in a 75-gallon-per-hour, Manton-Gaulin, 2-stage homogenizer. The samples were then cooled and examined.

In the case of added solids-not-fat, viscosities were determined by use of a Brookfied Synchro-Lectric viscometer which expresses the viscosity of a fluid product as "relative viscosity" when measured at 20°C. (68°F.). The viscosity measurements were made on the product after it had been held at about 40°F. for 24 hours and tempered to 20°C. (68°F.) for 30 minutes.

The fat and solids-not-fat were determined on the unadulterated sample by the use of the Mojonnier test. These data were used to calculate the fat and solids-not-fat for the adulterated samples. The homogenization temperature was 135°F. For the addition of solids-not-fat to the milk, a 25-percent aqueous concentration of nonfat dry milk solids was used. Various amounts of this suspension were then added to the milk before homogenization. The Farrall index (1953) was determined on those samples containing added solids-not-fat. This index is equal to the number of fat globules of two microns in diameter which would have the same volume as the fat globules which exceeded two microns in diameter.

<u>Study of the effect of various homogenization procedures upon fat rising on homogenized milk</u>

The effect of the procedure used in homogenization upon the amount of fat rising on a bottle of homogenized milk was determined by treating pasteurized nonhomogenized milk as follows:

1. Milk was poured into the inlet tank of the homogenizer and homogenized at 135°F., bringing the pressure up to 2000 + 500 pounds per square inch by recirculating the milk through the homogenizer until this pressure was reached.

2. The homogenizer was allowed to run empty, and with the pressure still applied to the values, milk at 135°F. was admitted to the inlet tank.

After homogenization the milk was cooled, bottled and stored at 40°F. for one week and then examined for the presence of fat ring.

<u>Study of the effect of homogenization pressures upon some of the physical properties of homogenized milk</u>

Vat-pasteurized, noncooled, nonhomogenized milk was obtained from the College Creamery, brought to the laboratory and homogenized at 2000 + 500, 3000 + 500, 4000 + 500, 5000 + 500 and/or 6000 + 500 pounds per square inch at temperatures ranging from 110°F. to 130°F. The physical constants were determined on these samples as previously described.

<u>Study of the effect of recirculation of milk through the homogenizer upon some of the physical properties of the milk</u>

Five gallons of milk were homogenized at 2000 + 500 pounds per square inch at starting temperatures ranging from 102°F. to 135°F. After homogenizing one gallon, the remainder was recirculated through the homogenizer, one-gallon samples being removed after 5, 10, 20 and 30 minutes respectively. Each sample was then cooled in running water and saved for observation.

Study of the effect of pasteurization and homogenization temperature upon fat-ring formation

The effect of pasteurization temperature and homogenization temperature upon the degree of fat-ring formation on homogenized milk was studied using 20 gallons of raw milk divided into four lots of five gallons each. The first lot, pasteurized at 143°F. for 30 minutes, was divided into two portions, one of which was homogenized at pasteurization temperature and the other at 110°F. Likewise, the second lot, heated to 150°F. and held for 30 minutes, was divided and homogenized at the pasteurization temperature, and at 110°F. The third five-gallon lot was heated to 160°F., held for 30 minutes, halved, and homogenized at that temperature and at 110°F. Similarly, the fourth lot was heated to 175°F., held for 30 minutes, half of it homogenized at that temperature and the other half at 110°F. All milks were homogenized at pressures of 3000 + 500 pounds per square inch. After homogenizing, the milks were cooled in a five-gallon can with tap water, and when the temperature was below 60°F., several samples were taken from each lot, put in quart bottles, stored at 40°F. and examined one week later for the presence of fat ring. The United States Public Health Service homogenization index was also determined on these week-old samples by determining the fat content by the modified Babcock method as recommended by Trout and Lucas (1947). On two of the trials a Farrall index was made of the sample the same day it was homogenized, and the United States Public Health Service homogenization index determined on the sample after 48 hours.

For further study on the effect of pasteurization and homogenization temperature, ten-gallon samples of raw milk were heated to 190, 170 or

$150^{\circ}F$. and held for 30 minutes and each sample divided into several lots and homogenized at different temperatures using 3000 + 500 pounds per square inch pressure in each case. One lot was homogenized at the pasteurization temperature and one at each interval of $20^{\circ}F$. below the pasteurization temperature down to and including $110^{\circ}F$. After homogenization, all samples were cooled to below $60^{\circ}F$., bottled in quart bottles, stored at $40^{\circ}F$. for one week and examined with the naked eye for degree of fat-ring formation.

Eighty gallons of milk were treated as described under "Creation of fat ring" in the section entitled "Analysis of fat-ring material". After one week of storage these were then examined for degree of fat-ring formation.

Study of the influence of the composition of the fat present upon some of the physical properties of the homogenized product

The influence of the various fats upon certain properties of homogenized milk was determined by melting and homogenizing into skimmilk, fats of various sources and compositions. The skimmilk was obtained from the College Creamery and analyzed for total solids. Fats were added to the skimmilk in such amounts as to adjust the fat content of the whole to 3.5 percent. With the exception of the first analysis in which unsalted butter was used, butter oil served as a control. The skimmilk was heated to $140^{\circ}F$., the fat added and the whole then homogenized at 3000 + 500 pounds pressure per square inch through the homogenizer previously described. One-gallon samples were used in each case except in the case of trimyristin. After homogenizing, all samples were set in cold running water, cooled and analyzed for surface tension, curd tension and heat stability. Samples were set aside for viscosity determinations 24 hours later and for observations for fat ring after storage of one week at 40°F. In some cases the flavor scores and criticisms were made and recorded.

EXPERIMENTAL RESULTS

Homogenization index

The influence of heat shocking bottled homogenized milk upon the homogenization index after 24 hours storage is shown in Table I. The data show that for milk 24 or 48 hours old, heat shocking will tend to increase the index indicating that more fat is poured off in the top 100 ml. portions in these samples than in those that did not receive the warming treatment. When milk became 72 hours old the previous warming treatment in general did not increase the homogenization index, but remained below the index for the samples which received no heat treatment. This can be seen graphically in Figure 1.

Influence of sampling procedure upon homogenization index

The influence of sampling technique is shown in Table II. Here, in the "A" samples, an effort was made to allow the fat ring to adhere to the bottle, excluding it from the test portions, whereas, in the "B" samples, an attempt was made to include the ring, where one occurred, in the bottom portion. This latter procedure is the one likely to be followed by inspectors unless special precautions are taken. Thus, it can be expected that the "B" technique would show a lower index. This is borne out by the data. The effect of heat-shocking is brought out in Figures 2 and 3. These data show a greater difference in the index due to sampling procedure in the samples which were heat-shocked than in those which were not so treated, showing that this heat treatment tends to accelerate the fat rising which appears to be highly essential in fat-ring formation. However, on those samples not heat-shocked, the fat ring is compact and firm and will not remix readily into the samples. When the samples are 72 to 144 hours old a leveling off of the index occurs, and the samples show similar compactness and behavior of the fat ring, both in the control and heat-shocked samples.

Fat-ring analysis

The nature and appearance of the fat ring are shown in Figures 4, 5 and 6. The first photograph shows the fat ring immediately after some of the milk was poured from the bottle; the second after a lapse of about two minutes at room temperature; and the third after an additional time lapse of about two minutes.

Three samples of fat-ring material from homogenized milk, analyzed for fat by the Mojonnier test, were found to contain 58.6, 42.8 and 57.0 percent fat, respectively. The first of these three samples was examined for solids-not-fat and protein. It was found to contain 5.65 percent solids-not-fat, 1.5 percent protein and, by difference, 4.15 percent of lactose and ash. An interesting observation was made during the Mojonnier testing. When water was added to the sample in the Mojonnier dish and the sample warmed, free fat floated to the surface, indicating the sample contained some non-emulsified or churned fat.

Data in Table III show the percentage of fat, solids-not-fat and ash in the fat-ring material resulting from milk processed in different manners. After heating at 190°F. for 30 minutes, milk was homogenized at various temperatures, cooled to 60°F., bottled and stored for one week. Half of the original sample was held at 40°F. for 24 hours before homogenizing. These latter samples were heated quickly to the homogenization temperature and immediately homogenized, cooled and bottled.

The fat-ring material was taken from quart bottles of week-old homogenized milk with a spatula and warmed slightly to facilitate mixing. However, difficulty in thorough mixing of the sample was encountered. Even though they were poured rapidly from one container to another, the de-emulsified fat rose rapidly to the surface when the weighing procedure was started. This could easily account for some of the seemingly variable data reported in Table III, especially in regard to the solids-not-fat content.

The fat and solids-not-fat analysis on the ring material does not show that either those samples homogenized at high temperatures or those at lower temperatures contain a ring material with a greater percentage of fat or solids-not-fat.

The percentage of ash in the ring material increased as the temperature of homogenization increased in both series of samples. This shows that fat-ring material developing on those samples of milk homogenized at the lower temperatures have less mineral content than those rings developing on milk homogenized at higher temperatures. This lower concentration of mineral matter is probably the cause of a more tenacious ring forming at the lower temperatures of homogenization due to a lower concentration of stabilizing agent.

Evaporation of water from gelatin solutions in test tubes

Gelatin concentrations of 0.0 to 10 percent were weighed into test tubes, allowed to stand uncovered in the laboratory for 120 hours and the loss in weight and the presence of ring material on the walls of the test tubes were noted daily. The percentage of daily and total weight losses at each concentration during the 120-hour exposure to room atmosphere are given in Table IV. In general, the evaporative capacity of the solutions at the concentrations used were similar. However, the most evaporation occurred when no gelatin was added.

Data on the evaporative capacity of lesser concentrations of gelatin in water solutions are presented in Table V. Data in Tables IV and V can not be combined because the trials were conduced under different atmospheric conditions. The tube containing 0.5-percent gelatin is not included in Table V because it showed an abnormally low evaporative capacity. The series 0 to 1.0-percent concentration was repeated and the 0.5-percent solution showed normal evaporative capacity, therefore the first determination was considered in error.

A study of ring formation on these tubes showed that, in the higher concentrations of the gelatin, a plug of the colloid actually formed all across the surface of the solution and the tubes could be inverted without spillage. Those tubes containing the lower concentrations of gelatin exhibited some plug formation, but were progressively less firm as the concentration was lowered.

Evaporation of water from milk

Data showing the variation in the composition of 11 test tubes of homogenized milk and the percentages of weight losses in each tube are recorded in Table VI. These data indicate that milk loses approximately the same weight regardless of the percentage of total solids present. One noticeable point in this table was the variability of weight loss during exposure. This was probably due more to the atmospheric conditions of exposure than to any factor in the milk.

The observations on the presence of fat ring on these samples showed that the ring became more prominent with decreasing percentage of solids and with increasing length of holding time.

Influence of addition of surface-active washing powder to homogenized milk upon fat-ring formation

The prominence of fat-ring formation in quart bottles of homogenized milk which had varying amounts of a one-percent solution of "Blue Ribbon" washing powder substituted for that same quantity of milk is shown in Table VII. The data show that, in the concentrations used, this compound has no influence upon the prominence of fat-ring formation in quart bottles of homogenized milk.

Influence of heating homogenized pasteurized milk upon its physical properties

Nine trials were made on the effect of heating pasteurized homogenized milk upon some of its physical properties. The data are reported in Table VIII. The heat stability of the heated samples was lower than that of the milk not heated. The curd tension was lowered in most cases, although it was below 15 grams in all cases. The surface tension of the heated samples was slightly below that of the samples given no heat treatment other than pasteurization. Both surface-tension and curd-tension data result from averages of three or more trials.

The data show no definite indication that the fat-ring defect is caused by heating the milk to a high temperature after homogenization. One-third of the trials showed more fat-ring development on the heat-treated samples; one-third showed more ring material on the unheated samples, while the other one-third of the trials showed no greater prominence on either sample.

Thus, while heating does cause some changes in physical properties of the homogenized milk, as evidenced by heat-stability changes as well as in surface-tension and curd-tension alterations, the exposures used in these trials neither accelerated nor prevented the formation of fat ring on homogenized milk.

Effect of composition upon some physical properties of homogenized milk

1. Addition of water to milk

A case of homogenized milk in quart bottles was treated by substituting increments of water for milk in the range of 0 to 16 percent. These were stored at 40°F. for one week and the prominence of fat ring noted. Observations showed that when greater amounts of water were substituted, the ring became more prominent.

Three bottles of homogenized milk testing 8.54 percent solids-not-fat were obtained from the College Creamery. The first was used as a control, the second was adjusted to eight percent solids-not-fat with water, and the third adjusted to nine percent solids-not-fat with non-fat dry milk solids. After standing at $40^{o}F$. for one week, these samples were examined for fat ring. The sample to which the solids-not-fat was added showed little fat rising; the control showed some creaming, but the one to which water had been added showed the most fat rising.

Four series of tests were made on the effect of added water in various amounts to pasteurized milk before homogenization upon the surface tention, curd tension and heat stability of the milk. The results are recorded in Table IX.

Addition of water to milk in the quantities used in this study showed very little effect on the surface tension of the milk. All surface tensions were between 46.3 and 46.9 dynes per cm.

Heat stability tests show that, in general, as more water is added the time necessary to precipitate the protein by heat becomes longer. However, some variation occurred; the unadulterated samples in two cases were more stable than some in the same series which had water added.

Curd-tension studies show that with the first addition of water (one pint per gallon of milk) the curd tension increased, but in general, thereafter, the value decreased with decreasing percentage of total solids in the milk. This is shown clearly in Figure 7 in which the average of four trials is recorded.

The data on fat-ring formation show that, in general, as the percentage of water increases the tendency for the fat ring to form is more pronounced.

2. Addition of cream to milk

The results of added cream upon the surface tension of homogenized milk, recorded in Table X, show the surface tension to be quite constant. Thus, although one inconsistency was noted, the surface-tension values of the homogenized milk remained unaltered due to added cream before the homogenization process.

The addition of cream at the levels used in this study was found to affect the curd tension of the homogenized milk very little. The curd tensions of all samples were under 12 grams. However, those samples having the most fat have, on the average, lower curd tensions than the others (Table XI).

The effect of added cream upon heat stability of homogenized milk is shown in Table XII. A definite decrease is noted with the first addition of cream. This is brought out by the average of the samples resulting from no addition of cream and that resulting from the addition of 0.5 lb. of cream per gallon of milk where a decrease of 16 minutes in heat stability was noted. After this first addition of cream, little decrease in heat stability was noted upon further additions of cream.

The presence of fat rings in various intensities were noted on each sample (Table XIII). As the fat content was increased, the presence of the fat ring or cream layer was more in evidence. Some samples had such a tough cream layer on the surface that it was impossible to pour the milk from the bottle until this layer was broken.

3. Addition of solids-not-fat

Surface-tension data show that as the solids-not-fat portion of the milk increased there was a slight tendency for the surface tension to increase (Table XIV). The averages of six trials show an increase from 46.36 dynes in the original milk to 46.78 dynes in the product after two pounds of a 25-percent concentration of nonfat dry milk solids in water were added per gallon of milk. Individual samples varied somewhat, but the average of the six shows a small increase as the solids content is increased.

The addition of solids-not-fat to the milk before homogenization increased the curd tension of the milk (Table XV). The data showed an increase in curd tension from 14.3 grams in the unaltered sample to 35.8 grams in those samples having two pounds of a 25-percent concentration of solids-not-fat in water added per gallon of milk before homogenization.

The addition of solids-not-fat to milk before homogenization decreased the heat stability of the homogenized milk (Table XVI). The average heat stability of the six control samples was 151.3 minutes at 120°C. That of the treated samples was 118 minutes. The relationship between the curd tension, heat stability and solids concentration of milk is shown graphically in Figure 8.

A slight increase in viscosity of homogenized milk due to added solids-not-fat is shown in Table XVII. When solids-not-fat are added in the proportion of 0.5 pound of a 25-percent concentration per gallon of milk, there is a small decrease in viscosity in four cases out of the six trials conducted. However, additions of the solids-not-fat in greater proportions than 0.5 pound of the concentration per gallon of milk causes the viscosity to increase over the control sample. Noted also is the fact that in the unadulterated samples, the milks with higher fat concentrations have greater viscosities than those of lower fat contents.

The relative degree of fat rising on homogenized milk which had previously been fortified with added nonfat dry milk solids is shown in Table XVIII. Most samples show a greater degree of fat-ring formation in those cases where a greater amount of solids-not-fat was added; the averages show a definite trend in this direction.

Various homogenization procedures as affects fat rising on homogenized milk

The data on homogenization procedures show that the homogenizer may be permitted to operate empty under full pressure before admitting the milk, and yet the milk will be homogenized adequately so far as freedom from a fat-ring development after one week is concerned.

Effect of homogenization pressures upon some of the physical properties of homogenized milk

Data relative to the curd tension, surface tension and heat stability of milk resulting from different homogenization pressures are recorded in Table XIX. As the pressure is increased above 3000 pounds per square inch, the curd tension, in general, tends to increase also, but this is quite irregular and not true in each individual case. Surface tension also is shown to be slightly higher on those samples homogenized at higher pressures, though this is so slight as to be rather insignificant.

Heat stability tests show merely that some other factor together with homogenization pressure affects this property. One series shows a constant drop in stability, the next a constant rise and the last two series show erratic results.

Relative viscosity measurements show that as the homogenization pressure is increased, the viscosity will decrease (Table XX). The results within a series are somewhat erratic, but when all series are averaged this relationship can be seen clearly. The fat-ring observations show that the higher homogenization pressures are helpful in preventing this condition, though only in the case of high-fat milk was this defect particularly objectionable.

Effect of recirculation of milk through the homogenizer upon some of the physical properties of the milk

Data on the influence of recirculation of milk through a Manton Gaulin, Model E, 75-gallon-per-hour, 2-stage homogenizer operated at 2000 + 500 pounds pressure per square inch are recorded in Tables XXI, XXII and XXIII.

Data show that the curd-tension value is lowered somewhat with the additional homogenization afforded by recirculation of the milk for five minutes. However, beyond this, further homogenization did not lower the curd tension of the milk.

The data on surface tension show that this physical property is quite constant regardless of the recirculation of milk through the homogenizer. The lowest value observed was 46.1 dynes and the highest was 47.2 dynes; not a large difference even in the extremes.

The viscosity of homogenized milk, as measured after 24 hours storage at 40°F., was quite constant regardless of recirculation time up to the 30-minute recirculation period. At that time the viscosity value tended to be higher than on any of those samples recirculated shorter periods of time (Tables XXII and XXIII). Thus, recirculation of milk through the homogenizer for 30 minutes appeared to cause a slightly higher viscosity than when the milk was not recirculated or when recirculated only up to 20 minutes.

Homogenization definitely decreases the heat stability of the milk as measured by immersion of the milk in an oil bath at 120°C. (Tables XXII and XXIII).

The presence of fat ring on the milk after seven days storage at 40°F. is shown in Tables XXII and XXIII. As would be expected, the ring material did not appear on those samples rehomogenized the longest period of time, becoming less prominent as rehomogenization progressed.

The mechanical treatment afforded by the homogenizer can be converted to heat energy as shown in Table XXIII. More of this energy is converted to heat at the lower temperatures of homogenization. Considering the 22 times which the milk was passed through the homogenizer at the conclusion of the recirculation, this temperature increase averaged only about 1.5°F. per passage through the machine.

<u>Pasteurization and homogenization temperatures as affects efficiency of homogenization</u>

Data relative to the degree of fat rising on milk pasteurized and homogenized at different temperatures are shown in Table XXIV.

Apparently milk pasteurized at 143°F. can be homogenized at 110°F. with less formation of a heavy cream layer or plug on the surface after storage of one week than when higher pasteurization temperatures are used. However, if milk is pasteurized at 150°F. or above and is homogenized at 110°F. the likelihood for cream plug development is great.

The United States Public Health Service homogenization indices determined on this week-old milk are shown in Table XXV. The milk homogenized at 110°F. shows a greater index in every case than that milk homogenized at the pasteurization temperature. This difference is greater at the higher temperatures of pasteurization.

The degree of fat rising noted on milk pasteurized at 150, 170 and 190°F. and homogenized at intervals of 20°F. down to 110°F. after storage of one week at 40°F. is recorded in Table XXVI. Four trials were made and the average of these showed clearly that as the temperature of homogenization was lowered from the temperature of pasteurization the development of the fat ring became more pronounced. When the milk was homogenized at 110°F. after pasteurization at 150, 170 or 190°F. a plug developed in every case to such an extent that the milk would not pour from the bottle. Homogenization of milk at the pasteurization temperature lessens the formation of this fat ring regardless of the temperature in the range of 143 to 190°F.

The Farrall indices determined on the fresh homogenized milk and the United States Public Health Service homogenization indices determined on the same milk 48 hours after homogenization are shown in Table XXVII. The milks were heated to 150, 170 or 190°F. and held for 30 minutes at that temperature and homogenized at 20°F. intervals down to 110°F. The data show that the Farrall index was not a reliable guide as to the homogenization efficiency so far as fat rising was concerned, although some correlation was noted between these indices. The United States Public Health Service homogenization indices increase as the temperature of homogenization decreases.

Data on the degree of fat rising on week-old milk heated at 190°F. for 30 minutes and homogenized at various temperatures are recorded in Table III. One series was homogenized immediately after pasteurization and the other series was held at 40°F. for 24 hours prior to tempering for homogenization. Data showed, in the case of the milk being warmed to the homogenization temperature after being held at 40°F. for 24 hours, that the fat rings were more prominent than when the milk was homogenized at that same temperature immediately following pasteurization. Fat rings were absent in both cases at a homogenization temperature of 190°F., but became progressively more pronounced as the temperature of homogenization was lowered.

The nature of the fat ring developing on milk homogenized at different temperatures is shown in Figures 9, 10, 11, 12 and 13. Milk was pasteurized by heating to 170°F. for 30 minutes and portions of it homogenized at 170, 150, 130 and 110°F. After storage of one week at 40°F. the samples were photographed to show the degree of fat rising on each. The ring material itself from the milk homogenized at 110°F. was withdrawn on a spatula and photographed (Figure 13).

Influence of specific fats upon some of the physical properties of homogenized milk

Data on the influence of the nature of the fat upon the curd tension of the resulting product is given in Tables XXVIII, XXX, XXXII and XXXIV. These values vary somewhat, however it may be noted that when hydrogenated cottonseed oil flake or hydrogenated tallow was used as the sole source of fat, the curd tension tended to be slightly higher than when other fats were used for this purpose.

These tables also record data on surface tension studies on milk with substituted fats. Apparently, the type of fat has a much more pronounced effect on surface tension than on curd tension. Data in Table XXX show that the hydrogenated fat causes an increase in surface tension of the homogenized product. Most of the products made with foreign fats have surface tensions above that of milk. Exceptions to this are white lard oil, castor oil, raw linseed oil, palm oil, tallow, tributyrin and lanolin.

The only fat of the group used in these tests which seems to have any definite effect on the heat stability of the product is tributyrin. As is shown in Tables XXXII and XXXIV, the samples containing only tributyrin as the source of fat decreased the heat stability about 28 minutes.

Those samples containing tributyrin as a sole source of fat had a greater relative viscosity than most of the other samples (Tables XXXII and XXXIV). The cottonseed oil product also had a fairly high relative viscosity (Table XXXII). All viscosity measurements were within the range of 3.0 to 4.2 centiposes, a relatively narrow range on the group as a whole.

Flavor scores and criticisms were made on some of the samples containing substituted fats. Those samples containing tributyrin and trimyristin as a sole source of fat were very bitter. Most of the others tasted oily. "Velvet", a trade name for a vegetable product, and "Marbase (S)", also a trade name, are fats recommended by their manufacturers for use in ice cream and milk as substitutes for butterfat in these products. The synthetic milks made from these fats were acceptable products as shown by flavor scores (Tables XXIX and XXXV).

The prominence of the fat ring present on the sample after standing at 40°F. for one week seemed to be more pronounced on samples containing certain kinds of fat. Data in Tables XXXIII and XXXV show that, in the case of the tributyrin samples, no fat ring was noted but, in the case of lanolin, the samples had a cream plug on them to the extent that the milk would not pour from the bottle. This was true also in the case of "Marbase (C)" in two trials (Tables XXIX and XXXV), and in the case of "Marbase (S)" in one trial (Table XXXV). Noteworthy also was the observation that the sample containing hydrogenated tallow showed no fat-ring formation (Table XXXI).

There may be a correlation between the Farrall index and the efficiency of homogenization of milk, but when the butterfat has been substituted with certain foreign fats there seems to be little correlation between this index and the formation of a fat ring on the product. The tributyrin sample exhibited a large index, yet no fat ring appeared at all (Table XXXIII). In some instances there were few individual fat globules present, but the fat appeared in masses under the microscope. In some cases the fat-substituted milk precipitated when mixed into the glycerol-water mixture in preparation for the determination of the Farrall index.

TABLE I

THE INFLUENCE OF HEAT-SHOCKING AND STORAGE ON FAT RISING IN HOMOGENIZED MILK AS SHOWN BY THE UNITED STATES PUBLIC HEALTH SERVICE HOMOGENIZATION INDEX

Heat Treatment of Bottled Milk	The U. S. P. H. S. Homogenization Indices of Milk Stored at 40°F.							
	Trial Number							
	1	2	3	4	5	6	7	Average
				24-hour storage				
None	5.13	6.98	4.44	0.0	5.00	0.0	0.0	3.08
Heat shocked	5.13	6.82	6.52	0.0	4.88	0.0	2.77	3.73
				48-hour storage				
None	9.76	10.87	10.42	0.0	9.52	2.78	5.40	6.96
Heat shocked	9.76	12.77	10.42	0.0	9.30	5.41	5.40	7.58
				72-hour storage				
None	13.95	18.75	14.00	0.0	15.56	10.53	10.52	11.90
Heat shocked	11.90	13.04	14.00	2.86	13.33	5.41	7.89	9.78
				96-hour storage				
None	15.91	14.58	19.23	16.22	17.39	10.53	15.00	15.55
Heat shocked	13.95	14.28	17.31	5.56	17.39	7.89	12.82	12.74
				120-hour storage				
None	17.78	27.46	24.07	—	20.83	12.82	15.00	19.66
Heat shocked	13.95	24.00	20.75	10.81	22.44	12.82	15.00	17.11
				144-hour storage				
None	18.18	30.19	24.45	16.21	25.49	15.00	12.50	20.29
Heat shocked	18.18	26.92	24.45	10.52	26.92	15.00	14.63	19.52
				168-hour storage				
None	23.40	29.63	28.57	13.89	26.92	15.00	17.07	22.07
Heat shocked	26.53	25.00	29.31	12.82	26.92	15.00	19.04	22.09

TABLE II

THE INFLUENCE OF SAMPLING TECHNIQUE ON THE UNITED STATES PUBLIC HEALTH SERVICE HOMOGENIZATION INDEX

Heat Treatment of Bottled Milk	Sampling Procedure*	The U. S. P. H. S. Homogenization Indices					
		Trial Number					
		1	2	3	4	5	Average
				24-hour storage			
None	A	4.44	2.86	4.88	2.78	0.0	2.99
	B	4.44	0.0	5.00	0.0	0.0	1.89
Heat-shocked	A	6.52	5.56	4.88	2.78	2.77	4.50
	B	6.52	0.0	4.88	0.0	2.77	2.83
				48-hour storage			
None	A	10.42	0.0	9.30	2.78	5.40	5.58
	B	10.42	0.0	9.52	2.78	5.40	5.62
Heat-shocked	A	10.42	13.54	9.30	5.41	5.40	8.81
	B	10.42	0.0	9.30	5.41	5.40	6.10
				72-hour storage			
None	A	15.69	0.0	13.33	10.53	10.52	10.01
	B	14.00	0.0	15.56	10.53	10.52	10.12
Heat-shocked	A	15.69	0.0	15.21	7.89	7.89	9.34
	B	14.00	2.86	13.33	5.41	7.89	8.69
				96-hour storage			
None	A	19.23	13.89	15.21	10.53	12.50	14.27
	B	19.23	16.22	17.39	10.53	12.82	15.24
Heat-shocked	A	19.23	18.42	19.15	10.26	10.25	15.46
	B	17.31	5.56	17.39	7.89	10.52	11.73
				120-hour storage			
None	A	20.75	21.05	20.83	12.82	12.50	17.59
	B	24.07	(lost)	20.83	12.82	15.00	18.18
Heat-shocked	A	22.22	10.52	24.00	15.00	17.07	17.76
	B	20.75	10.81	22.44	12.82	15.00	16.36
				144-hour storage			
None	A	25.45	20.51	25.49	15.00	15.00	20.09
	B	24.45	16.21	25.49	15.00	12.50	18.73
Heat-shocked	A	26.79	12.82	26.92	17.07	17.07	20.13
	B	24.45	10.52	26.92	15.00	14.63	18.30
				168-hour storage			
None	A	26.79	18.42	26.92	15.00	17.07	20.84
	B	28.57	13.89	26.92	15.00	17.07	20.29
Heat-shocked	A	31.67	15.00	26.92	17.07	19.04	21.94
	B	29.31	12.82	26.92	15.00	19.04	20.62

* A = ring material excluded from both portions
B = simple decantation

TABLE III

THE COMPOSITION OF FAT-RING MATERIAL WHEN MILK PASTEURIZED AT 190°F. FOR 30 MINUTES WAS HOMOGENIZED AT 3000 + 500 POUNDS PRESSURE AT VARIOUS TEMPERATURES

Homogenization Temperature	Analysis of fat-ring material			Prominence of fat-ring after one week at 40°F.
	Fat	S.N.F.	Ash	
(°F.)	(%)	(%)	(%)	(degrees)
Control: Past. Unhomo.	51.04	3.60	0.314	++++
	Homogenized After Pasteurization			
190				0
170				0
150	55.34	4.77	0.460	+
130	43.32	4.66	0.445	++
110	61.94	4.40	0.322	++
90	65.94	8.59	0.287	++++
70	58.67	6.96	0.297	++++
	Homogenized After 24 Hours at 40°F.			
70	60.11	4.28	0.262	++++
90	62.25	6.00	0.265	++++
110	63.30	6.19	0.265	++++
130	64.32	3.71	0.285	+++
150	(68.64% total solids)*		0.337	+
170	55.36	5.23	0.339	+
190				0

* Sample for fat test lost.

TABLE IV

THE INFLUENCE OF CONCENTRATION UPON EVAPORATIVE CAPACITY OF GELATIN SOLUTIONS

Concentration of Gelatin (%)	Weight Loss (%) During Each of Five Consecutive 24-Hour Storage Periods					Total Wt. Loss After 120 Hours (percent)	Difference Due to Concentration
	1st	2nd	3rd	4th	5th		
0	0.663	0.563	0.655	0.599	0.716	3.196	
1	0.648	0.535	0.627	0.581	0.694	3.085	-0.111
2	0.640	0.527	0.629	0.573	0.691	3.060	-0.025
3	0.653	0.529	0.644	0.566	0.724	3.116	+0.056
4	0.632	0.529	0.638	0.574	0.704	3.077	-0.109
5	0.635	0.524	0.631	0.569	0.696	3.055	-0.002
6	0.624	0.516	0.596	0.581	0.691	3.008	+0.003
7	0.627	0.513	0.606	0.566	0.677	2.989	-0.019
8	0.655	0.517	0.614	0.566	0.695	3.047	+0.058
9	0.655	0.514	0.629	0.571	0.706	3.075	+0.028
10	0.635	0.514	0.614	0.568	0.688	3.019	-0.056

TABLE V

THE INFLUENCE OF CONCENTRATION UPON THE EVAPORATIVE CAPACITY OF GELATIN SOLUTIONS

Concentration of Gelatin (%)	Weight Loss (%) During Each of Five Consecutive 24-Hour Storage Periods					Total Wt. Loss After 120 Hours (%)	Difference Due to Concentration
	1st	2nd	3rd	4th	5th		
0.0	0.610	0.769	0.573	0.662	0.571	3.185	
0.01	0.613	0.762	0.576	0.673	0.581	3.205	+0.020
0.02	0.593	0.771	0.561	0.646	0.563	3.134	-0.071
0.03	0.605	0.765	0.564	0.664	0.546	3.144	+0.010
0.04	0.618	0.783	0.586	0.674	0.580	3.241	+0.097
0.05	0.626	0.775	0.584	0.682	0.589	3.256	+0.015
0.06	0.612	0.759	0.594	0.654	0.572	3.191	-0.065
0.07	0.603	0.757	0.586	0.667	0.560	3.173	-0.018
0.08	0.610	0.766	0.595	0.663	0.574	3.208	+0.035
0.09	0.612	0.773	0.600	0.676	0.567	3.228	+0.020
0.10	0.618	0.777	0.605	0.677	0.590	3.267	+0.039
0.20	0.587	0.772	0.586	0.661	0.577	3.183	-0.084
0.30	0.662	0.750	0.597	0.651	0.570	3.230	+0.047
0.40	0.611	0.762	0.582	0.654	0.560	3.169	-0.061
0.50	(data lost)						
0.60	0.608	0.766	0.593	0.648	0.588	3.203	+0.034
0.70	0.601	0.768	0.592	0.656	0.579	3.196	-0.007
0.80	0.591	0.763	0.582	0.643	0.562	3.141	-0.055
0.90	0.615	0.756	0.593	0.652	0.558	3.174	+0.033
1.00	0.614	0.773	0.597	0.664	0.570	3.218	+0.044

TABLE VI

THE INFLUENCE OF COMPOSITION UPON THE EVAPORATIVE CAPACITY OF HOMOGENIZED MILK

Nature of Treatment			Total Solids	Weight Loss(%) During Each of Five 24-Hour Storage Periods					Total Wt. Loss After 120 Hours
Milk Used	Water Added	S.N.F. Added		1st	2nd	3rd	4th	5th	
(Ml.)	(Ml.)	(Grams)	(%)						(%)
9.5	0.0	0.5	16.68	0.193	0.174	0.144	0.227	0.220	0.958
9.6	0.0	0.4	15.81	(lost)	0.184	0.137	0.185	0.227	--
9.7	0.0	0.3	14.93	0.157	0.168	0.140	0.208	0.224	0.897
9.8	0.0	0.2	14.05	0.144	0.169	0.131	0.185	0.206	0.835
9.9	0.0	0.1	13.17	0.136	0.177	0.137	0.166	0.205	0.821
10.0	0.0	0.0	12.30	0.155	0.151	0.150	0.170	0.201	0.827
9.9	0.1	0.0	12.17	0.191	0.165	0.136	0.183	0.222	0.897
9.8	0.2	0.0	12.05	0.147	0.170	0.140	0.212	0.217	0.886
9.7	0.3	0.0	11.93	0.157	0.159	0.158	0.155	0.223	0.852
9.6	0.4	0.0	11.80	0.157	0.168	0.159	0.180	0.191	0.855
9.5	0.5	0.0	11.68	0.149	0.159	0.163	0.171	0.200	0.842

TABLE VII

THE INFLUENCE OF A SURFACE-ACTIVE WASHING POWDER IN HOMOGENIZED MILK UPON FAT-RING FORMATION

Amount of one percent surface-active solution added per quart of homogenized milk	*Prominence of fat-ring after seven days at 40°F.
(Ml.)	
None	++
None	++
0.5	++
1.0	++
1.5	++
2.0	++
2.0 ml. water only	++

* + = slight fat-ring
++ = distinct fat-ring
+++ = prominent fat-ring
++++ = cream plug

TABLE VIII

THE EFFECT OF HEATING HOMOGENIZED MILK TO 165°F. FOR 30 MINUTES UPON THE HEAT STABILITY, CURD TENSION, SURFACE TENSION AND FORMATION OF FAT-RING

Sample Number	Heat Treatment	Heat Stability (Minutes)	Curd Tension (Grams)	Surface Tension (dynes/cm)	Comparison of Intensity of fat-ring after seven days at 40°F.
1	None	150	10.5	48.3	
	Heated	90	6.5	47.8	greater
2	None	130	15.0	48.5	greater
	Heated	80	13.0	46.4	
3	None	120	9.0	48.0	greater
	Heated	100	11.0	47.3	
4	None	140	13.0	46.8	greater
	Heated	140	8.0	45.6	
5	None	125	12.5	47.4	
	Heated	85	11.7	46.5	greater
6	None	120	14.0	47.3	equal
	Heated	95	10.5	46.2	
7	None	125	7.0	47.0	equal
	Heated	90	10.0	46.3	
8	None	115	9.0	46.7	
	Heated	105	15.0	46.7	greater
9	None	100	8.0	46.3	equal
	Heated	60	7.0	46.0	
Average	None	125	10.9	47.4	equal
	Heated	94	10.3	46.5	

TABLE IX

THE INFLUENCE OF ADDED WATER UPON THE PHYSICAL PROPERTIES OF HOMOGENIZED MILK

Sample	T.S.:H_2O Ratio*	Surface Tension	Curd Tension	Heat Stability	Prominence of fat-ring After Seven Days
(No.)		(Dynes/cm)	(Grams)	(Minutes)	(Degree)
Series I					
1	1:8.28	46.5	6	150	+
2	1:8.83	46.8	13	105	++
3	1:9.38	46.7	8	115	++
4	1:9.92	46.2	6	130	++
5	1:10.40	46.7	5	160	+++
Series II					
1	1:8.57	46.6	8	155	+
2	1:9.14	46.7	11	147	++
3	1:9.70	46.9	12	149	++
4	1:10.27	46.6	9	162	++
5	1:10.83	46.6	10	172	++
Series III					
1	1:8.11	46.6	6	120	++
2	1:8.68	46.3	8	158	+++
3	1:9.17	46.4	10	148	+++
4	1:9.71	46.5	8	183	+++
5	1:10.25	46.3	7	180	++
Series IV					
1	1:7.41	46.6	11	140	+
2	1:7.91	46.6	14	142	+
3	1:8.41	46.3	8	158	++
4	1:8.90	46.8	7	165	+++
5	1:9.39	46.6	6	176	+++

* First sample in each series analyzed for fat and SNF, the others calculated.

TABLE X

THE INFLUENCE OF INCREASING SOLIDS CONCENTRATIONS UPON THE SURFACE TENSION OF HOMOGENIZED MILK

Analysis of Original Milk		Surface tension of milk with increments of table cream added. (lbs./gallon)				
Fat	SNF	0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent)	(percent)	(dynes)	(dynes)	(dynes)	(dynes)	(dynes)
* 3.20	7.71	46.5	45.9	46.5	45.8	46.4
* 3.27	7.87	46.3	46.5	46.4	46.4	46.5
** 3.17	7.60	46.5	46.4	46.5	46.3	44.2
** 3.75	8.43	46.3	46.6	46.5	46.6	46.4
** 3.63	8.02	46.3	46.6	46.1	46.7	46.5
Average		46.38	46.40	46.40	46.36	46.00

* 19.64% fat in cream added
** 18.60% fat in cream added

TABLE XI

THE INFLUENCE OF INCREASING SOLIDS CONCENTRATIONS UPON THE CURD TENSION OF HOMOGENIZED MILK

Analysis of Original Milk		Curd tension of milk with increments of table cream added. (lbs./gallon)				
Fat	SNF	0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent)	(percent)	(grams)	(grams)	(grams)	(grams)	(grams)
* 3.20	7.71	12	7	8	9	5
* 3.27	7.87	7	9	7	8	7
** 3.17	7.60	9	11	9	9	8
** 3.75	8.43	9	11	10	10	8
** 3.63	8.02	4	10	7	8	7
Average		8.2	9.6	8.2	8.8	7.4

* 19.64% fat in cream added

** 18.60% fat in cream added

TABLE XII

THE INFLUENCE OF INCREASING SOLIDS CONCENTRATIONS UPON THE HEAT STABILITY OF HOMOGENIZED MILK

Analysis of Original Milk		Heat stability of milk with increments of table cream added (lbs./gallon)				
Fat	SNF	0.0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent)	(percent)	(minutes)	(minutes)	(minutes)	(minutes)	(minutes)
* 3.20	7.71	150	125	125	125	127
* 3.27	7.87	152	136	135	145	127
** 3.17	7.60	154	132	130	129	129
** 3.75	8.43	115	112	110	115	100
** 3.63	8.02	120	105	105	103	103
Average		138.2	122.0	121.0	123.4	117.2

* 19.64% fat in cream added
** 18.60% fat in cream added

TABLE XIII

THE INFLUENCE OF INCREASING SOLIDS CONCENTRATIONS UPON DEGREE OF FAT RISING ON HOMOGENIZED MILK AFTER SEVEN DAYS STORAGE AT 40°F.

Analysis of Original Milk		Degree of fat rising on milk with increments of table cream added (lbs./gallon)				
Fat	SNF	0.0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent)	(percent)					
* 3.20	7.71	+++	++++	++++	++++	++++
* 3.27	7.87	+++	+++	+++	+++	++++
** 3.17	7.60	0	+++	+++	+++	++++
** 3.75	8.43	0	++	++	+++	+++
** 3.63	8.02	++	+++	++++	++++	++++

* 19.64% fat in cream added
** 18.60% fat in cream added

TABLE XIV

THE INFLUENCE OF INCREASING SOLIDS-NOT-FAT CONCENTRATIONS UPON THE SURFACE TENSION OF HOMOGENIZED MILK

Analysis of Original Milk		Surface tension of milk with increments of a 25% concentration of SNF in water added (lbs./gallon)				
Fat	SNF	0.0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent)	(percent)	(dynes)	(dynes)	(dynes)	(dynes)	(dynes)
3.32	7.85	45.9	46.8	46.9	47.0	46.9
3.69	8.76	46.4	46.5	46.7	46.6	46.6
4.00	9.02	46.6	46.6	46.6	47.0	47.2
3.39	7.73	46.5	46.5	46.4	46.6	46.7
3.65	8.75	46.4	46.2	45.9	46.4	46.5
3.75	8.74	46.4	46.3	46.7	47.0	(lost)
Average		46.36	46.48	46.56	46.76	46.78

TABLE XV

THE INFLUENCE OF INCREASING SOLIDS-NOT-FAT CONCENTRATIONS UPON THE CURD TENSION OF HOMOGENIZED MILK

Analysis of Original Milk		Curd tension of milk with increments of a 25% concentration of SNF in water added (lbs./gallon)				
Fat	SNF	0.0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent	(percent)	(grams)	(grams)	(grams)	(grams)	(grams)
3.32	7.85	10	22	27	30	35
3.69	8.76	14	22	26	31	34
4.00	9.02	17	23	27	32	37
3.39	7.73	12	18	23	33	35
3.65	8.75	19	23	27	34	38
3.75	8.74	14	20	25	30	(lost)
Average		14.3	21.3	25.8	31.6	35.8

TABLE XVI

THE INFLUENCE OF INCREASING SOLIDS-NOT-FAT CONCENTRATIONS UPON THE HEAT STABILITY OF HOMOGENIZED MILK

Analysis of Original Milk		Heat stability of milk with increments of a 25% concentration of SNF in water added (lbs./gallon)				
Fat	SNF	0.0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs
(percent)	(percent)	(minutes)	(minutes)	(minutes)	(minutes)	(minutes)
3.32	7.85	170	140	128	125	126
3.69	8.76	163	147	142	134	116
4.00	9.02	132	118	115	115	111
3.39	7.73	170	157	150	135	129
3.65	8.75	143	128	122	114	108
3.75	8.74	127	125	125	122	(lost)
Average		151.3	135.8	130.3	124.1	118.0

TABLE XVII

THE INFLUENCE OF INCREASING SOLIDS-NOT-FAT CONCENTRATIONS UPON THE VISCOSITY OF HOMOGENIZED MILK

Analysis of Original Milk		Relative viscosity of milk with increments of a 25% concentration of SNF in water added (lbs./gallon)				
Fat	SNF	0.0	0.5 lb.	1.0 lb.	1.5 lbs.	2.0 lbs.
(percent)	(percent)	(cp.)	(cp.)	(cp.)	(cp.)	(cp.)
3.32	7.85	3.4	3.3	3.8	4.4	4.3
3.69	8.76	3.9	3.8	3.8	4.2	4.8
4.00	9.02	3.9	3.7	4.3	4.3	4.3
3.39	7.73	3.3	3.4	3.6	4.0	4.3
3.65	8.75	3.5	3.8	4.2	4.9	4.4
3.75	8.74	3.9	3.6	4.5	4.6	(lost)
Average		3.65	3.60	4.03	4.40	4.42

TABLE XVIII

THE INFLUENCE OF INCREASING SOLIDS-NOT-FAT CONCENTRATIONS UPON THE DEGREE OF FAT RISING ON HOMOGENIZED MILK

Analysis of Original Milk: Fat	Analysis of Original Milk: SNF	Relative degree of fat-ring formation with increments of a 25% concentration of SNF in water added (lbs./gallon): 0.0	0.5	1.0	1.5	2.0
(percent)	(percent)	(degree)	(degree)	(degree)	(degree)	(degree)
3.32	7.85	+	+	++	+	++
3.69	8.76	++++	++++	++++	++++	++++
4.00	9.02	++++	++++	+++	++++	++++
3.39	7.73	0	+	+	++	+++
3.65	8.75	+	+++	+++	+++	++++
3.75	8.74	+++	++++	+++	+++	(lost)
Average		2.0	2.83	2.66	2.83	3.40

TABLE XIX

THE INFLUENCE OF HOMOGENIZATION PRESSURES UPON CURD TENSION, SURFACE TENSION AND HEAT STABILITY OF HOMOGENIZED MILK

Series	Fat	Homogenization Pressure	Homogenization Temperature	Curd Tension	Surface Tension	Heat Stability
	(%)	(lbs./sq. in.)	($^{\circ}$F.)	(grams)	(dynes/cm)	(minutes)
		2000 + 500	110	8	46.6	94
		3000 + 500	110	8	46.6	94
I	3.6	4000 + 500	110	13	46.5	93
		5000 + 500	110	10	46.9	91
		6000 + 500	110	9	46.9	90
		2000 + 500	125	12	46.5	90
		3000 + 500	125	10	46.2	90
II	3.6	4000 + 500	125	11	46.7	94
		5000 + 500	125	13	46.6	102
		6000 + 500	125	12	46.5	120
		2000 + 500	130	12	46.3	146
		3000 + 500	130	13	46.5	128
III	3.7	4000 + 500	130	13	46.6	130
		5000 + 500	130	14	46.8	131
		6000 + 500	130	14	47.0	154
		2000 + 500	130	10	46.6	104
		3000 + 500	130	11	46.3	128
IV	4.1	4000 + 500	130	9	46.6	99
		5000 + 500	130	12	47.0	98
		6000 + 500	130	11	47.0	103

TABLE XX

THE INFLUENCE OF HOMOGENIZATION PRESSURES UPON VISCOSITY AND FAT-RING FORMATION ON HOMOGENIZED MILK

Series	Fat	Homogenization Pressure	Homogenization Temperature	Relative Viscosity	Presence of Fat-ring After Seven Days at 40°F.
	(%)	(lbs./sq. in.)	(°F.)	(cp.)	(degree)
I	3.6	2000 + 500	110	4.1	+
		3000 + 500	110	4.8	+
		4000 + 500	110	3.8	0
		5000 + 500	110	3.8	0
		6000 + 500	110	3.6	0
II	3.6	2000 + 500	125	4.4	+
		3000 + 500	125	3.6	0
		4000 + 500	125	4.0	0
		5000 + 500	125	3.8	0
		6000 + 500	125	3.4	0
III	3.7	2000 + 500	130	4.2	+
		3000 + 500	130	4.0	+
		4000 + 500	130	4.3	0
		5000 + 500	130	3.9	0
		6000 + 500	130	3.7	0
IV	4.1	2000 + 500	130	4.0	++++
		3000 + 500	130	3.8	++
		4000 + 500	130	4.0	++
		5000 + 500	130	4.0	+
		6000 + 500	130	4.0	0

TABLE XXI

EFFECT OF RECIRCULATING MILK THROUGH A HOMOGENIZER UPON THE CURD AND SURFACE TENSIONS OF MILK

Series Number	Sample Number	Minutes Recirculated	Starting Homogenizing Temperature	Curd Tension	Surface Tension
			(°F.)	(grams)	(dynes/cm.)
1	1	0	138	20	46.7
	2	5		16	46.6
	3	10		15	46.5
	4	20		13	46.8
	5	30		10	46.1
2	1	0	139	21	46.4
	2	5		14	46.3
	3	10		14	46.6
	4	20		13	46.6
	5	30		17	46.6
3	1	0	132	9	46.3
	2	5		8	46.9
	3	10		7	47.2
	4	20		8	47.2
	5	30		7	46.9
4	1	0	102	11	46.7
	2	5		8	46.5
	3	10		9	46.7
	4	20		11	46.9
	5	30		11	47.0

TABLE XXII

EFFECT OF RECIRCULATING MILK THROUGH A HOMOGENIZER UPON VISCOSITY AND HEAT STABILITY OF THE MILK AND UPON FAT-RING FORMATION AFTER STORAGE AT 40°F. FOR ONE WEEK

Series Number	Sample Number	Minutes Recirculated	Starting Homogenizing Temperature	Viscosity	Heat Stability	Presence of Fat-ring
			(°F.)	(cp.)	(minutes)	(degree)
1	1	0	138	3.8	148	++
	2	5		4.0	146	+
	3	10		3.5	142	0
	4	20		3.6	146	0
	5	30		4.3	146	0
2	1	0	139	3.9	147	+
	2	5		4.2	147	0
	3	10		3.4	145	0
	4	20		3.9	142	0
	5	30		4.0	123	0
3	1	0	132	4.0	151	++
	2	5		3.9	150	+
	3	10		3.4	146	+
	4	20		3.6	145	0
	5	30		4.4	138	0
4	1	0	102	4.2	166	+++
	2	5		3.6	157	++
	3	10		4.0	146	+
	4	20		3.3	145	+
	5	30		3.8	138	0

TABLE XXIII

EFFECT OF RECIRCULATING MILK THROUGH A HOMOGENIZER UPON THE TEMPERATURE, HEAT STABILITY AND VISCOSITY OF THE MILK, AND PRESENCE OF FAT RING ON THE MILK AFTER SEVEN DAYS STORAGE AT 40°F.

Sample Number	Minutes Recirculated	Temperature at Which Sample Was Withdrawn	Heat Stability	Viscosity	Fat Ring After 7 Days Storage at 40°F.
		(°F.)	(minutes)	(cp.)	(degree)
Series I (Series IV in Tables XX and XXI)					
1	0	102	166	4.2	+++
2	5	112	157	3.6	++
3	10	121	146	4.0	+
4	20	134	145	3.3	+
5	30	150	138	3.8	0
Series II					
1	0	135	148	4.0	++
2	5	138	130	3.6	+
3	10	141	129	4.4	+
4	20	152	123	4.0	0
5	30	162	114	4.4	0
Series III					
1	0	134	133	3.9	++
2	5	138	131	4.4	+
3	10	144	130	3.7	+
4	20	152	129	3.9	0
5	30	164	114	4.0	0
Series IV					
1	0	131	145	4.2	++
2	5	141	134	4.0	+
3	10	146	133	3.5	+
4	20	156	124	4.4	0
5	30	167	101	4.4	0
Series V					
1	0	128	140	3.4	++
2	5	135	134	4.3	+
3	10	141	133	3.7	0
4	20	151	132	3.4	0
5	30	162	101	3.7	0

TABLE XXIV

RELATIVE PROMINENCE OF FAT RING ON MILK PASTEURIZED AND HOMOGENIZED AT DIFFERENT TEMPERATURES AFTER ONE WEEK'S STORAGE AT 40°F.

Pasteurization Temperature (°F.)	Homogenization Temperature (°F.)	Trial Number				Average
		1	2	3	4	
143	143	+	++	++	++	1.75
	110	+	++	+++	++	2.00
150	150	++	++	++	++	2.00
	110	++++	+++	++++	++++	3.75
158	158	++	++	+	+	1.50
	110	++++	++++	+++	++++	3.75
175	175	++	+	+	+	1.25
	110	+++	++++	+++	++++	3.50

TABLE XXV

UNITED STATES PUBLIC HEALTH SERVICE HOMOGENIZATION INDICES ON ONE WEEK-OLD MILK PASTEURIZED AND HOMOGENIZED AT DIFFERENT TEMPERATURES

Pasteurization Temperature	Homogenization Temperature	Trial Number 1	2	3	4	Average
143	143	36.8	32.3	34.5	40.4	36.0
	110	43.5	36.8	35.7	42.5	39.6
150	150	30.1	32.1	33.3	32.0	31.9
	110	34.8	33.3	35.3	35.9	34.8
158	158	23.4	25.8	33.3	33.3	28.9
	110	38.6	40.2	26.5	43.1	37.1
175	175	19.1	23.7	21.7	27.0	22.8
	110	50.0	57.9	42.0	53.9	50.9

TABLE XXVI

DEGREE OF FAT RISING ON MILK PASTEURIZED AND HOMOGENIZED AT DIFFERENT TEMPERATURES

Pasteurization Temperature (30 minutes) (°F.)	Homogenization Temperature (3000 + 500 lbs.) (°F.)	Trial Number 1	2	3	4	Average
150	150	++	++	++	++	2.00
	130	+++	+++	+++	++	2.75
	110	++++	++++	++++	++++	4.00
170	170	+	+	++	+	1.25
	150	+++	++	++	+	2.00
	130	+++	+++	+++	+++	3.00
	110	++++	++++	++++	++++	4.00
190	190	+	+	+	++	1.25
	170	+	+	+	++	1.25
	150	++	++	++	++	2.00
	130	+++	+++	++++	++++	3.50
	110	++++	++++	++++	++++	4.00

TABLE XXVII

THE INFLUENCE OF PASTEURIZATION AND HOMOGENIZATION OF MILK AT VARIOUS TEMPERATURES UPON THE FARRALL INDEX AND UNITED STATES PUBLIC HEALTH SERVICE HOMOGENIZATION INDEX

Pasteurization Temperature (°F.)	Homogenization Temperature (°F.)	Sample 1		Sample 2	
		Farrall Index	U.S.P.H.S. Homo Index	Farrall Index	U.S.P.H.S. Homo Index
150	150	25.4	7.69	180.0	10.0
	130	33.0	14.63	86.8	10.0
	110	65.8	17.44	182.0	18.6
170	170	35.6	7.69	58.0	9.09
	150	101.2	10.97	35.8	11.25
	130	83.0	16.27	82.4	16.67
	110	(clumps) 214.4	43.54	306.6	39.13
190	190	8.4	5.00	246.2	8.10
	170	78.0	11.11	52.6	10.25
	150	150.4	12.19	44.0	10.00
	130	165.2	13.95	153.8	16.28
	110	387.0	45.16	370.0	25.00

TABLE XXVIII

CURD TENSION, SURFACE TENSION, HEAT STABILITY AND VISCOSITY OF FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Curd Tension	Surface Tension	Heat Stability	Relative Viscosity
	(No.)	(grams)	(dynes/cm.)	(minutes)	(cp.)
Animal:					
Butterfat	1	23	46.6	156	3.9
	2	27	46.5	152	3.5
Mutton tallow	1	24	46.3	149	3.2
	2	24	46.7	144	3.4
White Lard oil	1	26	43.0	168	3.4
	2	26	41.9	154	3.7
Vegetable:					
Castor oil	1	28	46.2	149	3.3
	2	28	45.6	143	3.6
Marbase (C)	1	24	47.5	161	3.4
	2	23	47.4	150	3.8
Marbase (S)	1	25	48.2	165	3.8
	2	25	47.5	158	3.5
Velvet	1	27	50.3	163	3.7
	2	25	48.2	153	3.4

TABLE XXIX

FARRALL INDEX, FLAVOR AND PROMINENCE OF FAT RING ON FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Farrall Index	Flavor Score	Flavor Criticism	Prominence of Fat Ring after Storage at 40°F. for 1 week
	(No.)				(degree)
Animal:					
Butterfat	1	46.6	38	Lacks fine flavor	+
	2	97.6			+
Mutton tallow	1	44.6			+++
	2	*			+++
White Lard oil	1	*			+
	2	*			0
Vegetable:					
Castor oil	1	*			++
	2	*			++
Marbase (C)	1	20.2	35	Oxidized	++
	2	125.2			++++
Marbase (S)	1	28.0	38	Flat, cooked	+++
	2	105.8			+++
Velvet	1	*			++
	2	*			++

* Few individual globules, much free fat.

TABLE XXX

CURD TENSION, SURFACE TENSION, HEAT STABILITY AND RELATIVE VISCOSITY OF FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Curd Tension	Surface Tension	Heat Stability	Relative Viscosity
	(No.)	(grams)	(dynes/cm.)	(minutes)	(cp.)
Animal:					
Butterfat	1	22	46.1	152	4.0
	2	22	46.0	141	3.9
Hydrogenated tallow	1	29	49.5	152	3.7
	2	24	50.0	150	3.6
Tallow	1	20	43.0	152	3.2
	2	19	43.0	150	4.0
Vegetable:					
Hydrogenated cottonseed oil flake	1	28	49.8	152	3.1
	2	27	49.9	152	3.6
Palm oil	1	29	42.0	152	3.4
	2	20	40.2	152	3.7
Peanut oil	1	26	49.1	151	3.4
	2	25	48.6	150	3.9
Raw linseed oil	1	20	43.2	152	3.6
	2	21	43.7	152	3.8

TABLE XXXI

FARRALL INDEX, FLAVOR AND PROMINENCE OF FAT RING ON FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Farrall Index	Flavor After 96 Hours		Prominence of Fat Ring after Storage at 40°F. for 1 week
			Score	Criticism	
	(No.)				(degree)
Animal:					
Butterfat	1	61.4	34	old, fruity	+++
	2	36.6			+++
Hydrogenated tallow	1	*	30	oily	0
	2	*			0
Tallow	1	12.0			+
	2	44.2			+
Vegetable:					
Hydrogenated cottonseed oil flake	1	*	33	oily	++
	2	*			++
Palm oil	1	8.0			0
	2	26.4			0
Peanut oil	1	49.2	30	oily	+++
	2	166.0			+++
Raw Linseed oil	1	*			+
	2	14.0			+

* Few individual globules, much free fat.

TABLE XXXII

CURD TENSION, SURFACE TENSION, HEAT STABILITY AND RELATIVE VISCOSITY OF FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Curd Tension	Surface Tension	Heat Stability	Relative Viscosity
	(No.)	(grams)	(dynes/cm.)	(minutes)	(cp.)
Animal:					
Butterfat	1	27	46.0	150	3.8
	2	24	46.5	150	3.3
Lanolin	1	25	39.1	158	3.4
	2	23	39.3	158	3.7
Vegetable:					
Cocoanut oil	1	26	49.9	150	3.2
	2	18	45.5	150	3.7
Corn oil	1	26	50.4	150	3.4
	2	23	49.1	150	3.9
Cottonseed oil	1	25	51.0	150	3.9
	2	25	48.0	150	4.2
Olive oil	1	23	50.9	146	3.2
	2	19	50.0	145	4.0
Other:					
Tributyrin	1	17	41.6	127	4.0
	2	18	41.6	127	4.0
Trimyristin	1	23	50.8	160	3.0

TABLE XXXIII

FARRALL INDEX, FLAVOR AND PROMINENCE OF FAT RING OF FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Farrall Index	Flavor After 48 Hours		Prominence of Fat Ring after Storage at $40^{o}F$. for 1 week
			Score	Criticism	
	(No.)				(degree)
Animal:					
Butterfat	1	8.2	33	fruity	0
	2	11.2			+
Lanolin	1	408.2			++++
	2	82.4			++++
Vegetable:					
Cocoanut oil	1	8.2	30	oily	+
	2	32.8			+
Corn oil	1	31.6	29	oily	++
	2	21.8			++
Cottonseed oil	1	16.8	30	oily	++
	2	32.6			++
Olive oil	1	13.8	30	oily	++
	2	34.2			+++
Other:					
Tributyrin	1	100.2	0	bitter	0
	2	812.0			0
Trimyristin		*	0	bitter	+

* Large particles of free fat.

TABLE XXXIV

CURD TENSION, SURFACE TENSION, HEAT STABILITY AND VISCOSITY OF FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Curd Tension	Surface Tension	Heat Stability	Relative Viscosity
	(No.)	(grams)	(dynes/cm.)	(minutes)	(cp.)
Animal:					
Butterfat	1	24	46.4	163	3.6
	2	17	46.4	150	3.8
Vegetable:					
Cocoanut oil	1	17	46.7	158	3.9
Corn oil	1	24	48.8	159	3.8
	2	17	48.5	145	3.6
Cottonseed oil	1	(lost)	47.6	158	3.3
Marbase (C)	1	22	48.3	163	3.5
	2	18	47.3	155	3.6
Marbase (S)	1	17	47.8	163	3.5
	2	17	48.0	155	3.5
Peanut oil	1	17	50.5	158	3.8
	2	12	49.4	148	4.2
Velvet	1	24	50.3	163	3.5
	2	15	48.0	142	3.4
Other:					
Tributyrin	1	18	41.5	128	3.9
	2	12	41.4	118	4.2

TABLE XXXV

FARRALL INDEX, FLAVOR AND PROMINENCE OF FAT RING ON FAT-SUBSTITUTED HOMOGENIZED SYNTHETIC MILK

Type of Fat Used	Trial	Farrall Index	Flavor After 24 Hours Score	Flavor After 24 Hours Criticism	Prominence of Fat Ring after Storage for 1 week at 40°F.
	(No.)				(degree)
Animal:					
Butterfat	1	73.2	35	Unclean,rancid	++
	2	45.2			++
Vegetable:					
Cocoanut oil		61.2			++
Corn oil	1	25.2			+
	2	35.8			++
Cottonseed oil		*			+
Marbase (C)	1	67.0	36	oily	+++
	2	65.0			++++
Marbase (S)	1	66.6	39	cooked	++++
	2	65.6			+++
Peanut oil	1	(lost)			+++
	2	44.8			+++
Velvet	1	15.4	37	Unclean,cooked	0
	2	27.8			++
Other:					
Tributyrin	1	25.6			0
	2	27.6			0

*Clumps of fat present.

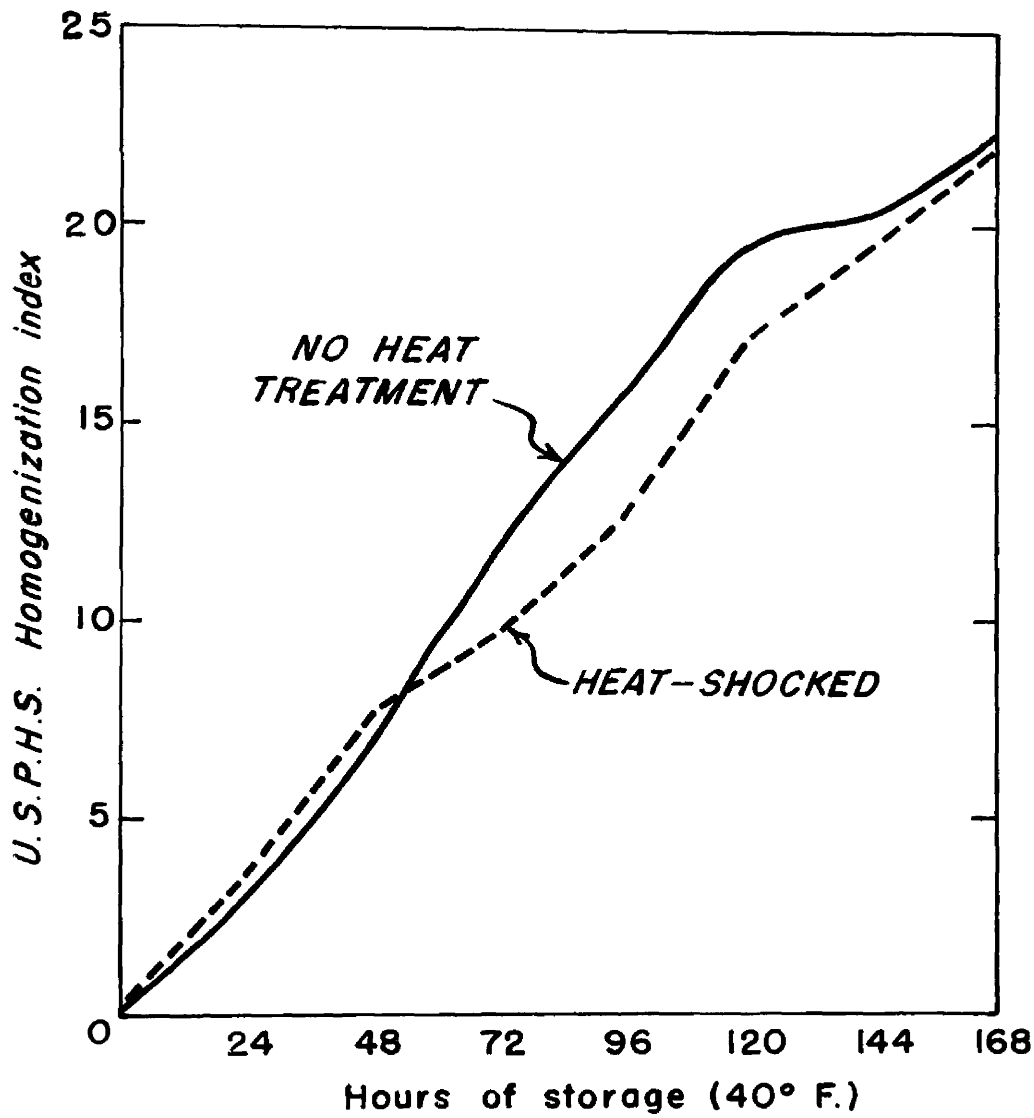

Figure 1. United States Public Health Service Homogenization index on milk of different ages some of which was warmed to room temperature 24 hours before sampling.

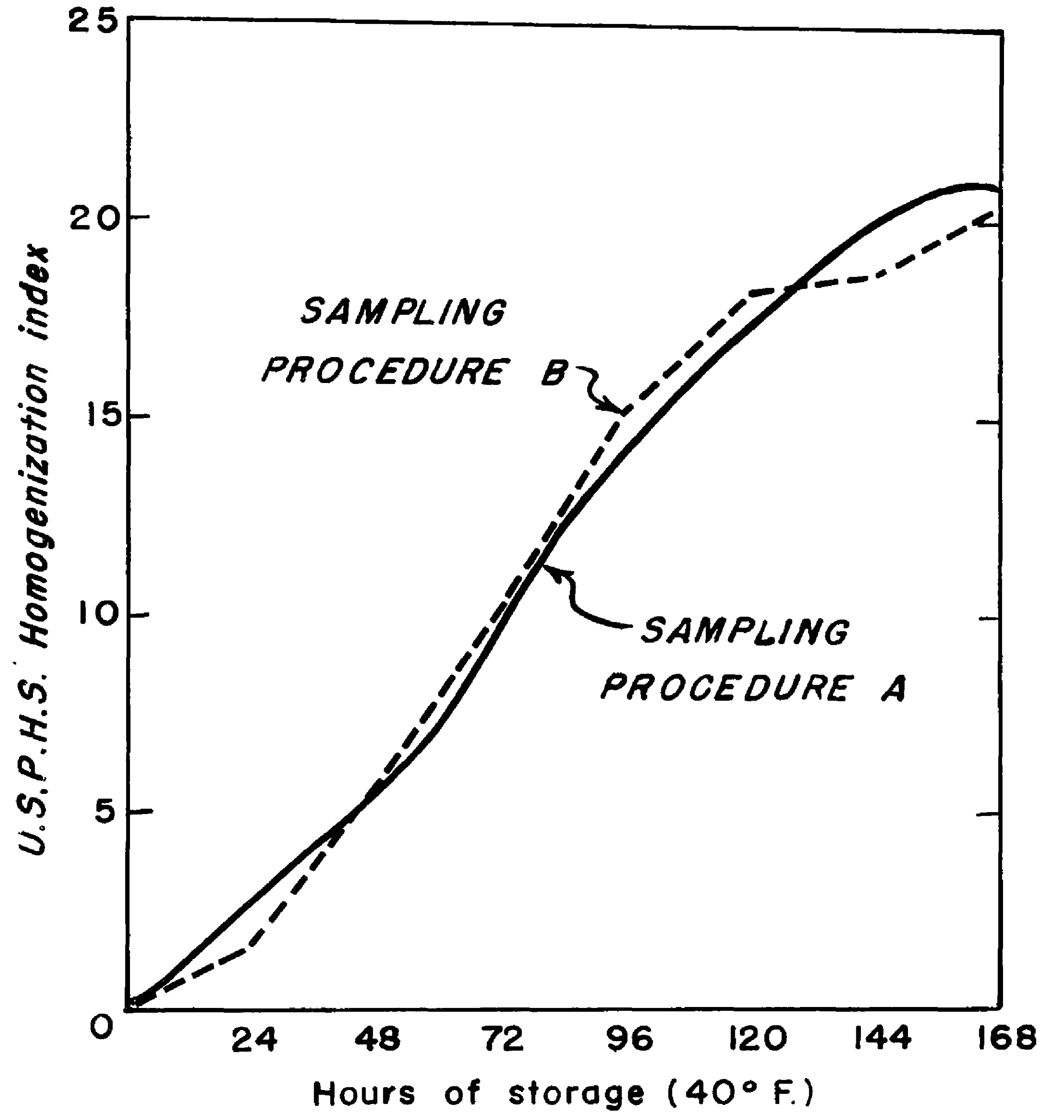

Figure 2. United States Public Health Service Homogenization index on milk of different ages sampled by two different procedures.

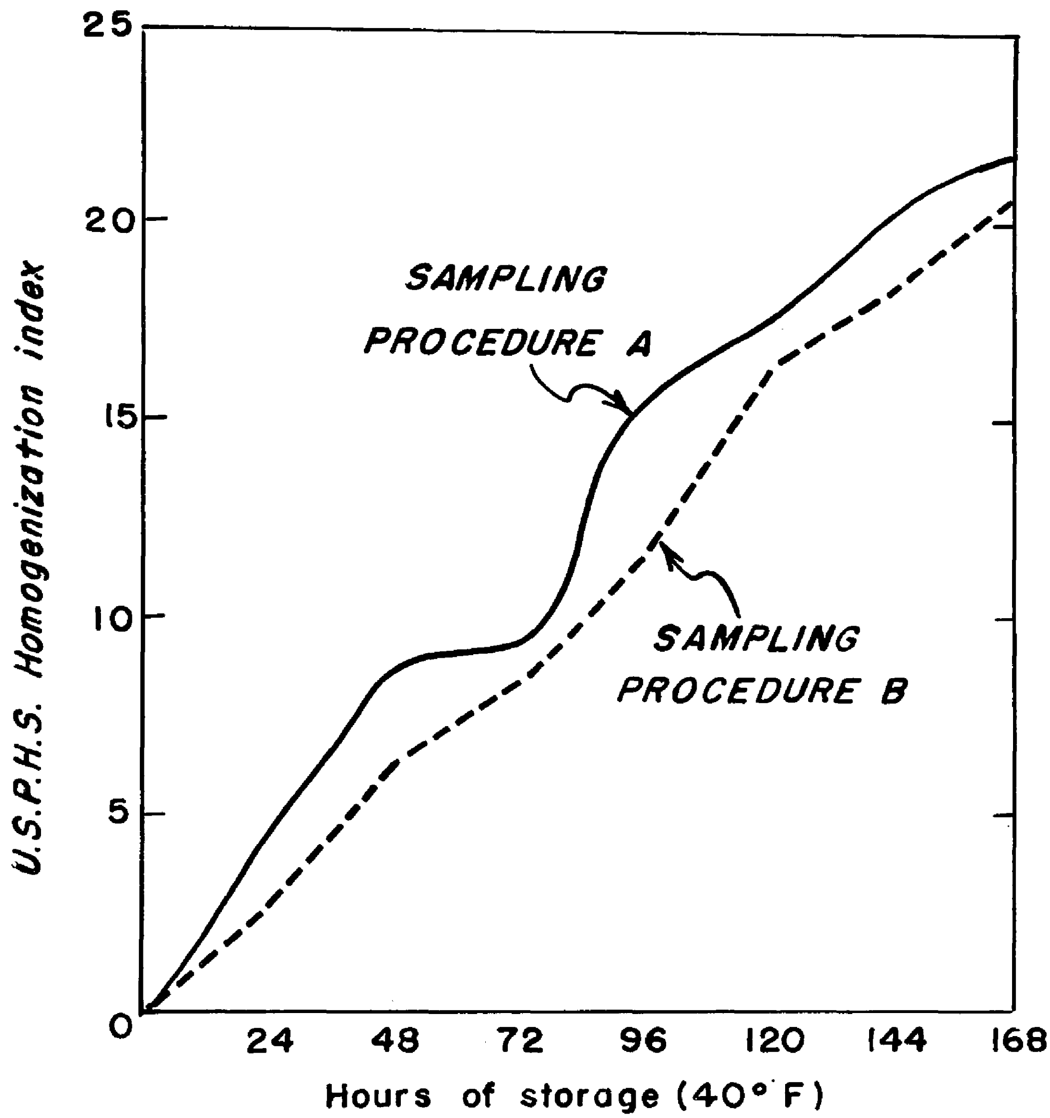

Figure 3. United States Public Health Service Homogenization index on milk of different ages warmed to room temperature 24 hours prior to sampling and sampled by two different procedures.

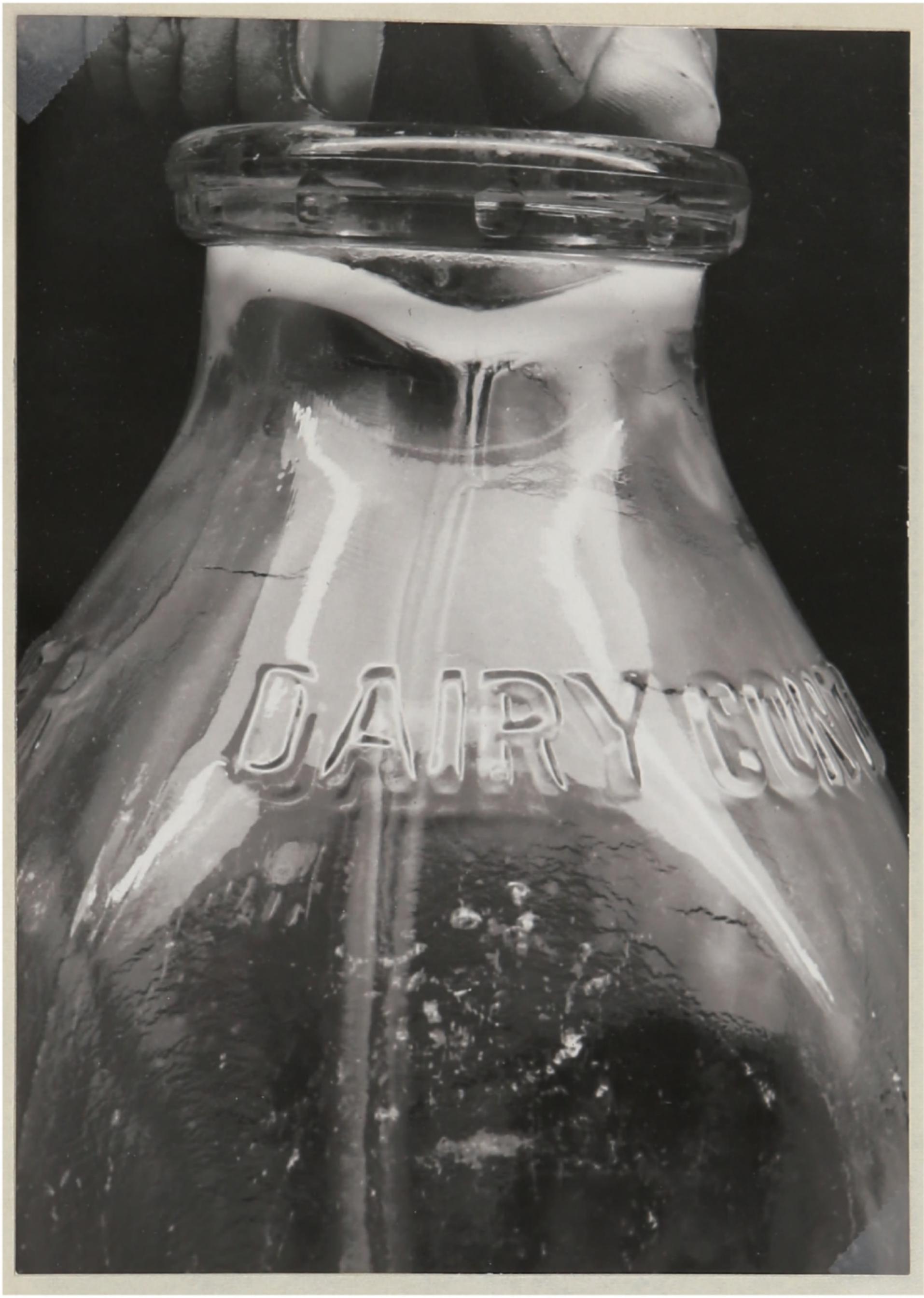

Figure 4. Appearance of fat ring soon after pouring the milk from a quart bottle.

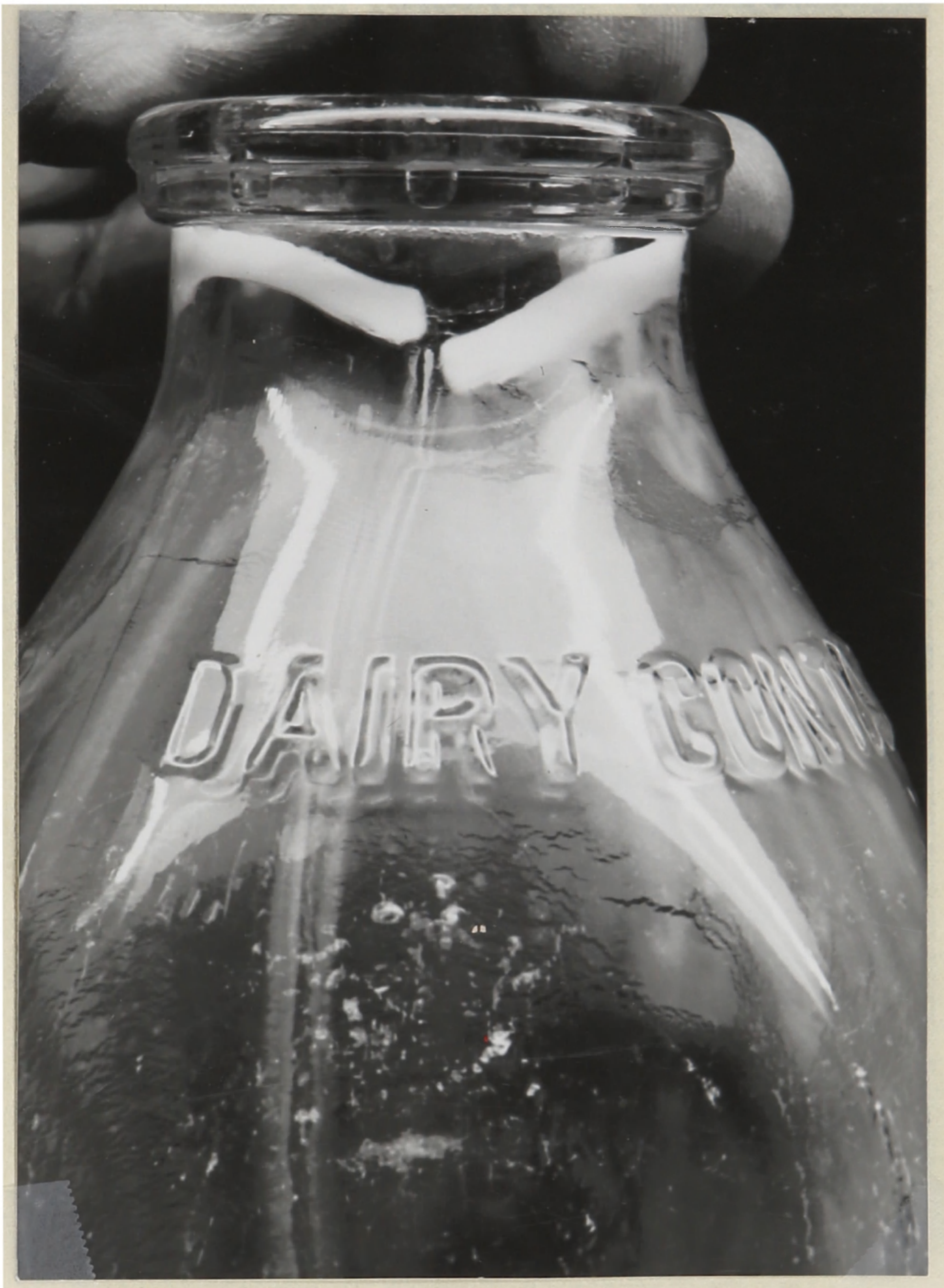

Figure 5. Appearance of fat ring about two minutes after pouring the milk from a quart bottle.

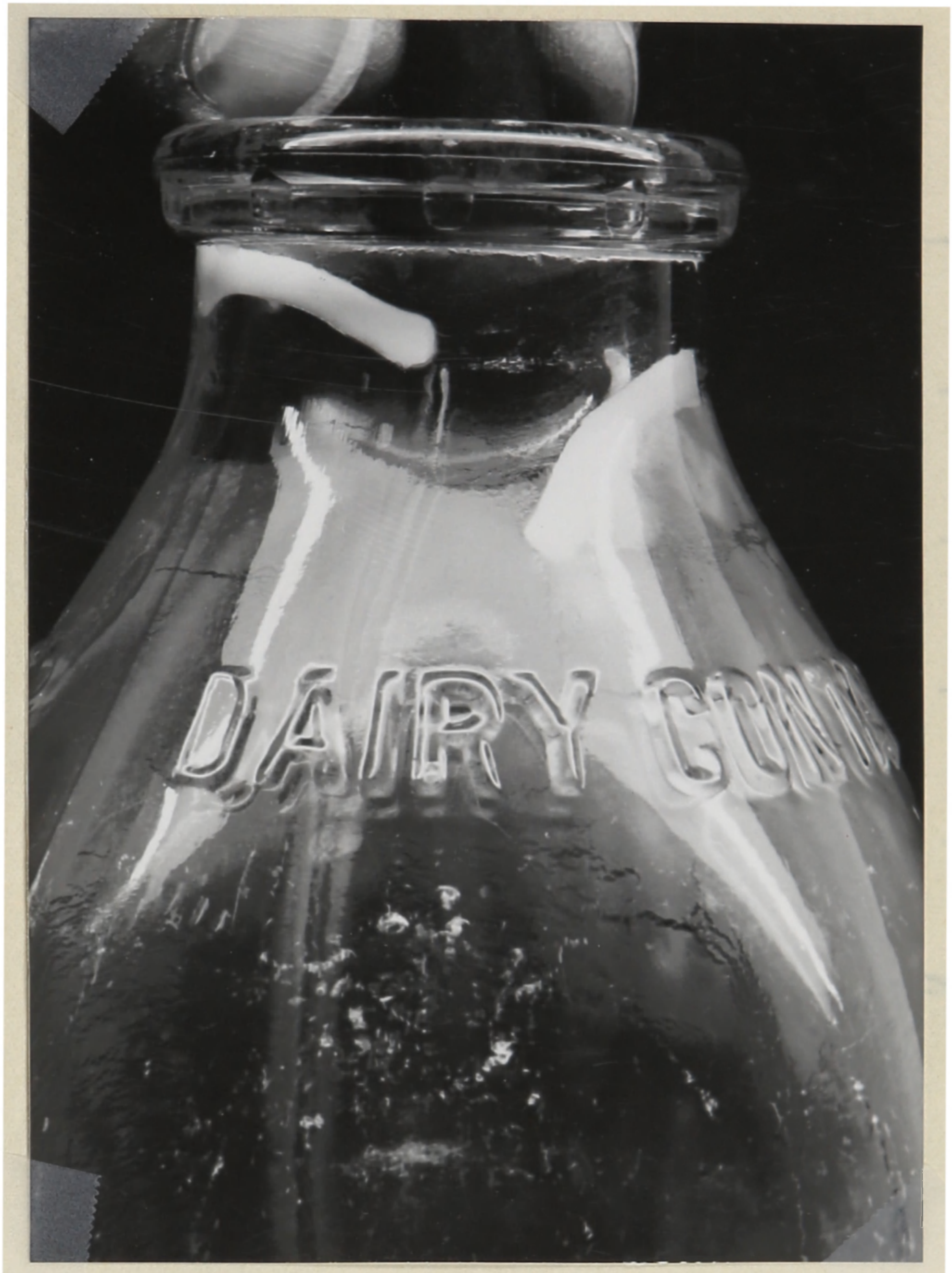

Figure 6. Appearance of fat ring about four minutes after pouring the milk from a quart bottle.

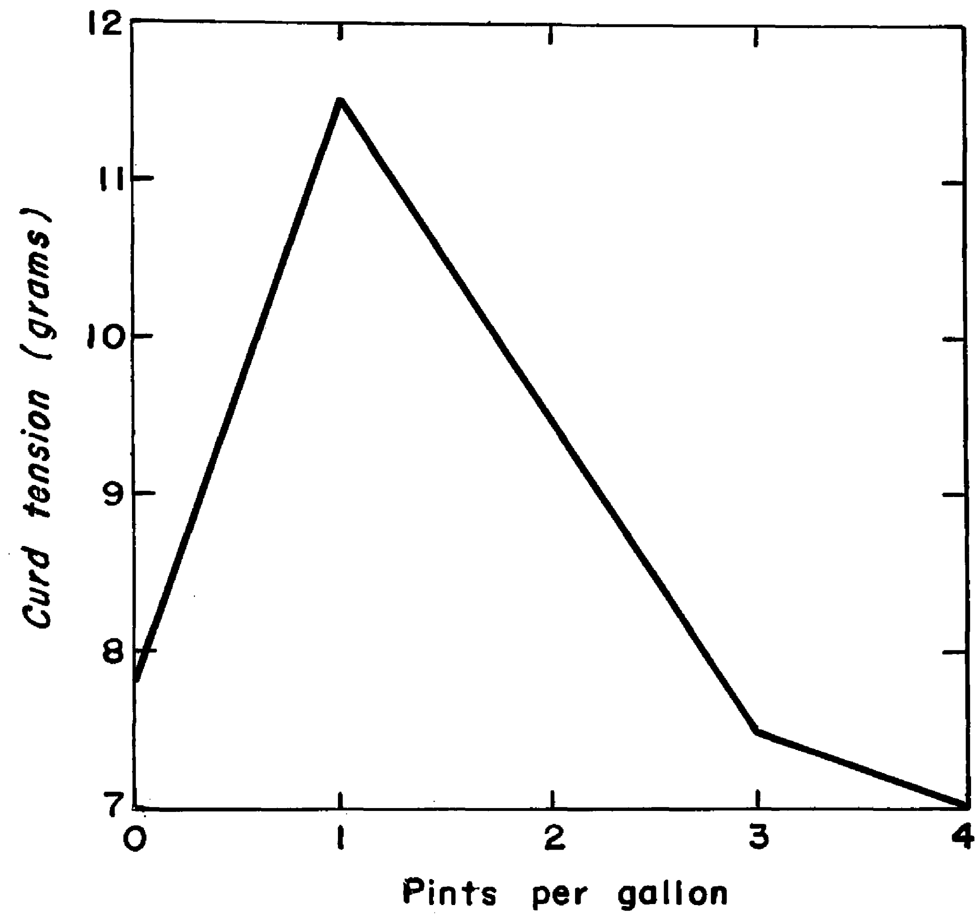

Figure 7. Influence of added water upon the curd tension of homogenized milk.

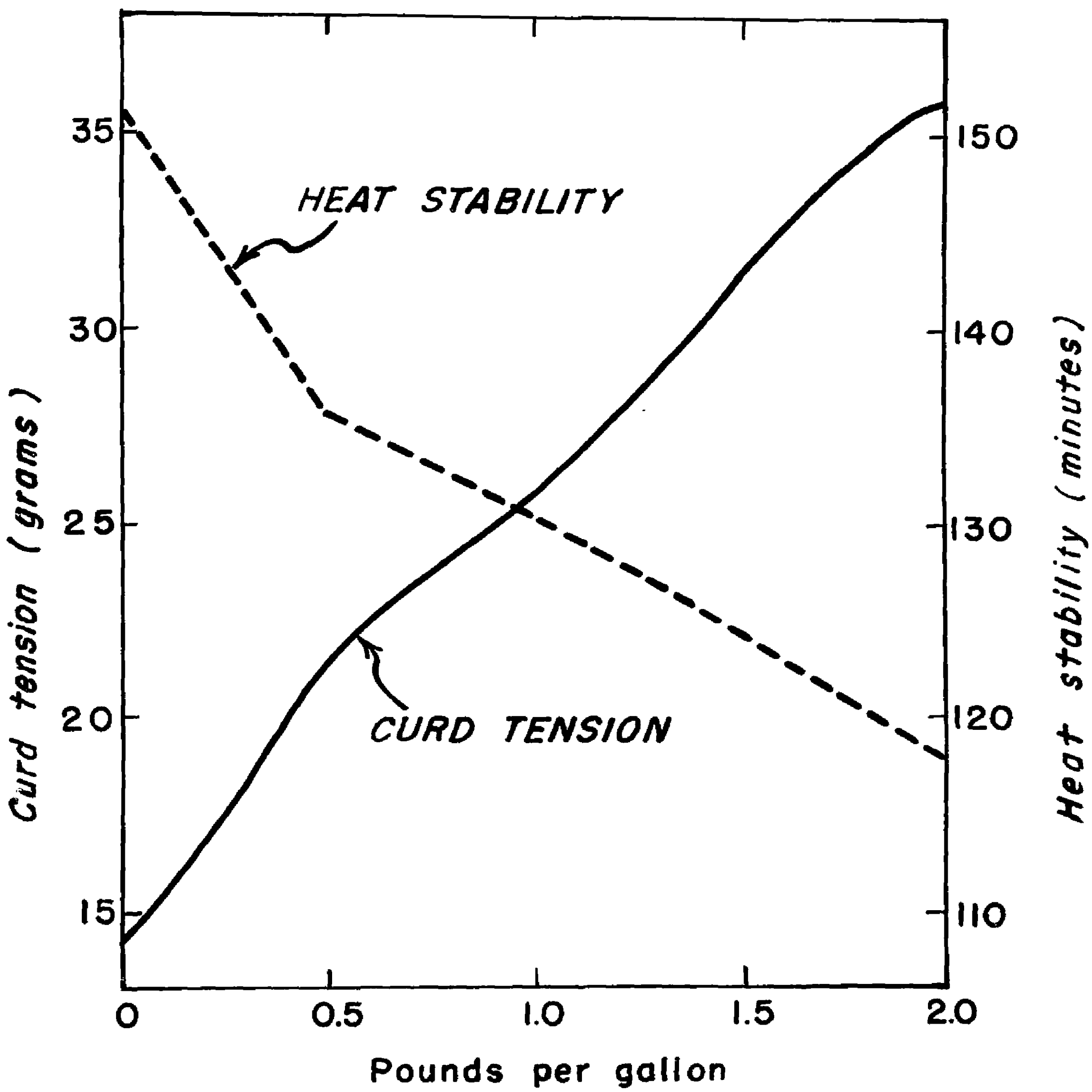

Figure 8. Influence of added solids-not-fat to milk upon the curd tension and heat stability of the homogenized product.

Figure 9. Appearance of fat ring on milk pasteurized at 170°F. for 30 minutes and homogenized at 170°F.

Figure 10. Appearance of fat ring on milk pasteurized at 170°F. for 30 minutes and homogenized at 150°F.

Figure 11. Appearance of fat ring on milk pasteurized at 170°F. for 30 minutes and homogenized at 130°F.

Figure 12. Appearance of fat ring on milk pasteurized at 170°F. for 30 minutes and homogenized at 110°F.

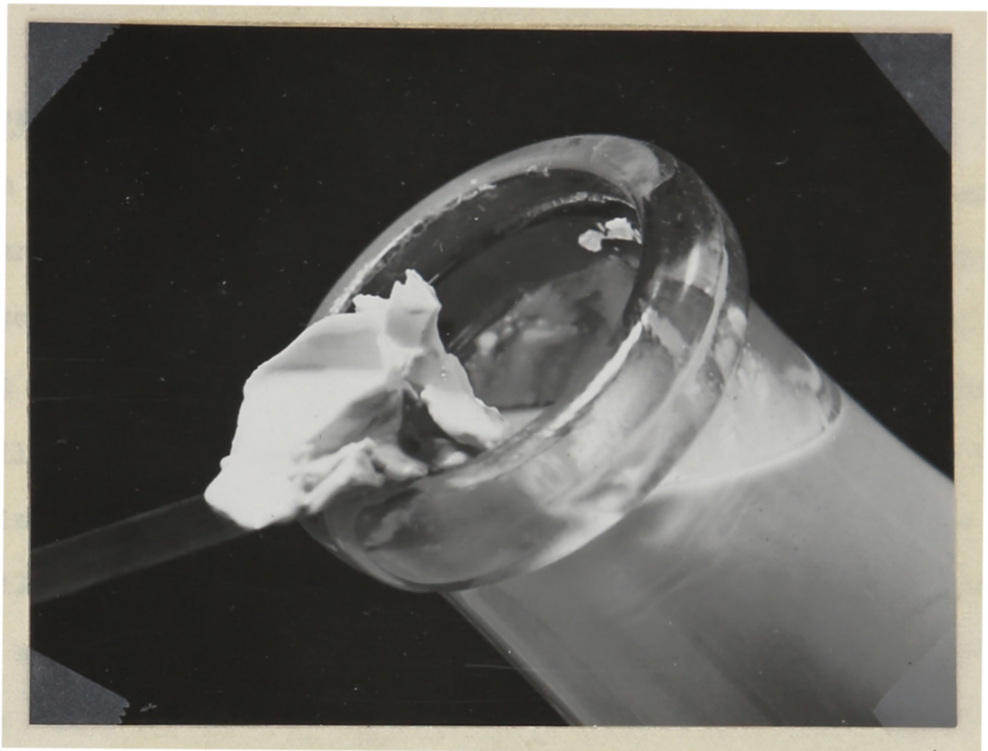

Figure 13. Fat ring material withdrawn from milk pasteurized at 170°F. and homogenized at 110°F.

DISCUSSION

Homogenization index

Data obtained show that heat shocking (merely warming to room temperature and then recooling) will accelerate the rising of fat on homogenized milk which has been in the bottle less than 48 hours. Such treatment of fresh milk will yield a greater index than on 48-hour bottled milk similarly heat shocked. This increased index is logical in that, with an increase in temperature of the milk, a slight decrease in viscosity will occur, thus allowing the fat globules more ease in rising. A second effect of higher temperature is that the fat tends to melt partially, giving a greater degree of plasticity to the fat globules, thus allowing for a greater ease of upward flow.

Milk which had been bottled 72 hours or longer failed to show this increase in homogenization index due to heat shocking. This indicated that the fat which would eventually come to the surface of the homogenized milk, would do so in 72 hours unaided by temperature increases above 40°F.

The method of sampling has been shown to be of importance when obtaining the top 100 ml. of a quart sample for homogenization index studies (Trout and Sheid, 1942). The present studies confirms these observations. Particularly was this true when a fat ring was present. In all cases the top 100 ml. was poured off, as recommended by Trout and Sheid (1942). However, in some cases the fat ring was not included in either portion of the sample, thus giving the effect that the fatty material was not a part of the original sample. In other cases the ring which clung to the bottle when the upper 100 ml. was decanted, was left in the bottle and mixed in with the lower portion of the quart sample. This latter procedure probably is the one which careless technicians might use when checking efficiency of homogenization by this method. These studies show that if such a procedure is followed a lower index will result.

Fat-ring analysis

The fat-ring material was shown to be about one-half fat, though this figure will vary with the character of the ring itself, some being much more tenacious and compact than others. The ring was found to have a relatively low protein content (1.5 percent), a condition which favors coalescence of the fat globules and destabilization of fat.

Analyses of fat-ring material resulting from milk homogenized at various temperatures show that the ring material contains a greater percentage of ash as the temperature of homogenization is increased. General belief is that any minerals in the fat-globule membrane are in association with the protein adsorbed there. Thus, a lower mineral content indicates a lower protein content. The lesser protein content of the rings occurring on milk homogenized at low temperatures results in heavier rings appearing on those samples. Because of this lesser protein content the fat forms a less stable emulsion.

The fact that the ring material "oils off" and the curd and water portion quickly settles out when warmed slightly shows that the oil-in-water emulsion of the milk was partially broken. This phenomenon is capitalized upon in the Creamery Package method for the continuous manufacture of butter (1946). In this process cream of about 80 percent fat is passed through a homogenizer at 75 to 90°C. (167 to 194°F.) at a pressure of about 90 atmospheres (1323 pounds). The resulting product can then be separated by use of a settling tank or a separator to a fat content of about 98 percent. This demonstrates the emulsion-breaking capacity of a homogenizer.

The product being homogenized in the case of butter manufacture is a high fat product. Consequently little opportunity exists for protein from the serum portion to adsorb onto the homogenized fat globule. In the case of milk, however, the protein content of the serum is higher and the homogenized fat globule in milk has a greater opportunity for stabilization than in a high-fat product. It is feasible however, that some of the same results could be obtained at lower-fat concentrations and even at lower temperatures than used for continuous butter manufacture.

<u>Effect of evaporation of water from gelatin solutions and from milk</u>

Studies on the rate of evaporation of water from different strengths of gelatin solutions show that the concentration of gelatin has little influence upon the weight loss due to evaporation. The greater concentration, however, tended to form a solid plug on the surface of the material indicating that as water is evaporated from the surface, the colloid forms a quite impervious film which stops further evaporation. The solutions of lower concentration showed no such film and a little higher evaporative capacity. Studies on homogenized milk showed, however, that milk loses approximately the same weight regardless of the percentage of total solids present. Observations showed also that the ring became more prominent upon dilution and with increasing lengths of holding time. Thus, evaporation plays a minor role in fat-ring formation.

Heating homogenized milk

The homogenization and pasteurization sequence varies in different plants and in different geographical areas. Thus, the possibility exists that where pasteurization follows homogenization, the effect of the heat treatment on the homogenized product might tend to cause more destabilization of fat. The homogenized fat globules, being smaller and possessing a greater surface area, might be more suspectible to the effect of heat upon its surrounding proteinaceous membrane. No doubt some protein is drawn from the serum portion of the milk to help coat the homogenized fat globule. Heat, being the denaturing agent, would then produce denaturation of the protein with greater ease than if the protein were in the serum portion. However, it was found that holding homogenized milk 30 minutes at 165°F. did not accelerate nor prevent fat-ring formation, but it did lower the heat stability, curd tension and surface tension. The lowered heat stability, of course, is due to the fact that the milk had been heated to 165°F. for 30 minutes prior to the examination of it for heat stability. The homogenizer played no part in this lowering of heat stability, because both samples were homogenized before being heated.

The lowering of the curd tension of the milk by heat treatment has been reported many times. The reason for this has never been adequately explained, but Doan (1938) states that it seems likely that the calcium ion is decreased, the electrostatic charge of the casein micelles increased, some albumin rendered insoluble and the protein itself denatured. The surface tension lowering might also be due to some denaturation of the albumin which then gathers on the surface of the milk.

Composition of milk as it affects some of its physical properties

1. Addition of water

An earlier observation on the effect of dilution of milk upon fat-ring prominence in test tubes was verified using milk in quart bottles. The greater the dilution of the milk with water, or the less the amount of added solids-not-fat in the sample of milk, the more prominent the fat ring. This can be explained on the basis that, with dilution, the stabilizing material is also diluted, thus freeing more water-insoluble fat, allowing it to rise to the surface.

The addition of water in the amounts used evidently was of such small quantities that the surface tension was not increased by the dilution, but was of sufficient quantity to increase the heat stability. This increased heat stability is probably due to the decreased solids concentration.

The explanation as to why the curd tension of milk increases with the first addition of water (12 percent added), but decreases thereafter is probably concerned with the calcium ion concentration in relation to other ions. Evidently, as some calcium is taken from the casein particle by the water, a more stable and harder curd will form, but with a greater removal of the calcium from the casein a weaker or softer curd results. Sommer and Hart (1919, 1922) have discussed this phenomenon in regard to heat stability of milk, showing that the addition of calcium or citrate ions will lower or increase the heat stability of milk.

2. Addition of cream.

The addition of cream to milk before homogenization greatly increased the fat-rising after storage of one week at 40°F. This can be attributed to an increase in the dispersed phase without a corresponding increase in the stabilizing agent.

The addition of the cream did not alter the surface tension, decreased the curd tension only slightly and decreased the heat stability materially. The curd tension was quite low due to the homogenization and may have shown a larger decrease if it were not for this fact. Hill (1923) stated that the fat content does not affect the curd tension of milk, but reported further that skimmilk had a higher curd tension than did the whole milk from which it came. However, Trout et al. (1935) found that the fat percentage in the milk was a factor. Other workers have shown that the curd tension was more closely correlated with the casein content of milk.

The decreased heat stability is due to the increased total solids concentration.

3. Addition of solids-not-fat

The addition of solids-not-fat to milk before homogenization caused a slight increase in the degree of fat-ring development. This suggests that the added protein had been denatured and would not resorb onto the fat globule.

Added solids-not-fat also caused a slight increase in the surface tension of milk, a definite increase in the curd tension, decreased heat stability and a slight increase in viscosity.

Solutions usually decrease in surface tension upon the addition of greater amounts of solute (McBain, 1950), but if this property increases upon increasing concentration it denotes negative sorption, which means that the solute is kept away from the surface of the liquid. If applied to milk in the case above, it would mean that the added solids-not-fat are negatively adsorbed and do not orientate in the surface of the liquid. Another explanation might be that the added solids-not-fat tend to stabilize and adsorb any free fatty acid or other surface-tension depressants present which thus removes these surface-active materials from the surface of the liquid.

Effect of homogenization pressures upon some of the physical properties of milk

Fat-ring observations show that high homogenization pressures are helpful in preventing this condition. This suggests that if the fat globules are broken into pieces sufficiently small, the prominence of the ring will be lessened.

The slight increase in curd tension due to high homogenization pressures is so slight that it hardly deserves mention, especially in light of the fact that there is no such consistent increase in individual milks. All the curd tensions of homogenized milk were below 14 grams, sufficiently low for properly homogenized milk. Any increase in curd tension due to higher pressures of homogenization could easily be considered as chance. Theophilus et al. (1934), Doan and Welch (1934) and Trout et al. (1935) found that a greater curd tension reduction resulted with greater pressures. However, Doan and Mykleby (1943) found that a pressure of 2500 pounds per square inch was sufficiently high to give maximum lowering of the curd tension. Pressures beyond 2500 pounds per square inch had little advantage so far as further lowering of curd tensions were concerned.

The increase in surface tension values due to higher homogenization pressures is very slight, but quite consistent, especially at pressures of 5000 and 6000 pounds per square inch. An apparent explanation for this is that with more efficient homogenizatinn a greater amount of the surface-active materials present in milk are adsorbed on to the colloidal particles which do not orient themselves in the surface. Thus, the surface is more free of these materials and tends to approach the surface tension of water. Webb (1933) also noted that higher homogenization pressures resulted in a greater surface tension.

The heat stability tests on milk homogenized at various pressures indicate that some factor other than pressures of homogenization is involved. A feasible explanation is that as milk is homogenized a greater amount of protein is adsorbed onto the fat globules leaving a greater ratio of salts to protein in the plasma portion than had been the case in nonhomogenized milk. The change in the ratio of calcium and magnesium to phosphates and citrates may change in the course of more efficient homogenization and, thus, the heat stability may increase or decrease depending upon the original concentrations of the salts in the milk. This change possibly may be brought about by the action of ionic calcium attaching itself to a casein particle which is then adsorbed onto a fat globule. This reaction would lower the ionic calcium in the serum portion of the milk.

On the average of four trials, it was shown that with higher homogenization pressures the viscosity of the milk was decreased. This indicates greater homogenization efficiency at the higher pressures, in that the smaller the fat globules are, the less will be the resistance of the liquid to flow.

Recirculation of milk through the homogenizer

Though the recirculation of milk through a homogenizer or the homogenization of milk more than once is impractical commercially, it is of interest academically to note its effect upon the milk. Milk was recirculated through the homogenizer for varying lengths of time and calculations made as to the theoretical number of times the milk was homogenized before portions of the milk were removed from the circulation process.

More efficient homogenization is suggested by the act of recirculation as noted by the disappearance of the fat ring when the milk underwent longer periods of recirculation.

Measurements show that the curd tension of milk is reduced further by recirculating the milk for five minutes, that is, homogenizing 1.56 times instead of just once. However, further homogenization showed no additional lowering of curd tension, thus indicating that the first homogenization was improved upon slightly by a second passage through the machine.

Surface tension measurements show slight increases for milk the more times it is homogenized, though upon homogenization of the milk 22 times the value drops over that homogenized only ten times. This is in line with the work of Webb (1933) in regard to homogenization pressures.

The high viscosity of milk homogenized 22 times probably is due to the heat generated by the process rather than by the homogenization itself. Caffyn (1951) found that the viscosity of homogenized milk would decrease with increases in temperature up to about 60°C. (140°F.), but above this tempeature the viscosity increased with a rise in temperature. Several workers have shown that homogenization increased the viscosity of milk over the same milk before homogenization.

Recirculation of milk through the homogenizer results in a decreased heat stability of the milk and this effect is more pronounced as the recirculation is continued. This might be explained on the basis that as milk is homogenized more efficiently, a greater amount of protein is drawn from the plasma portion leaving a greater concentration of calcium ion per unit of protein. This increased ratio of calcium ion to protein causes destabilization and thus lowers the heat stability. Another explanation might be that with more efficient homogenization, the protein is exposed to a greater surface area when coating the fat globules. When on the surface of these globules, the protein is in somewhat of a quiescent stage. The application of heat logically would have a more pronounced effect on these proteins when in this state than when in a colloidal state in the plasma portion of the milk.

Some of the mechanical energy from the homogenizer is converted to heat energy and the heat transferred to the milk as the milk is being homogenized. A temperature rise of about 35°F. is noted for the total of 22 times the milk is passed through the homogenizer. As would be expected however, the temperature rise is greater at the lower temperatures of homogenization. This rise of a little over 1°F. per passage is well below that secured by other workers who found as much as a 17°F. increase when homogenizing at 5000 pounds pressure and at 40°F.

<u>Influence of pasteurization and homogenization temperatures upon fat destabilization</u>

The reason for the heavier fat ring appearing on milk which was homogenized at temperatures below the pasteurization temperature can not be definitely stated at this time. However, some facts appear and hypothesis can be proposed:

a) Adsorption of protein or other stabilizing agents is essential for the stabilization of fat in milk.

b) The higher the temperature of pasteurization, the more protein will be denatured or otherwise altered in character.

c) Denatured protein has different properties than the native protein; a decreased hydration and often transformation of a hydrophilic sol into a hydrophobic sol occurs.

d) The lowered capacity of protein to adsorb onto the fat particle is not due entirely to the effect of heat, but in conjunction with the effect of cooling.

The first hypothesis is supported by Glasstone (1946) and other workers on the stability of emulsions. The second is supported by Rowland (1933-34) (1937) and by Haurowitz (1950). The third is supported by McBain (1950) and the fourth by data reported in this paper.

Sommer (1952) discusses the composition of the fat-globule membrane reported by different workers, and concluded that it is composed largely of protein and phospholipids. Thus, we can assume that the stabilizing agent for fat in milk is protein and phospholipids.

In addition, the works of Rowland (1933-34, 1937), show a larger amount of albumin and globulin denaturation occurs with increases in temperature and length of holding time. Further, Haurowitz (1950) showed that there was an equilibrium set up between denatured protein and protein in the native state, and this equilibrium shifts with changes in the temperature of the system. At the higher temperatures a larger quantity of the protein is in the denatured state, and, conversely, at the lower temperatures. He explained also that as the temperature was increased the reaction "native protein ⟶ denatured protein" was carried out, that an increase in entropy occurs which amounts to 180 calories per degree per mole. This large increase in entropy is the driving force in denaturation so that at the higher temperatures, the reaction will actually be exergonic and no energy will need be applied for the reaction to proceed.

Thus, with decreases in temperature an increasing amount of energy will need to be applied if the protein is to go again into its native state, although the question of reversibility is not yet answered.

According to McBain (1950) denaturation of protein causes loss of hydration and often causes the conversion of a hydrophilic sol into a hydrophobic sol. With this loss of water-holding capacity of the protein, one of the factors promoting stabilization is lost because the protein will no longer hold the fat within the body of the liquid. Any stability which the colloid possesses will be due to electric charge and none to the fact that water is adsorbed onto the particle. With denaturation and the unfolding of the protein particle, it might be assumed that the electric charge on the protein is lowered also because of the greater distance between the two polar groups on the protein molecule.

However, this lower capacity of the protein to adsorb onto the fat particle is not due entirely to the effect of heat, but also to the effect of cooling. This is evidenced by the fact that milk can be homogenized at the pasteurization temperature and no fat ring will develop. But milk pasteurized at this same temperature and cooled somewhat before homogenization will form the ring. Thus, the phenomenon of lowered protein-adsorption capacity is due not only to the effect of heat, but to the effect of heat in conjunction with the cooling effect. According to the kinetic theory, cooling of the milk protein particles causes them to possess less energy and move slower within the liquid. With this slow movement, comes less opportunity for a protein molecule to collide with a fat particle.

Thus, it is shown that protein adsorption is necessary for fat stabilization and that this protein adsorption tendency becomes decreased after the milk has been heated to a higher temperature then cooled before homogenization.

Another effect of dehydration of protein particles is decreased viscosity of the liquid (Glasstone, 1946). This has the effect of giving greater ease to the rising of fat in the liquid.

A patented process of continuous buttermaking by The Creamery Package Company (1946) reported by Wiechers and Goede (1950) uses a homogenizer to break the fat emulsion. This process is carried out at about 171°F. and at about 1300 pounds pressure on a product of about 80 percent fat. Although this process involves a higher temperature, lower pressure and a product of higher fat content than that used in the present study, some of the same causes of de-emulsification might well occur.

<u>Foreign fats homogenized into skimmilk</u>

In regard to the character of the fat, it has been demonstrated in this study that the presence of sterols will hasten the fat-ring formation on homogenized milk. This is evidenced by the presence of the large plug on the sample containing lanolin as the added fat. Lanolin contains a high percentage of sterols. These sterols, being insoluble in water, can, in a large measure, regulate the hydrophilic nature of the fat and lecithin with which they are associated in the milk. Corran (1943) discusses the effect of lanolin when in the same system as lecithin.

The lanolin has the effect of contributing to a water-in-oil emulsion while the lecithin has the effect of promoting an oil-in-water emulsion. Thus, in oil-in-water emulsions, the larger the quantity of lanolin (or sterols) present, the greater the instability of the emulsion.

Samples to which tributyrin was added show a complete absence of fat-ring formation indicating that the fat does not contain substances which are hydrophobic. Yet, it can not be said that this fat exhibits negative sorption, because the surface tension is decreased on those samples containing this fat, as well as on those containing lanolin. It is feasible, thus, that tributyrin does tend to rise on the milk, but because of its low melting point and greater solubility in water the fat does not appear as a plug or ring on the milk.

The greater curd tension in those samples containing hydrogenated cottonseed oil flake or hydrogenated tallow in comparison to those containing butterfat is likely due to the high melting point of the former two fats. Because these fats are solidified at the temperature at which the curd tensions are determined, the penetration of the knife is retarded somewhat. Likewise, the melting point of tributyrin, being quite low, is all liquid at 90°F. thus tending to weaken the bonding between the protein particles.

Most of the foreign fats used in this study cause an increase in surface tension, which indicates negative sorption. The greatest decrease in surface tension was noted when lanolin was used. This indicates that this fat is drawn to the surface of the milk or has a positive sorption characteristic.

The decrease in heat stability exhibited by the use of tributyrin indicates that this fat underwent some lipolysis before the heat stability of the sample was measured. However, a sample of the fat was dissolved in alcohol, a drop of phenolphthalein added and, upon the first addition of 0.1 N. sodium hydroxide, the mixture turned pink indicating the acid content of the tributyrin was mil. The extreme bitterness of the sample, as revealed by flavor judgments after 24 hours, indicated that some hydrolysis had taken place in the samples. With the appearance of these fatty acids, an increase in acidity and a decrease in pH occur. This same phenomenon undoubtedly explains why the relative viscosity is slightly greater on the tributyrin added samples. However, all viscosity measurements are within such a short range, that any effect due to added fats is quite small.

The Farrall index shows some indication that a ring may or may not form on milk containing butterfat, but it was not possible, in this study, to show a relationship between the Farrall index and the formation of a fat ring on fat-substituted homogenized milk.

SUMMARY

Extensive studies have been made on the causes and prevention of fat-ring formation on bottled homogenized milk. These studies involved not only milk fat but also several foreign fats, which necessitated further investigation into some of the physical properties of normal homogenized milk and of homogenized synthetic milks.

Homogenization index studies show that heat-shocking will accelerate the rise of fat in homogenized milk, if done within 72 hours of bottling. However, if heat-shocking occurs after 72 hours, it will have little effect upon the index. The method of sampling is of importance when the fat ring is present. To obtain a true indication of the homogenization efficiency as judged by the United States Public Health Service homogenization index, the ring material must be included in the upper portion of the quart sample. This ring, however, being largely de-emulsified fat, is relatively immiscible and thus when the fat ring is present, an accurate test can not be made of the sample.

The fat-ring material was found to contain about one-half fat, largely in the de-emulsified state, and about 5.5 percent solids-not-fat.

Studies of water evaporation from gelatin solutions of concentrations of 0.01 to 10 percent show that the weight loss due to evaporation varies very little among solutions of different concentrations. Weight losses from milk exposed to the air of a household refrigerator showed that evaporation was of little importance so far as the development of ring formation was concerned. Loss due to evaporation did not vary materially with milk of different compositions ranging from 11.68 to 16.68 percent total solids.

Pasteurization of homogenized milk at 165°F. for 30 minutes neither accelerated nor prevented fat-ring formation on homogenized milk, but this heat treatment lowered the heat stability, curd tension and surface tension of the milk.

Dilution of milk with water resulted in a more prominent fat ring and increased heat stability of the milk. However, the surface tension of the milk was affected very little, whereas, the curd tension was increased with the first addition of water, but decreased upon further added increments of water.

The addition of cream to milk before homogenization greatly accelerated the fat rising, slightly decreased the curd tension, materially decreased the heat stability, but did not alter the surface tension.

The addition of solids-not-fat to milk before homogenization enhanced fat-ring formation and resulted in a slightly greater surface tension, a definite increase in curd tension, a decreased heat stability and a slight increase in viscosity.

Higher-than-normal homogenization pressures prevented fat-ring formation, caused an increase in surface tension and a decreased viscosity.

Rehomogenization of milk by recirculation as many as 22 times, resulted in a more stable fat emulsion, as shown by the absence of a fat ring, and a decreased heat stability of the milk.

When milk was homogenized at the pasteurization temperature, fat-ring formation was kept at a minimum; but if the milk were cooled somewhat before homogenization, the fat-ring development was enhanced. The higher the pasteurization temperature followed by subsequent cooling to a specific temperature, the greater was the degree of fat rising on milk after homogenization.

Of the 19 different foreign fats from various sources which were homogenized into skimmilk, the one that yielded the greatest amount of fat-ring formation was lanolin. When lanolin was used, the fat ring appeared quite prominent, indicating that this fat, containing a higher percentage of sterols, when mixed into milk tended to be hydrophobic. However, when tributyrin was incorporated by homogenization into skimmilk, no fat ring was noted, indicating a greater hydrophilic nature than when lanolin was the source of fat. The viscosities on foreign-fat samples were quite constant. Most samples tasted oily, but the tributyrin and trimyristin samples tasted extremely bitter. When fats such as "Marbase (S)", "Marbase (C)" and "Velvet", which are recommended for use in "filled milks", were used, a non-objectionable tasting product was obtained.

Data indicated that the Farrall-index method for microscopic determination of homogenization efficiency was of little or no value so far as foreseeing the fat-ring defect when fats of a foreign source were used in the milk.

CONCLUSIONS

The fat-ring material sometimes occurring on homogenized milk is composed of about one-half fat, largely in the de-emulsified state, and a relatively small amount of protein.

The testing of homogenized milk for homogenization efficiency by the United States Public Health Service homogenization index is less reliable when a fat ring is present on the milk than when it is not. The ring material will not remix readily into the sample.

The addition of cream to milk before homogenization or the dilution of homogenized milk with water favor fat-ring formation. The addition of solids-not-fat also slightly intensifies this defect. Evaporation of water from milk plays no part in the destabilization of the fat.

Homogenization of milk at temperatures below that at which it was pasteurized is conducive to fat-ring formation. For example, if milk were pasteurized at 150°F. for 30 minutes, then cooled to 110°F. before being homogenized, the likelihood that a fat ring would develop is great. But, if the milk were homogenized at 150°F., the possibility of fat-ring formation would be greatly lessened.

High homogenization pressures or rehomogenization lessen the prominence of the fatty ring. Further heating of homogenized pasteurized milk neither accelerates nor prevents its formation.

Fats containing a large amount of sterols, such as lanolin, are "serophobic" and will form a heavy cream plug when homogenized into pasteurized skimmilk.

Three fat products, "Marbase (S)", "Marbase (C)" and "Velvet", sometimes used to replace butterfat in frozen desserts as well as in "filled milks", as used in this study, have little influence on fat-ring formation. When homogenized into high-quality pasteurized skimmilk, these foreign fats did not yield a bitter, nauseating flavor as was noted when tributyrin and trimyristin fats were used.

When hydrogenated fats are homogenized into pasteurized skimmilk the fats apparently have little effect upon fat-ring formation, but do cause the products to have high curd tensions.

The physical properties of milk, such as curd tension, surface tension, heat stability and viscosity are of little value in the detection of potential fat-ring formation on homogenized milk. Likewise, while the Farrall index is of value when normal homogenized milk is used, it is of little or no value in foreseeing fat-ring development when foreign fats are used.

When a dairy plant operator experiences difficulty in producing homogenized milk free of fat ring, he should investigate the following possible contributing factors:

a) Homogenization pressure

b) Homogenization temperature, especially with reference to that of pasteurization

c) Contamination of homogenized milk with nonhomogenized milk

d) Mechanical condition of the homogenizer

e) Possible dilution of the milk with water or the occurrence of milk naturally low in solids-not-fat

Experiments reported in this paper show that when normal milk is homogenized immediately after pasteurization, a) at the pasteurization temperature, b) at a pressure of 3000 pounds per square inch on the first stage and 500 pounds on the second stage, c) with a properly operating homogenizer, and d) with no contamination by nonhomogenized milk, a product can be produced which will show little or no fat-ring formation after one week's storage at 40°F.

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