EFFECT OF OXYGEN ON THE DEVELOPMENT OF OFF-FLAVORS IN UHT MILK

by

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INTRODUCTION

Ultra-high-temperature (UHT) milk is milk processed at a combination of times and temperatures in the ranges of 130-150°C, for 2-8 s, and aseptically packaged to produce a commercially sterile product (35). After processing and packaging, the product requires no refrigeration (17). UHT milk is not a new idea. It has been on the European market for over 25 years and has captured 25% of the fluid milk market in France, 40% in Switzerland, and 50% in both Germany and Italy. It has been produced in the United States since 1940 but only within the past 2 years have Brik Pak (non-hermatically sealed) type cartons been legalized for sterile non-acid foods such as milk (12,17). Some advantages of this type of UHT milk are that it has a longer shelf life and requires no refrigeration upon storage. By eliminating refrigeration, the cost of storage and distribution are reduced because there is no longer a need for refrigerated-trucks or-display cases. Also, it can be stockpiled if there is an oversupply of milk, as well as used for recreational or away from the home consumption (12).

With such appealing advantages, why is UHT milk not readily accepted in the United States? The abundance of fresh fluid milk, adequate energy, and refrigeration has prevented a ready market for this milk, but with rising energy costs, it may be more in demand in the near future (17). A major obstacle in its acceptance is a serious off-flavors that develop during storage. Immediately after processing, UHT milk has a highly cooked flavor which dissipates with time and later develops stale and/or oxidized flavors upon storage (12). The American consumer finds these flavors objectionable. Futhermore, they are accustomed to the gallon size container, whereas UHT milk is most often sold in unrefrigerated half liter and liter size Tetra Brik Pak containers. Exceptional marketing strategies will be necessary to change consumer attitudes that high quality fresh milk must be refrigerated. Also, the unknown relative costs of implementing UHT processing systems is an economic barrier confronting the dairy-processing industry and dairy-distributors (17).

This research was designed to probe the nature of the flavor problems that develop upon storage of UHT milk. The specific objective, however, was to investigate the role of dissolved oxygen as a contributor to flavor deterioration in sterile milk during storage. A deoxygenating system and an indirect UHT system were first designed and constructed. Afterwards, both skim and whole milk were sterilized and subjected to a four month shelf life study. The effects of oxygen, antioxidants, and storage temperature were examined. The following parameters were monitored: sterility, dissolved oxygen levels, concentration of antioxidants, changes in volatile materials, oxidation, rancidity, and flavor.

REVIEW OF LITERATURE

UHT SYSTEMS

Direct

The various operations of direct UHT systems have been described by many authors (13,27,35,43,51). In these systems, sterilization is accomplished either by injecting steam into milk or milk into steam. The resulting products increase in temperature rapidly and are diluted by about 10% with water. During cooling, the milk is passed through a vacuum chamber where the steam-condensate is removed. Burton (13) comments that direct systems are more complex, require additional plant- and control- systems, and are more expensive both in purchasing and processing costs.

Indirect

No actual contact occurs between the product and the heating agent (usually steam) employed with indirect systems (13,27,35,43,51). Milk is heated through a barrier (usually stainless steel corrugated plates or tubes) which separates the milk from the heating agent.

FLAVOR OF STERILE MILK

Effect of process

Direct vs. indirect. Jelen (27) feels that both direct and indirect processing impart a UHT flavor detectably different from pasteurized-homogenized milk; however, the direct methods yield products with superior flavor attributes. The higher total heat treatment of indirect methods produces a more intense cooked flavor. In an interview by Sandberg-Howe (41), Dr. Dennis Westhoff from University of Maryland explains that "Since milk, at times 'burns' onto this stainless steel pipe, I don't think you'll find anybody in UHT research who will tell you that indirect systems are as good as direct as far as flavor goes." He continues that "the lack of turbulence in the 'free-falling-film-system' lends itself to a better flavor and the milk never touches a piece of metal, hotter than itself. You never have 'burn-on' ". Shew (43) reported that a disadvantage of direct systems is that the product is subjected to steam distillation which removes off-flavors but also removes some characteristic volatile flavors of milk resulting in a flat flavor.

Time and temperature. In the United States there is no official (federal or state) definition for sterilized milk products packaged in flexible paper containers, whereas in Europe, the term commercial sterilization implies processing temperatures between 130-149°C with a holding time of at least 1 s. These time-temperature combinations vary somewhat from country to country (2). Like the U.S., the Canadian Food and Drug Act states no legal definition of UHT processing, but the definition of "a continuous sterilization process operating at temperatures higher than 130°C (usually in the vicinity of 140-150°C for several seconds)" is generally accepted (28).

Burton (13) points out that as the processing temperature increases, the time must decrease, otherwise the product will be overprocessed. The time-temperature combination of processing is related to the heat treatment necessary to kill microorganisms. If the present processing conditions are insufficient to kill spores, a small increase in temperature (about 1.5°C) could compensate for the effect of doubling the holding time.

Haytham et al. (26) studied the effects of heat treatment by measuring volatile compounds and relating them to flavor. In their research, milk was indirectly processed at 140°C for 3 s and 90 s and stored at ambient temperatures for 112 d in the dark. After 7 d of storage, samples held for 3 s during processing had flavors described as cabbage-like, slight astringent and cooked whereas those held for 90 s were described as cooked, fishy, and rice-pudding-like. After 1 mo, the 3 s samples were slightly stale while those held for 90 s were definitely stale and had flavors similar to the ones detected early in storage. The shorter holding time appeared to produce a more acceptable product flavorwise.

Likewise in a study by Hansen et al. (24) a steam injection system was used to process milk at various temperatures and holding times. Milk was processed at 138, 143, and 149°C for 20.3 s; 143 and 149°C for 6.9 s; and 149°C for 3.4 s. At 0 wk, milk held for 3.4 s at 149°C gave the best predicted flavor score with a decrease in score as the heat treatment increased. Therefore when considering flavor, the least severe heat treatment appears to produce the best quality product.

Effect of storage time and temperature

The effect of the processing time and temperature does not appear to be as critical in flavor alteration as the effect of storage time and temperature. Burton (13) states that the flavor of any milk based product deteriorates on storage and that the rate is faster at higher storage temperatures. Many researchers have found UHT milk stored at refrigeration temperatures retains an acceptable flavor longer than milk stored at higher temperatures (8,10,23,24,25).

Ashton (8) explained that the flavor of UHT milk changes in five stages. 1) Immediately after processing the milk may have cabbage-like flavors or sulfurous odors to some degree. 2) After 2-3 d storage at 4.4-21.1°C, the taste and smell is more acceptable and is comparable to pasterurized milk. A higher storage temperature (26.7-37.8°C) results in rapid disappearance of the initial taste and smell, but a slight residual cooked flavor remains. 3) After 5-12 d storage at 4.4-21.1°C, the milk has its best flavor—pleasant creamy taste similar to/or slightly richer than pasteurized-homogenized milk. 4) After 12-18 d, the milk stored at 4.4-21.1°C develops a flat chalky or a residual cooked flavor similar to milk stored at 32.2°C for 2-3 d. 5) After about 19 d of storage at 21.1-37.8°C, a strong residual cooked flavor is detectable and secondary flavors possibly due to oxidation may develop and become stronger with age. Samples stored below 21.1°C may also develop similar flavors but they are probably milder at this stage and become worse with age.

Hansen et al. (25) found flavor changes occurring upon storage to be similar to those described by Ashton (8). Milk was processed by direct steam injection at temperatures of 107, 121, 135, and 143°C with holding times of 1.5 or 5 s and at 135 and 143°C for 10 s. Samples tempered to 4°C [Note: Temperature recommended by ADSA for judging milk is 15-21°C (60-70°F)] and then tasted by twenty-five untrained panelist were described. After 2 d storage, they had a strong cooked flavor but the milk processed at 135 and 143°C for 10 s was scored acceptable. (In opposition to these findings, other researchers (26,34,50) have found a strong cooked flavor in samples receiving a more severe heat treatment.) On day 9, a slight cooked flavor was apparent with a sweet caramel taste which was still present at 16 d. By the 23rd and 30th day of storage, the milk had developed a very bland, flat flavor, except for the sample processed at 143°C which was still slightly sweet. Milks processed at 135 and 143°C for 10 s were rated acceptable to good during the study from day 2-30. The maximum flavor score for milk processed at 135 and 143°C were from 16-23 d.

Bassette and Jeon (10) found that UHT milk develops an objectionable stale flavor during storage and its intensity depends upon storage time and temperature. Milk processed by steam infusion was slightly stale after 1 mo of storage at room temperature and the stale intensity increased upon extended storage. However, milk stored under refrigeration had relatively high acceptability scores after 3 mo storage. The stale intensity and flavor acceptability were inversely related.

Hansen et al. (24) used prediction curves to relate flavor scores, storage temperature, and processing conditions. From these prediction curves (plots of predicted taste panel scores computed from the fitted regression lines against storage temperature for various storage times and process treatments) they determined that there were critical storage temperatures where the flavor of milks with certain heat treatments would either increase or decrease. A relatively mild heat treatment (149°C for 3.4 s) required storage below 12°C for flavor improvement to occur; an intermediate treatment (143 and 149°C for 6.9 s) required a storage temperature between 12°C and 32.5°C; while a more severe heat treatment (149°C for 20.3 s) required storage below 32.5°C for flavor improvement. The samples with the severe heat treatment improved up through 12 wk but thereafter the flavor decreased. The highest predicted scores for each process treatment were at 4°C storage. From the prediction curves it appears that milk processed with a mild heat treatment requires a lower storage temperature to maintain good quality. When the milks were evaluated by an untrained taste panel, samples having a mild heat treatment were acceptable for only 1-2 wk whereas milks with an intermediate to severe heat treatment generally became acceptable around 4 wk and lasted through 20 wk (24).

In another study by Hansen and Swartzel (23), an equation to predict taste panel scores by relating the percent fat, storage time, and storage temperature was derived. The equation was reliable only after 12 wk of storage because flavors resulting from processing treatments are more important the first 12 wk. They found that, in general, milks with higher fat contents received higher flavor scores at all storage temperatures; however, as the storage temperature increased, flavor scores decreased.

CHEMICAL CHANGES ASSOCIATED WITH FLAVOR CHANGES IN STERILE MILK

Autoxidation

Effect of dissolved oxygen. Chemical and flavor changes resulting from the dissolved oxygen concentration in UHT milk have been investigated by many researchers (2,18,29,50,54). Zadow and Birtwistle (54) concluded that the development of off-flavors in UHT milk upon storage were dependent on the level of dissolved oxygen after processing and the storage temperature. Samples stored at 2°C maintained a 'very good' flavor independent of oxygen concentration. However, as the storage temperature was elevated, the dissolved oxygen concentration became important. When samples were stored at 20°C, samples with an initially high oxygen content were very good at first but developed rancid and oxidized flavors upon storage; samples with a low oxygen content were rated 'poor' the first few weeks but increased to 'acceptable'; and samples with an intermediate oxygen content were consistantly rated 'excellent' throughout the trial. Samples stored at 38°C developed browning and rapidly developed off-flavors independent of the oxygen content. These researchers concluded that an intermediate concentration of dissolved oxygen allowed for oxidation of the free sulfydryls, therefore, reducing the cooked flavor, while a high oxygen concentration would maintain an excess of oxygen after oxidation of free sulfhydryls, resulting in an increase in the development of the rancid and oxidized flavors (54).

Unlike Zadow and Birtwistle (54), Thomas et al. (50) found that flavor acceptability was independent of the oxygen concentration after the first few weeks when UHT milk was stored at 20 ±4°C. Milks with an initially high oxygen concentration was preferred up to 8-13 d, but thereafter, the dissolved oxygen concentration did not influence flavor. An increase in flavor acceptability with the first few days was related to a decrease in free sulfydryl content and cabbagey flavors. Milks with high oxygen contents exhibited more rapid losses of these compounds whereas they persisted several weeks in milks with a low oxygen contents.

Jeon et al. (29) found that aldehydes appeared to be the most important compounds contributing to the off-flavor. An increase in aldehydes was dependent on oxygen concentration and the storage temperature. They postulated that formation of n-hexanal was related to the oxygen concentration because n-hexanal was formed rapidly in milk stored at 35°C until the oxygen was depleted. At this time, n-hexanal formation ceased. However, at 22°C where available oxygen concentration remained higher, the hexanal concentration continuously increased. The pattern of changes in concentrations of n-pentanal, n-heptanal, and n-octanal during storage were similar to n-hexanal.

Decreases in the amount of dissolved oxygen were observed upon storage (29,50,54). Jeon et al. (29) found that oxygen content decreased more rapidly and to a lower concentration with increased storage temperatures. However, Earley and Hansen (18) found that the dissolved oxygen concentration increased at a storage temperature of 24°C, but decreased at 40°C. They explained this by postulating that no autoxidative reactions occurred at 24°C (indicated by a decrease in n-alkanals and no detectable off-flavors), therefore, since the carton was permeable to oxygen, the concentration increased because it was not being used during autoxidation. However, at 40°C the oxygen was utilized in chemical reactions as fast as it entered the carton. Long chain alkanals were present at concentrations 8-13 times greater than short chain alkanals; however, they decreased upon storage at 24 and 40°C. According to Parks et al. (39), long chain n-alkanals are found in fluid milk and associated with its typical flavor; they are not products of oxidation. Short chain n-alkanals were also found to decrease with storage. Samples stored at both temperatures did not develop oxidized flavors; however, the samples stored at 40°C were unpalatable after 6-8 wk storage.

Mehta and Bassette (36) found that milk stored in cartons more permeable to oxygen (polyethylene lined) developed a stale flavor faster than those stored in cartons less permeable to oxygen (aluminum foil-lined). Also, an increase in compounds associated with oxidized flavors (n-propanal, n-pentanal, and n-hexanal) paralleled the stale flavor development.

Cooked flavor. Immediately after processing, UHT milk has a highly cooked flavor that dissipates with time (22,25,26,36,42,44,50,54). The cooked flavor is related to the liberation of volatile sulfur compounds or free sulfhydryl groups (2). Hansen and Melo (22) found that increases in sialic acid and active sulfhydryls groups may result from the unfolding of protein moieties thus exposing: 1) glycoproteins containing sialic acid and 2) "buried" sulfhydryls and/or reduction of disulfide bridges in milk protein. They also found whey proteins were the most susceptible to denaturation while caseins were least affected. Melo and Hansen (37) found that beta-lactoglobulin was a major source of available sulfhydryls, that it was more susceptible to denaturation, and formed more reactive sulfhydryls than alpha-lactalbumin. The reactive sulfhydryls may be involved in a variety of reactions (eg. lost by volatization, oxidized to disulfides, or "reburied" in the protein structures). Studies suggest only a small portion are actually volatilized. Storage temperature influences what will happen to reactive sulfhydryls; room temperature favors their oxidation (40). Oxygen in milk will oxidize free sulfhydryls and improve the flavor initially (50,54). Non-oxidized reactive sulfhydryls may be involved in sulfhydryl-disulfide interchange reactions with various proteins (40).

Haytham et al. (26) studied the effect of heat treatment on milk and related volatile compounds to flavor changes. Milk was processed at 140°C for 3 and 90 s, stored at ambient temperatures for 112 d. The concentration of all compounds were higher in the milk held for 90 s. Hydrogen sulfide, carbonyl sulfide, methanethiol, dimethyl sulfide, carbon disulfide, and dimethyl disulfide were found in heated milk. During storage, decreased concentration of hydrogen sulfide, methanethiol, and carbon disulfide paralleled the decrease in a cabbagey off-flavor.

Scanlan et al. (42) heated milk to 146°C for 4 s by the indirect method and found the following compounds to be heated-induced: $C_{3-5,7-11,13}$, n-methyl ketones, the $C_{8,10,12}$ delta-lactones, benzaldehyde, furfural, phenylacetaldehyde, vanillin, oct-1-en-3-ol, n-heptanol, 2-butoxyethanol, maltol, acetophenone, benzonitrile, benzothiazole, and diacetyl. The concentration of diacetyl was 5 ppb in raw milk whereas it was 38 ppb in heated milk. They suggested the higher concentration in the heated milk contributes to the richer taste since it is above its flavor threshold (12 ppb) in raw milk.

Since the strong cooked flavor is objectionable in UHT milk, researchers are working on methods to reduce this flavor. Ferretti et al. (20) found the addition of

2-acetamidoethyl 2-acetamidomethane-thiolsulfonate (AETS) after sterilization greatly reduced the detectable cooked flavor. However, the compound became less effective with time.

Researchers at North Carolina State University, have been experimenting with the enzyme, sulfhydryl oxidase in hopes of reducing the initial cooked flavor in UHT milk (46,47). Sulfhydryl oxidase is an enzyme that has been isolated and purified from whey and partially characterized. Sulfhydryl oxidase uses oxygen as an electron acceptor to catalyze oxidation of thiols, producing disulfides and hydrogen peroxide. The exposed sulfhydryls in heat denatured milk proteins are excellent substrates for this reaction. Succinamidopropyl porous glass beads were used as supports for immobilized sulfhydryl oxidase. Immobilized sulfhydryl oxidase can then catalyze the oxidation of sulfhydryls and therefore eliminate the cooked flavor (47). Swaisgood et al. (47) predict that the ability of this sulfhydryl oxidase to eliminate the cooked flavor depends on the activity and stability of the enzyme during continuous processing of milk.

Antioxidants. For an antioxidant to be effective in retarding lipid oxidation it should reside either in the fat or at the fat globule surface (14,16). Many antioxidants have a preference for the oil phase (15), but protein binding of some antioxidants occurs (16), resulting in a portion of the antioxidant being held in the aqueous phase.

Cornell et al. (15) used a butteroil/water system to determine the partition cofficient of an antioxidant, which is the ratio of its concentration in the oil to water phases. Butylated hydroxyanisole (BHA) had the highest partition coefficient, 843; therefore it possessed a strong preference for the oil phase. The coefficients of the gallic acid esters increased with increasing chain length of the alcohol substituent with ethyl gallate and hexyl gallate being 0.24 and 71.0, respectively. Similarly, studies by Cornell et al. (16) showed that binding of the gallic acid esters to milk proteins increased with increasing alkyl chain length. BHA was found to have the same binding coefficient as propyl gallate. When an antioxidant with a high binding coefficient is present in milk, the proteins compete with the fat for the antioxidant.

King (31) found that an antioxidant is only effective in an emulsified form. Ethanol and hexane solutions of delta-alpha-tocopherol at concentrations of 400 ug/g milk fat were ineffective at reducing oxidation whereas emulsified tocopherol at concentrations as little as 25 ug/g milk fat provided almost complete protection from oxidation.

According to Sidhu et al. (45), an antioxidant should limit lipid oxidation in milk for at least 14 d, should not impart its own off-flavor, and should be acceptable as a food additive. In this study, antioxidants were added in an emulsified form at a rate of 10-15 mg/l of milk. Sesamol, BHA, BHA + propyl gallate, BHA + gamma-tocopherol, nordihydroguaiaretic acid (NDGA), and ethoxyquin were all found to be effective in controlling oxidized flavors for 14 d and reducing oxygen uptake to approximately the same concentrations observed in conventional milk after 10 d storage. Propyl gallate and Tenox 2 were only partially effective with Tenox 2 having slightly lower oxidized flavor scores.

Zadow et al. (55) studied the effect of antioxidants in UHT milk (150°C for more than 6 s) by adding a combination of 10 mg BHA, 10 mg tocopherol, and 50 mg Tween 80 per kg of milk. The milk was stored at either 2 or 20°C for 3 mo in cans. It was found that 3 out of 4 graders preferred the milk with antioxidants, while one grader objected to a metallic taste imparted by the antioxidants.

Nonenzymatic browning

Early Maillard reaction products, 1-amino-1-deoxy-2-ketose do not cause browning but are important because they are precursors of volatile flavor compounds in the early stages and melanoidins in the advanced stages of the Maillard reactions. Maillard browning is dependent on the time and temperature of heating, moisture, pH, and type of sugar. Of all these variables, temperature is probably the most important (38).

Zadow (52) studied the extent of browning in raw skim milk processed by direct and indirect methods from 97-145°C for 3 s and stored at 2°C with and without a 15-20 ml headspace in sterile bottles. Ferricyanide reducing (FR) and hydroxymethyl furfural (HMF) values were used to determine the extent of browning. In all cases, FR and HMF values increased with an increase in processing temperature. A sharp decline in FR and HMF values occurred during storage, with major changes complete after 40 h. The headspace oxygen in the bottles did not influence the rate of decrease in FR and HMF values during initial storage.

Thomas et al. (50) found FR values were dependent on oxygen concentration with a faster decrease apparent in samples with more dissolved oxygen. Ferricyanide reducing values decreased the first 12 d and after 20 d began to increase in samples with initially high (8.9 ppm) and medium (3.6 ppm) oxygen concentrations. However, they continued to decline in the samples with low initial oxygen concentrations up to 90 d storage.

Jeon et al. (29) found that an increase in storage temperature resulted in more browning in the milk. Very little browning occurred in milk stored at 3 and 22°C, whereas milk stored at 35°C developed more brown color with time.

Swartzel et al. (48) measured absorbance at 470 and 720 nm and related this to browning. They found samples receiving a more severe heat treatment had a greater initial absorbance. The absorbance decreased initially in all samples and then increased the remainder of a 60 wk test period. Samples stored at 40°C increased in absorbance at a faster rate once the increase began than did samples stored at 4°C. Results from this study, with respect to storage and processing temperatures, appear to be similar to those found by measuring FR and HMF.

Lipolysis

Milk lipase can hydrolyze triglycerides of milk fat to produce a rancid flavor. Since lipases that occur in most raw milk are usually heat labile (1), certain heat stable lipases may possibly alter the flavor of stored UHT milk. Some heat stable lipases are more resistant to heat than most microorganisms; therefore, these enzymes may not be fully inactivated at the UHT processing conditions designed to kill viable cells (1,28). Adams and Brawley (1) found that a time-temperature combination of 27 min at 100°C or 63 s at 150°C was required to inactivate a heat resistant lipase of Pseudomonas spp. MC50.

In a study by Andersson et al. (5), heat stable lipase from Pseudomonas fluorescens SIK W1 was added to pasteurized whole milk at concentrations of 188 and 564 units/l of milk. These milks were subsequently sterilized at 138°C ±2°C for 3 s and stored in 3 l Erlenmeyer flasks at 8°C for 22 d. After sterilization, lipase had lost 50% of its initial activity for both samples, but retained 40% of its activity after 22 d storage. Although lipase activity was relatively high, the milk remained sterile throughout the storage period. A rancid flavor was detected after 5-8 d in the high and 12-14 d in the low initial-lipase samples. The reference sample, containing inactivatied lipase, developed a rancid flavor after 22 d. Acid degree values (ADV) correlated with increases in rancid flavor development.

Earley and Hansen (18) UHT processed milk by steam injection at 138°C for 20.4 s and 149°C for 3.4 s. They found increases in ADV values in samples stored at both 24 and 40°C; however, no rancid flavor was detected. They concluded that the ADV determination method of Thomas et al. (49) is not an accurate measurement of hydrolytic rancidity in UHT milk (18).

Thomas et al. (50) found no increase in ADV values of samples processed either indirectly at 141°C for 3.6 s or directly at 143°C for 3 s and stored for 150 d at 20°C. Earley and Hansen (18) felt that Thomas et al. (50) either had a very low initial lipase in the raw milk or a more efficient inactivation of the lipase during UHT sterilization.

In an 8 mo storage study on concentrated milks (UHT, high-temperature short time, and conventionally sterilized) Loney et al. (34) found very little change in ADV values of samples stored at 4°C with a slight increase in samples stored at 37°C. They concluded that the heat process had little effect on the ADV value and no reference was made to any rancid flavor..

Staling

After processing, UHT milk has a strong cooked flavor which decreases with time. As the cooked flavor dissipates, a stale flavor becomes apparent (10,29,34,36,50). A possible masking effect by the cooked flavor on the stale flavor (or vica versa) may be occurring (10,36). The rate of the stale flavor development increases with an increase in storage time and temperature (34). Flavor scores of milk decrease with an increase in staling (10,29,34,36,50). Several researchers have associated an increase in carbonyl compounds with an increase in the stale flavor (10,29,32,36). Mehta and Bassette (36) reported the increase in stale intensity paralleled an increase in n-propanal, n-pentantal, n-hexanal, and an unidentified peak, whereas methyl sulfide and a 13.8 min GC peak decreased as the stale intensity increased. Conceivably methyl sulfide and the 13.8 min peak may be precursors of the stale flavor compounds. Milks packaged in polyethylene (PE) cartons had a greater increase in carbonyl compounds and stale intensity while exhibiting a rapid decline in methyl sulfide. The PE cartons were permeable to oxygen, whereas samples packaged in aluminum foil-lined cartons were much less permeable to oxygen and retained a desirable flavor longer. Thomas et al. (50) stated that stale and oxidized flavor development was independent of initial oxygen. They found flavor scores of sterile milk were influenced by increases in concentration of carbonyl compounds. Jeon et al. (29) states as the concentration of carbonyl compounds increased, the stale intensity increased and flavor scores decreased. Odd-numbered methyl ketones (C_{3-13}) are the most abundant compounds, but n-aldehydes ($C_{3,5-9}$) contributed most to the off flavor.

Bassette and Jeon (10) detected an increase in acetaldehyde, n-pentanal, and n-hexanal in samples stored at 25°C which was closely related to a decrease in flavor acceptability and an increase in stale intensity. In contrast, Earley and Hansen (18) found a decrease in short chain carbonyl compounds with storage at 24 and 40°C. The samples stored at 40°C were unpalatable after 6-8 wk.

Kirk et al. (32) studied the components in high-temperature short-time (HTST) fluid sterilized milk during storage at 4.4, 22, and 36°C. Milk was sterilized at 140.5 ±1°C for approximately 4 s. A stale flavor developed after 2 mo in samples stored at 22°C and 1 mo in those stored at 36°C. The rate of staling was a function of time and temperature. Compounds found in the sterilized milks were: ethanal, propanal, furfural, acetone, 2-butanone, ethanol, butanol, butanal, hexanal, heptanal, 2-pentanone, and 2-heptanone. Only the first seven components were found in fresh samples. From the gas liquid chromatography (GLC), it was apparent that the less volatile compounds had disappeared from the stale milk and the more volatile compounds had increased. Arnold et al. (7) did not observe any significant increase in the more volatile compounds of sterilized concentrated milk (SCM). This supports Kurtz (33) idea that the components contributing to the stale flavor either have a low volatility or are tightly bound by milk powder.

Arnold et al. (7) found that the longer chain methyl ketones, delta-deca-lactone, benzaldehyde, and o-aminoacetophenone were likely to be significant in the stale flavor. When a concentration of 5 ppb o-aminoacetophenone was added into fresh SCM, a stale flavor was detectable. Therefore, it is probably an important compound in the stale defect.

Ferretti and Flanagen (21) identified 44 compounds in 2 yr old stale skim milk powder; however, o-aminoacetophenone was not one of them. Flavor thresholds and the approximate concentrations were determined for each component, trying to establish the role of volatiles resulting from Maillard browning in the development of stale flavor. No single compound was associated with the typical stale flavor; however, 12 compounds appeared to be important. They were: 2-furaldehyde, 2-furfuryl butyrate, methylethylpyrazine, 2,3,5-trimethylpyrazine, N-ethyl-2-formylpyrrole, o-cresol, benzaldehyde, salicylaldehyde, cumene, naphthalene, benzothiazole, and 2-nonanone. A synthetic mixture made of all these compounds in their estimated concentration in fresh skim milk did not give a stale flavor. However, when 2.5 ppb o-aminoacetophenone was added to this mixture, 3 out of 6 expert judges felt it exhibited stale flavor similar to the original reconstituted milk powder and more stale than 2.5 ppb o-aminoacetophenone alone.

MATERIALS AND METHODS

SAMPLE PREPARATION

Source of milk

Ten gallons of raw skim and pasteurized-homogenized whole milks were obtained on subsequent weeks from the Kansas State University dairy plant and subjected to UHT processing. Both milks were subjected to the same treatments as outlined in the following section entitled SCHEMATIC OF TREATMENTS.

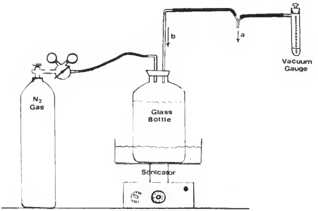
Addition of antioxidant

Tenox 2 (Eastman Kodak, Kingsport, Tenn.), an antioxidant mixture of 20% butylated hydroxyanisole (BHA), 6% propyl gallate, 4% citrate and 70% propylene glycol was introduced into the appropriate milks before processing. A concentrated antioxidant solution in water was added to the milk to give a final concentration of 400 ppm BHA, 120 ppm propyl gallate, and 40 ppm citrate on the fat basis in the milk.

Deoxygenation

A combination of nitrogen sweeping, sonication, and vacuum treatment was found to be effective in reducing oxygen levels in milk to less than 2 ppm (Fig.1). The system used involved placing a large 2.5 gal glass bottle into a 30°C sonicating water bath and flushing it with nitrogen gas (while sonicating) to displace the oxygen present in the bottle. The milk was warmed to 30°C before being put into the bottle. A vacuum of 20 mm Hg was pulled above the milk for 30 min with simultaneous sonication. This procedure resulted in extreme foaming of the milk initially; therefore it required careful attention until bubbles collapsed and the foaming action ceased. Nitrogen then was used to bring the system back to atmospheric pressure and to put a slight positive pressure on the system. A hose was connected from the sonicating flask to Tank C of the UHT system. The positive pressure in the deoxygenation flask pushed the milk through the hose into the tank without exposing the milk to atmospheric oxygen.

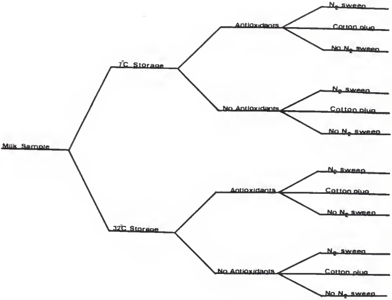
Fig. 1. Deoxygenation system. One arrow (a) illustrates a connection to the vacuum or the receiving tank depending on whether the milk was being deoxygenated or transferred to Tank C, respectively. A second arrow (b) indicates that the tube was placed in the down position when transferring milk to Tank C.



SCHEMATIC OF TREATMENTS

Prior to processing, milks were treated as shown in Fig. 2. The UHT milks were stored at two temperatures: 7°C and 32°C simulating refrigeration and an accelerated shelf storage, respectively. Within each storage temperature, milks were processed with and without antioxidant, and with one of three treatments designed to control oxygen concentrations. One sample set was nitrogen swept to reduce the dissolve oxygen and the bottles were capped with teflon-lined caps; a second set was not nitrogen swept and the bottles were capped with teflon-lined caps; and a third set received no nitrogen sweep and the bottles were plugged with sterile cotton plugs. The cotton plug allowed a relatively high concentration of dissolved oxygen in the milk throughout the storage period.

Fig. 2. Schematic diagram of treatments of whole and skim milks.



UHT TREATMENT

Design of a pilot-size UHT system

Milk was UHT treated in a laboratory scale indirect UHT system designed and constructed at Kansas State University (Fig. 3 and 4). The system was fabricated using 6.35 mm stainless steel tubing as a barrier between the heating agent and the milk. Nitrogen gas from tank A under 80 psi pressure propelled milk,

Fig. 3. Schematic of the indirect UHT system used in this study.

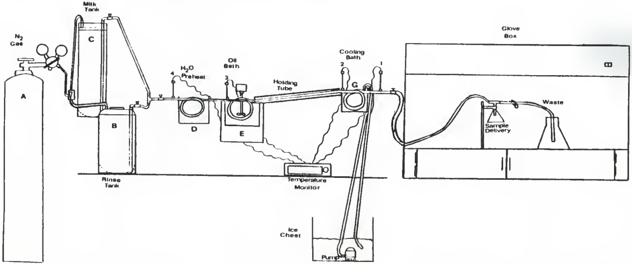


Fig. 4. Indirect UHT system used in this study.

rinse solution or cleaning solution through the system. Tank B, a 3 gal Millipore tank, was filled with water for rinsing the system and tank C, a Pepsi-Cola syrup container, held the preprocessed milk. Both tanks B and C had stainless steel tubes which extended to the bottom of the tanks, allowing the product to be forced out under pressure. The preprocessed milk was warmed to 30°C before being placed into tank C. Temperature probe #4 monitored the temperature of the preprocessed milk before it was circulated through the 6.35 mm stainless steel tubing immersed in a hot water bath (D) where it was preheated to approximately 82°C as monitored by temperature probe #3. The milk was then brought to sterilization temperature of 135°C in an oil bath (E) and held at this temperature for 5 s as it passed through an insulated holding tube (F). Temperature probe #2 monitored the sterilization temperature. After leaving the holding tube at 135°C, the milk was rapidly cooled to 11°C in a circulating ice-water bath (G). Water in the cooling bath was fed from an ice chest filled with ice and water by a small pump which circulated cold water into the bath while a hose syphoned the water back into the chest. A needle valve positioned after the cooling bath, was used to regulate the flow rate of the milk through the system. The sterilized milk was directed through tygon tubing into a glove box where it was either diverted to a waste flask or aseptically collected in amber glass bottles.

Cleaning procedure of the UHT system

Immediately after a processing run, the system was rinsed with water and then swept with 2.5 gal of a strong alkali solution (Klenzade - Kleer Brite formula HC-29 over 10% NaOH). Following the alkali treatment, the system was rinsed with water and then flushed with 2 gal of an acidic cleaning solution (Klenzade AC-3 Acid Detergent Cleaner). The system was rinsed with water again and then sanitized with a 100 ppm chlorine sanitizer (XY-12 Liquid Sanitizer). A final water rinse flushed chlorine from the system.

Sterilization procedure of the UHT system

Before the milk was processed, the system was sterilized. The tubing was disconnected immediately after the needle valve and all open ends were wrapped with aluminum foil. These parts of the system, sample bottles, and waste flasks were sterilized at 121°C for 25 min. The hot water bath and the oil bath were brought up to temperature and the system was flushed with a 100 ppm chlorine sanitizer (XY-12 Liquid Sanitizer, Klenzade), rinsed with clean water, and steam sterilized at 140°C for 15 min. The glove box was sanitized with 500 ppm XY-12 Liquid Sanitizer (Klenzade) and exposed to UV light for 1 h prior to processing to kill any surface organisms. The previously disconnected (autoclaved) tygon tubing and connector of the second section of the system was reconnected over a flame.

ANALYSES OF PREPROCESSED AND/OR UHT-PROCESSED MILK

Samples from the preprocessed milk (0 wk), the day after processing (Initial, 0.1 wk), and weeks 1, 2, 4, 8, 12, 16 were subjected to the following analyses.

Microbial

Total plate counts were determined for the preprocessed skim and pasteurized-homogenized whole milks as outlined in Standard Methods for the Examination of Dairy Products (4). The plates were incubated at 37°C for 48 h and the number of colonies counted.

Total aerobic and anaerobic counts were determined for the milk the day after processing (0.1 wk) and weeks 4 and 16. All milk samples except the preprocessed milks were incubated 1 wk at 32°C prior to plating to create sufficient growth so that any viable cells or spores present in the milk could be detected. Samples were plated in duplicate with dilutions of 10^0 and 10^{-1} for total aerobic counts on Plate Count Agar (Difco). Duplicate 10^0 dilutions were plated on Anaerobic Agar (Difco) for total anaerobic count. All plates were incubated at

37°C for 48 h and inspected for growth.

Proximate analysis

Proximate analysis test for fat, protein, total solids, and ash content were performed on the preprocessed milks.

Fat content. The fat content in the milk was determined in duplicate by the procedure in the Mojonnier Milk Tester, Bulletin 101 (6) on a Model D Mojonnier Milk Tester.

Protein content. The percent protein in the milk was determined in triplicate by the Kjeldahl procedure as outlined in AOAC (9).

Ash content. The ash content of the milks were determined in duplicate by the AOAC method (9).

Total solids. A modified AOAC method (9) was used to determine total solids. Duplicate 2 ml samples of milk were weighed into weighed disposable aluminum dishes. The samples were dried in a forced-air oven at 100°C for 4 h, cooled in a desiccator, and reweighed. The weight of the residual material was expressed as total solids.

Antioxidant

BHA extraction procedure. A modification of the steam distillation procedure of Bassette and Ward (11) was used to determine the concentration of BHA in milk. Fifty milliliters of milk and one drop of GE Antifoam 66 (100% active silicone defoamer) were placed in a digestion-distillation flask and steam distilled with a Kemmerer-Hallet type micro-Kjeldahl nitrogen distillation apparatus. Twenty-five milliliters of distillate was collected in a marked tube which was placed in an ice-water bath. Approximately 10 min were required for the distillation. The system was cleaned before and after each run by refluxing 5 min with boiled distilled water. One milliliter of carbon disulfide (Fisher Scientific) was added to the distillate from skim milk (2 ml to whole milk distillate) to extract

BHA. The mixture was shaken vigorously 100 times and allowed to stand at room temperature until the BHA containing carbon disulfide (CS_2) layer separated from the aqueous layer. The CS_2 layer was drawn off with a 9" Pasteur pipette, placed in a small stoppered vial, and refrigerated until time of analysis.

GLC analysis of antioxidant. Antioxidant levels were measured on a Hewlett-Packard Model 5880A Gas Chromatograph with a flame-ionization detector. A 1 ul sample was injected onto a 2' column of 0.2% OV-101 on 100/120 Chromosorb W/HP support. The GC signal was monitored by a Hewlett-Packard Level 4 data system. The flow rate of the carrier gas (nitrogen) was 25.1 ml/min, hydrogen was 29.5 ml/min, and air was 30 ml/min. The temperature program was as follows:

Oven temperature = 105°C
Injector temperature = 180°C
Detector temperature = 200°C
Oven temperature profile:
  Initial value = 105°C
  Initial time = 1.5 m= 1.5 min
  Program rate = 3°C/min
  Final value = 118°C
  Final time = 2 min
Post value = 170°C
Post time = 2 min

Antioxidant standard curves. Due to the difference in the fat contents of whole and skim milk, it was necessary to make separate standard curves for analyzing these two milks. Samples containing 0, 50, 100, 200, and 400 ppm BHA (fat basis) were analyzed by GLC. The peak areas were recorded from duplicate 1 ul injections of each standard and a curve drawn (Fig. 5 and 6). Regression equations were calculated and shown in Table 2.

Dissolved oxygen

The dissolved oxygen concentration of each sample was measured by an oxygen-permeable membrane electrode with a YSI (Yellow Springs Instruments, Yellow Springs, Ohio) monitor Model 53 at 25°C. Four milliliters of milk was placed into the sample chamber and stirred at a constant rate by a magnetic stir bar. A chart recorder (Linear 1200) was used to measure oxygen levels (nmolar) and these levels were converted to ppm dissolved oxygen.

Volatile materials

Steam distillation. The method of Bassette and Ward (11) was employed to determine volatile materials. Fifty milliliters of milk and one drop of GE Antifoam 66 were steam distilled in a distillation flask of a Kemmerer-Hallet type micro-Kjeldahl distillation unit. A 15 ml graduated conical test tube with 0.5 ml of boiled distilled water in the bottom was placed in an ice-water bath for collection of distillate from the condenser outlet. The distillation rate was about 5 ml of distillate in 5-6 min. After 5 ±0.1 ml of distillate was collected, the collection tube was quickly stoppered. Duplicate 2 ml aliquots were transferred into 5 ml serum vials, each vial containing 1.2 g of anhydrous sodium sulfate. The vials were stoppered and refrigerated until GLC analysis. The distillation apparatus was cleaned by connecting a flask which contained distilled water and allowing the system to sweep for about 5 min.

GLC analysis of volatile materials. The changes in volatile material were analyzed on an Aerograph Model 550-B gas chromatograph equipped with a flame-ionization detector and a 1 mv Brown-Honeywell recorder. Head-space gas was drawn off the prepared sample and injected onto a 304.8 x 0.318 cm stainless steel column packed with 20% Carbowax 20M on 80/100 mesh Chromosorb P solid support. The operating conditions were as follows:

Oven temperature = 100°C
Injector temperature = 164°C
Nitrogen flow rate = 13.4 ml/min
Hydrogen flow rate = 32.5 ml/min
Oxygen flow rate = 83.3 ml/min

The samples were prepared by placing a vial containing the sample in a 60°C water bath for 2 min and then on a mechanical shaker (Eberbach) for 5 min. After shaking, the serum cap was changed and the vial placed back into the 60°C water bath for 8 min. A 1 ml gas-tight syringe (Hamilton #1001) was inserted through the rubber serum cap and head-space gas was slowly drawn into and quickly expelled from the syringe five times; then, 1 ml of head-space gas was drawn into the syringe and injected into the chromatograph.

GLC standard curves. Standard curves were prepared by analyzing milk samples containing known concentrations of acetaldehyde, propanal, n-pentanal, and n-hexanal. Peak heights were determined from milk samples with nothing added and these peak heights were subtracted from the standard samples. A standard curve was drawn using the adjusted values for each compound (Fig.16, 17, 18, and 19) and a regression equation was determined (Table 14).

Thiobarbituric acid (TBA)

Lipid oxidation was measured by the method of King (30). In the protocol, 17.6 ml of milk was pipetted into a stoppered flask and warmed to 30°C. One milliliter of trichloracetic acid solution (100% w/v) and 2 ml of 95% ethanol were added to the milk. The flask was stoppered, shaken, allowed to set about 5 min, and filtered through No. 42 Whatman filter paper. One milliliter of 2-thiobarbituric acid solution (1.4 g TBA in 95% EtOH to make 100 ml) was added to 4 ml of filtrate. The solution was stoppered, mixed, and incubated in a 60°C water bath for 1 hr. After cooling the solution to room temperature, the optical density of the sample was measured on a Beckman DU Spectrophotometer at 532 nm using water as a blank.

Acid Degree Value (ADV)

The method of Thomas et al. (49) was used to determine rancidity in whole milk. Thirty-five milliliters of milk was placed in an 18 g 8% milk test bottle and 10 ml of BDI reagent (30 g Triton X-100 - a nonionic surface-active agent manufactured by the Rohm and Hass Co., Philadelphia, PA. and 70 g tetraphosphate made up to 1 l with distilled water) was added and thoroughly mixed. The bottle was boiled for 15 min, agitating after 5 min and 10 min. The sample was centrifuged for 1 min and aqueous methyl alcohol was added to bring the top of the fat column to the 6% graduation. The sample was again centrifuged for 1 min and then tempered for 5 min in a 130-140°F water bath taking note that the water bath level was above the top of the fat column.

After tempering, 1 ml of fat was transferred into a 50 ml Erlenmeyer flask using a 1 ml syringe. The fat was dissolved in 10 ml petroleum ether and 5 ml of absolute ethanol. Also, 10 drops of phenolphthalein indicator was added. The fat solvent was titrated to the first color change with standardized alcoholic KOH using a 10 ml microburette.

Flavor analysis

All samples were evaluated organoleptically by a 4-membered experienced taste panel at 0, 0.1, 1, 2, 4, 8, 12, and 16 wk of storage. A stale reference sample was available at each session for panelist to refresh their memory of the stale flavor. All samples were warmed to 24°C prior to taste evaluation. Each panelist was asked to evaluate the samples for any cooked, stale, and/or oxidized flavor on a scale of 1 (none) to 4 (pronounced). Also, panelists were asked to assign an overall flavor score to each sample using a hedonic scale ranging from 1 (dislike very much) to 7 (like very much) and encouraged to add any comments.

Statistical analysis

The experimental design for the analysis of variance (ANOVA) was a completely randomized design with a 3-way cross classification treatment structure. The model used is as follows:

$$Y_{ijk} = \mu + T_i + A_j + S_k + (TA)_{ij} + (TS)_{ik} + (AS)_{jk} + E_{ijk}$$

Y_{ijk} = response (eg., stale-flavor intensity, concentration of n-pentanal)
μ = mean
T_i = temperature effect
A_j = antioxidant effect
S_k = deoxygenation effect
$(TA)_{ij}$ = interaction between temperature and antioxidant
$(AS)_{jk}$ = interaction between antioxidant and deoxygenation
$(TS)_{ik}$ = interaction between temperature and deoxygenation
E_{ijk} = experimental error

Three-way interaction was used as an estimator of error since there was only one replication of the treatments. Furthermore, SAS GLM was used for analysis of the data because of missing data. All data from each test period and both milk types were analyzed separately using the above model.

RESULTS AND DISCUSSION

ANALYSES OF PREPROCESSED AND/OR UHT-PROCESSED MILKS

Microbial

Before processing, samples of raw skim and pasteurized-homogenized whole milks were evaluated for total plate counts. From duplicate tests, skim milk had an average count of 7.8×10^5 colony forming units (CFU)/ml and whole milk had an average count of 4.6×10^4CFU/ml. According to Zadow (53), there is no legal limit on microbial population of raw milk to be used for UHT milk, but it should be of high quality and at least meet standards required for pasteurized milk.

Both experimental UHT whole and skim milks maintained sterility over the four month study. Plate counts for total aerobic and anaerobic counts were virtually zero except for a few containing 1 CFU/ml. One CFU/ml was not considered significant and was probably due to chance contamination during the plating process. Furthermore, no visual contamination of the milk was observed. Spoilage occurred in a few cotton plugged samples stored at 32°C; therefore, they were excluded from further analysis. It is likely that contamination of these samples occurred during the filling process or upon storage in the 32°C incubator.

Proximate analysis

Preprocessed samples of skim and pasteurized-homogenized whole milks were subjected to proximate analysis. Results of these analyses are found in Table 1. All values obtained were within the normal expected range of the various components (whole milkfat 3.2-3.6%, skim milkfat 0-0.5%, protein 3-4%, ash ~0.7%, whole milk total solids 11.5-14.5%, and skim milk total solids 9-10%) (19).

Table 1. Proximate analysis of raw skim and pasteurized-homogenized whole milk.

Parameter measured	Skim	Whole
% Fat[a]	0.12	3.20
% Protein[b]	3.55	3.36
% Ash[a]	0.70	0.72
% Total solids[a]	9.27	12.00

[a] Average from analysis in duplicate

[b] Average from analysis in triplicate

Statistical analysis

All significant differences relating the following components or test of the UHT milks were determined at the 5% level between least square means (LSM) of treatments. Also, all tables (except 1, 2, and 14) referred to in the ANALYSES OF PREPROCESSED AND/OR UHT-PROCESSED MILKS and GLC ANALYSIS OF VOLATILE MATERIALS are found in Appendix A.

We were unable to determine statistical differences between samples at 0 and 0.1 wk because of the way samples were treated before and the day following processing. For example, all samples were refrigerated one day until they were placed in 7°C and 32°C storage. Even though these milks had been treated differently (antioxidant and dissolved oxygen) after one day of refrigeration, samples were essentially the same. The computer verified this and calculated no significant differences.

Antioxidants

Standard curves were prepared from analyses of the antioxidant BHA. Figures 5 and 6 illustrate the linear relationships that exists between peak areas and the concentrations of added BHA in both skim and whole milk. Regression

Fig. 5. Standard curve of BHA in skim milk on a 0.1 % fat basis.

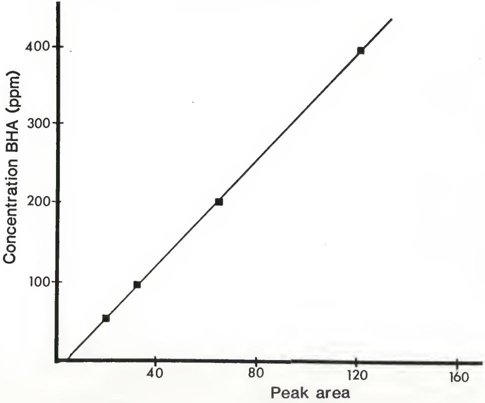
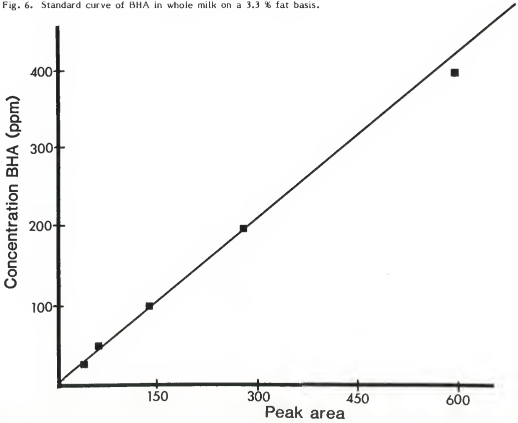


Fig. 6. Standard curve of BHA in whole milk on a 3.3 % fat basis.



equations were calculated from these curves and are presented in Table 2.

Tables 3 and 4 (Appendix A) show that within each treatment (at a specific week), samples with added antioxidant had approximately the concentration of BHA initially added and samples without added antioxidant were devoid of BHA. Although a statistical analysis was not performed from week to week, antioxidant concentration did not appear to change.

Table 2. Regression equations from standard curves of BHA in ppm.

Compounds	Regressions equations[a,b]
BHA in skim milk	$Y = -10.8164 + 3.3389\ X$
BHA in whole milk	$Y = 5.7757 + 0.6730\ X$

[a] Y = concentration in ppm.

[b] X = peak area at retention time of 4.32 min.

Dissolved oxygen

As shown in Tables 5 and 6 and Fig. 7 and 8, milk in bottles plugged with sterile cotton plugs had significantly higher concentrations of dissolved oxygen at both storage temperatures than did the capped bottles. The preprocessed skim milk had a very low (~0.25 ppm) dissolved oxygen content, possibly as a result of a high microbial load that consumed available oxygen prior to processing. However, Figs. 7 and 8 show that the concentration of oxygen increased in these samples during processing (~2 ppm). Also, as expected the cotton plugged samples increased even more during storage. The dissolved oxygen content decreased slightly at weeks 1 and 2 for whole and skim milks, respectively. This decrease may have occurred from oxidation of sulfhydryl groups formed during processing (50,54). By the 4th wk, dissolved oxygen concentrations had increased in skim milk and remained

Fig. 7. Dissolved oxygen concentration (ppm) vs. storage time in skim milk.

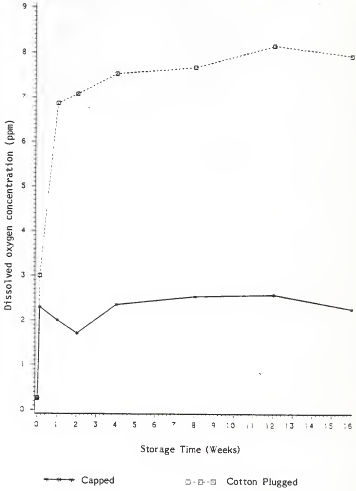
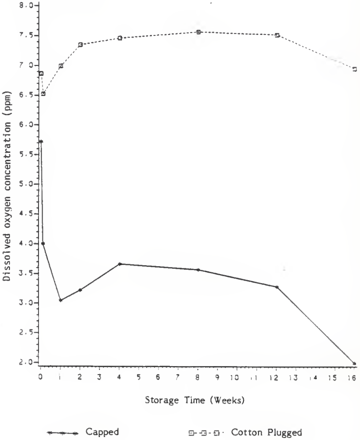


Fig. 8. Dissolved oxygen concentration (ppm) vs. storage time in whole milk.



relatively constant throughout remaining storage period. In whole milk, a decrease occurred upon extended storage and may have resulted from the involvement of oxygen in lipid oxidation. From Figs. 8 and 10, TBA values (a measure of lipid oxidation) increased in whole milk as the dissolved oxygen contents decreased. However, Allen and Joseph (3) found that only 7.1% (0.29 ± 0.02 mg/l) of the total oxygen consumed was involved in lipid oxidation; therefore, it would seem unlikely that the large decreases in dissolved oxygen were solely as a result of lipid oxidation.

Lipid oxidation by TBA test

Table 7 shows that within each treatment combination (at a specific week), LSM of the TBA values were not significantly different in the skim milk. Although it is not possible to determine significant difference for 0.1 wk (day after processing), Fig. 9 of skim milk shows a large increase in TBA values at both storage temperatures and then a decrease after a week. Cotton plugged samples without antioxidant (N CP) were found to have higher LSM of TBA values than the others at 0.1 wk (Table 7), therefore, causing the large increase seen in Fig. 9. This indicates that lipid oxidation occurred, and the formation of oxidation products (described later) reinforces this hypothesis. The subsequent decrease in TBA values may indicate that malonaldehyde (responsible for TBA color) may be a precursor in other reactions.

Table 8 shows little change in LSM of TBA values in whole milk until the 2nd wk where the LSM values in cotton plugged samples were significantly higher than the others. At the 16th wk, storage temperature, antioxidant concentration, and dissolved oxygen concentration influenced TBA values. Samples stored at 32°C, samples without antioxidant, and samples with higher dissolved oxygen concentrations (cotton plugged and no deoxygenation) had significantly higher TBA values than the other samples. Although no attempt was made to determine significant difference from week to week, Fig. 10 shows a prominent increase in

Fig. 9. Absorbance vs. storage time for TBA values in skim milk.

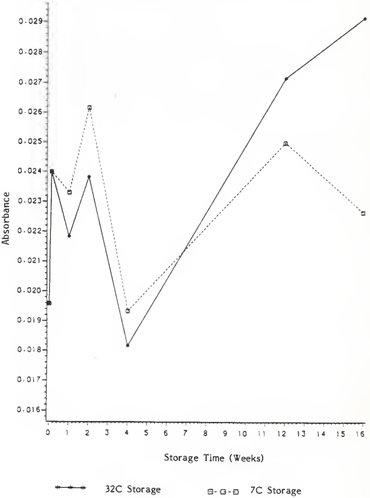
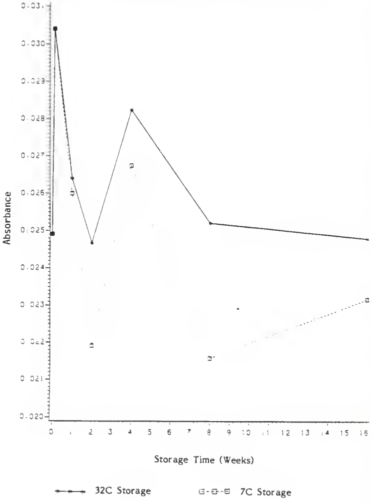


Fig. 10. Absorbance vs. storage time for TBA values in whole milk.



the TBA values in whole milk after the 4th wk with a larger increase in samples stored at 32°C. One might expect this because chemical reaction rates are greater at increased temperatures.

Rancidity by ADV test

Table 9 shows that LSM in whole milk were not significantly different up to and including the 4th wk, but thereafter they were significantly lower in samples stored at 7°C than those at 32°C. Figure 11 illustrates the effect of storage temperature. There was a greater increase in ADV's in samples stored at 32°C. At the 12th wk, the ADVs in samples stored at 7°C decreased while the 32°C samples increased. Although ADV's increased with time, samples were not criticized for having a rancid flavor. Earley and Hansen (18) also found increases in ADV's with no detectable rancid flavor in the milks.

GLC ANALYSIS OF VOLATILE MATERIALS

Acetone

Changes in the amount of acetone in stored skim and whole milks showed similar patterns (see Figs. 12 and 13); however, concentrations were higher in the whole milk. Acetone in samples stored at 32°C and capped gradually increased, whereas, acetone in samples stored at 7°C remained relatively constant with time. Scanlan (42) and Kirk et al. (32) found this ketone to be heat-induced. The capped samples had significantly higher LSM than the cotton plugged samples (Table 10 and 11). Whole milk capped samples stored at 32°C had significantly more than the 7°C capped samples only after the 12th wk and no significant difference was found in the skim milk samples between acetone concentration due to storage temperature. Figures 12 and 13 illustrate a gradual decrease in acetone in all cotton plugged samples. Samples stored at 32°C had significantly less acetone than the samples stored at 7°C after the 12th wk in the whole milk and between 1st-8th

Fig. 11. Acid Degree Values vs. storage time in whole milk.

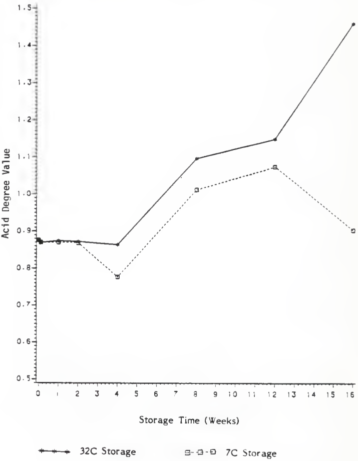


Fig. 12. Peak height vs. storage time for acetone in skim milk.

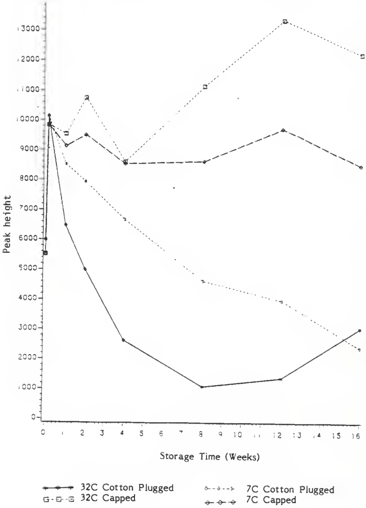
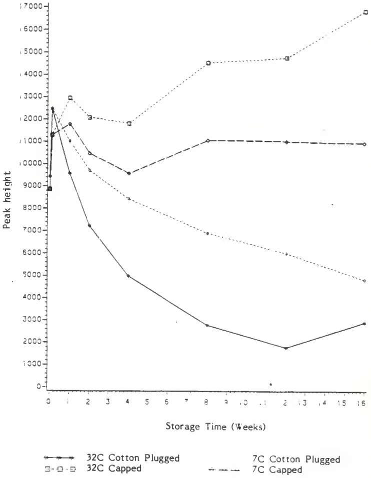


Fig. 13. Peak height vs. storage time for acetone in whole milk.



wk in the skim. The decrease of acetone in the cotton plugged samples at both storage temperatures, may have resulted from losses through the cotton plug or from being consumed in a reaction faster than it was being formed; thus greater losses at the higher storage temperature.

Methyl sulfide

Figures 14 and 15 both show that the concentration of methyl sulfide was very high the day after processing and then decreased with time. It was lost rapidly from the cotton plugged samples with a more gradual loss from the capped samples. The cotton plugged samples had a significantly lower concentration of methyl sulfide than the capped samples after 1 wk storage (Tables 12 and 13). The capped samples maintained a relatively high concentration even after 16 wk storage.

Acetaldehyde, propanal, n-pentanal, and n-hexanal

Standard curves were prepared from analyses of four significant aldehydes (acetaldehyde, propanal, n-pentanal, and n-hexanal) in whole milk. Figures 16-19 show linear relationships between peak height and concentration of these aldehydes. Regression equations were calculated for these curves and are presented in Table 14.

Fig. 14. Peak height vs. storage time for methyl sulfide in skim milk.

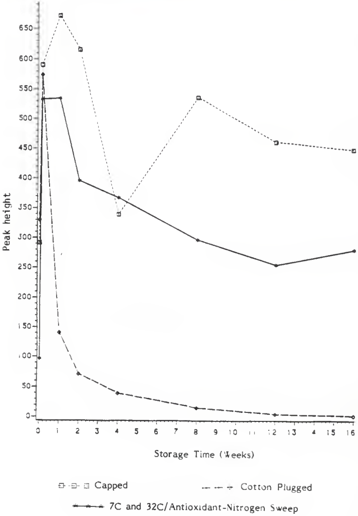


Fig. 15. Peak height vs. storage time for methyl sulfide in whole milk.

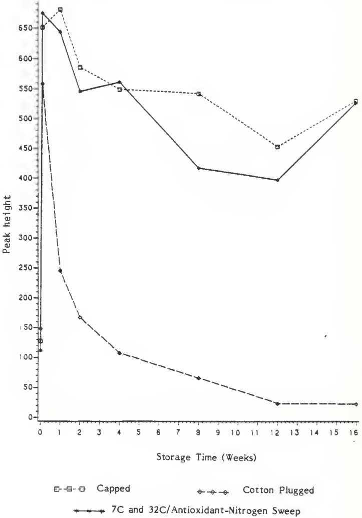


Fig. 16. Standard curve of acetaldehyde in whole milk from steam distillation GLC analysis.

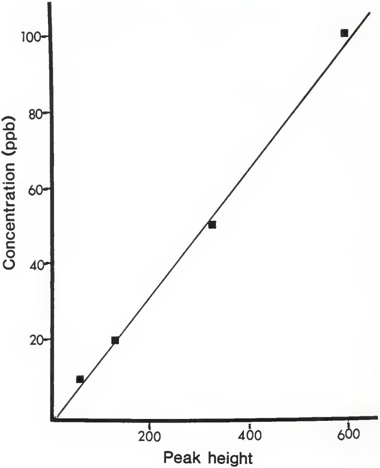


Fig. 17. Standard curve of propanal in whole milk from steam distillation GLC analysis.

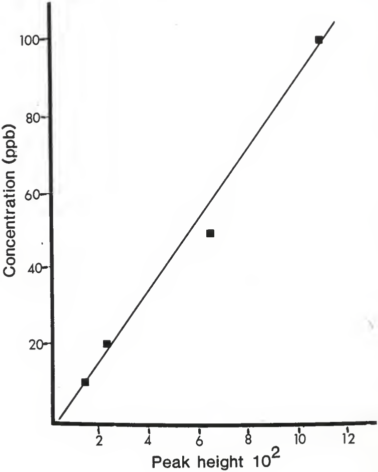


Fig. 18. Standard curve of n-pentanal in whole milk from steam distillation GLC analysis.

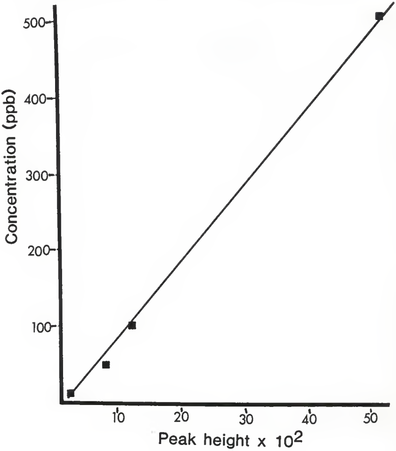


Fig. 19. Standard curve of n-hexanal in whole milk from steam distillation GLC analysis.

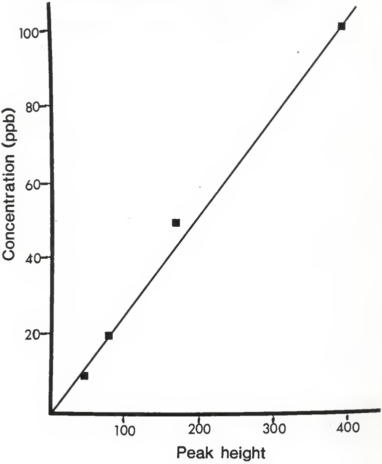


Table 14. Regression equations for standard curves of aldehydes in ppm.

Compounds	Regressions equations[a,b,c]
Acetaldehyde	$Y = 0.00457 + 0.000153\ X$
Propanal	$Y = 0.01008 + 0.000069\ X$
n-Pentanal	$Y = 0.04214 + 0.000087\ X$
n-Hexanal	$Y = 0.00086 + 0.000290\ X$

[a] Y = concentration in ppm.

[b] X = % of recorded full scale deflection (peak height) x attenuation factor in GLC analysis.

[c] Minor daily instrument variations were corrected for by assuming a hypothetical response of 1200 for 1 ppm acetone. Analyses of 1 ppm acetone (external standard) before and after a run gave a correction factor (eg., peak height of component x 1200 / peak height of 1 ppm acetone).

Similar patterns were observed for changes in concentration of acetaldehyde, propanal, n-pentanal, and n-hexanal during storage. In general, less of each compound was formed at 7°C than at 32°C. Also, samples containing antioxidants produced slightly less of these volatile materials at both storage temperatures than did the counterpart samples without antioxidants. As was shown by the acetone, volatile compounds decreased to low concentrations in the cotton plugged samples. Each of these aldehydes will be discussed in detail in the following paragraphs.

Figures 20, 21 and 22 show large increases in the concentrations over the preprocessed sample for acetaldehyde, propanal, and n-pentanal in skim milk at 0.1 wk (day after processing) in samples with high dissolved oxygen contents. The high heat treatment may have contributed to the formation of these reactive compounds, which in turn might be precursors for production of other compounds. Figures 23, 24, and 25 show similar increases in the concentrations of these three

Fig. 20. Concentration vs. storage time for acetaldehyde in skim milk.

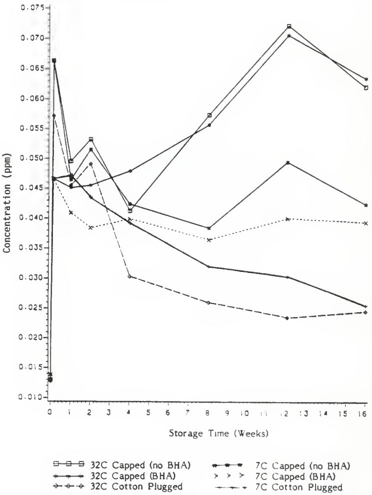


Fig. 21. Concentration vs. storage time for propanal in skim milk.

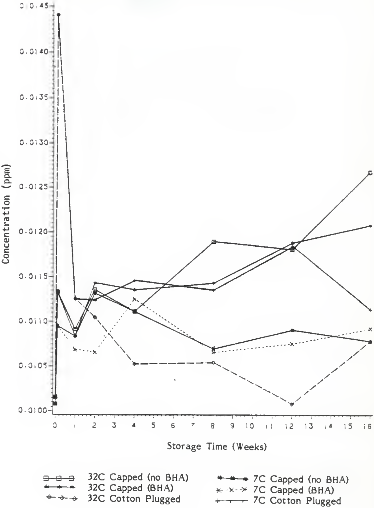


Fig. 22. Concentration vs. storage time for n-pentanal in skim milk.

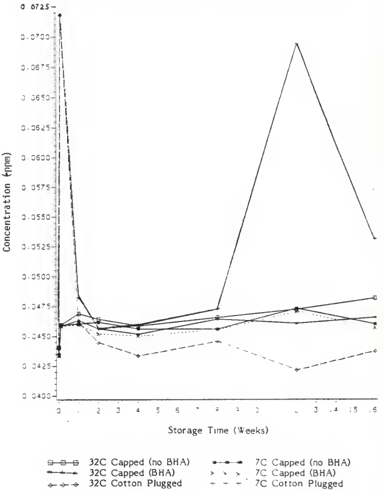


Fig. 23. Concentration vs. storage time for acetalaldehyde in whole milk.

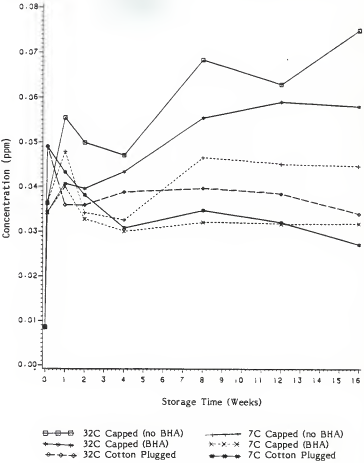


Fig. 24. Concentration vs. storage time for propanal in whole milk.

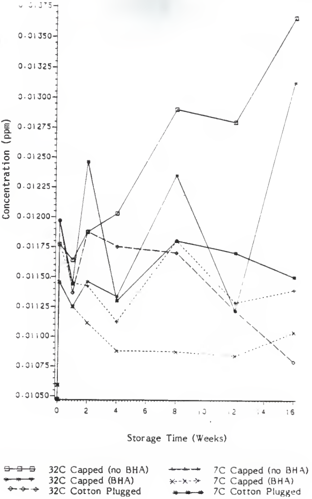
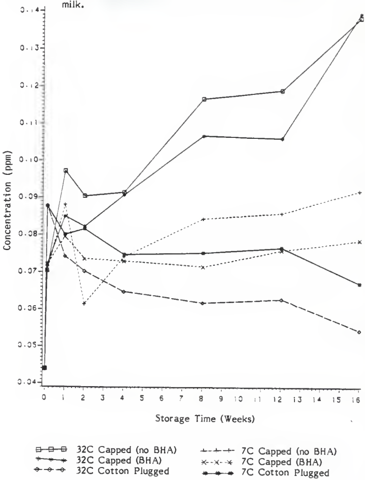


Fig. 25. Concentration vs. storage time for n-pentanal in whole milk.



compounds in whole milk.

Least square means from Tables 15 and 16 show that the concentration of acetaldehyde in whole milk was significantly higher from 4-16 wk at 32°C storage than at 7°C storage. In skim milk it was consistently higher under the same conditions. Also, although not significant, the LSM concentrations of acetaldehyde in samples containing antioxidant were lower than the samples without antioxidant in the whole milk whereas the antioxidant had little effect in skim milk. Since the antioxidant was somewhat effective at reducing the formation of acetaldehyde in whole milk, it is likely that it is an oxidation product. In general, the antioxidant appeared to be somewhat effective at retarding oxidation in this system. The cotton plugged samples had significantly lower concentrations of acetaldehyde, from the 4th-16th wk in the skim and at wks 4, 12, and 16 in the whole milks. This may have been due to escape of the very volatile compounds through the cotton plug.

Least square means of concentrations of propanal (Tables 17 and 18) in the 4-wk whole milk samples stored at 32°C were significantly higher than those stored at 7°C. Also, samples without antioxidants were significantly higher than those with antioxidant. Although not significant, the same trends were apparent throughout the 4th-16th wk of storage period. This also occurred in the skim milk, except at the 4th wk, samples containing antioxidant were significantly higher.

Figure 25 show trends and Table 20 significant differences in the concentration of n-pentanal in whole milk. Samples stored at 32°C had significantly higher concentrations of n-pentanal from the 4th-16th wk than those stored at 7°C. Cotton plugged samples had significantly lower concentrations of n-pentanal than capped samples over the same period, with samples stored at 32°C lower than 7°C. Whole milk samples containing the antioxidant again had lower concentrations of n-pentanal at both storage temperatures. A similar pattern was apparent in the skim milk (Fig. 22--note: the scales of concentrations of the two milks are different); however, the concentrations in skim milk were smaller (Table 19). In skim milk, the 7°C cotton plugged samples had consistantly higher concentration of n-pentanal and n-hexanal than the capped samples throughout the study and an unusually high concentration at the 12th wk.

Similarly, the 7°C skim milk in cotton plugged bottles had the highest concentration of n-hexanal (Table 21 and Fig. 26) throughout storage and it increased to a maximum at the 12th wk. The concentrations were consistantly higher in samples stored at 7°C. Patterns for n-hexanal concentrations in whole milk were different from those in skim (Table 22 and Fig. 27). In the whole milk at 4 and 12 wks, LSM of samples without antioxidant were significantly higher than those containing antioxidant and were consistantly higher throughout the study. The 7°C cotton plugged samples increased in hexanal throughout storage while in the 32°C cotton plugged samples it decreased in the skim milk.

Other peaks

Three interesting peaks of unknown identity occurred at retention times of 5.4, 6.0, and 10.0 min. Figures 28 and 29 show that both skim and whole milk produced a high concentration of the compound responsible for the 5.4 min peak the day after processing in the cotton plugged samples stored at 32°C. It subsequently decreased with time. This peak was significantly higher from the 1st-12th wk in the skim and wks 1 and 4-12 in the whole milk (see Tables 23 and 24). Also, skim milk had more of this material than whole milk.

In both milk samples (skim and whole) which were stored at 32°C and cotton plugged, the 5.4 min peak decreased, as the 6.0 min peak increased (Fig. 30 and 31). Tables 25 and 26 show a significantly higher peak height in both milks from the 8th-16th wk. Samples had approximately the same amount of this compound at the 8th wk in whole milk as it had at 12 wks in the skim milk.

The 10.0 min peak in the skim and whole cotton plugged samples stored at 32°C increased considerably at the 4th and 16th wk (Fig. 32 and 33). This increase was significant at the 4th and 16th wk in whole milk and at the 4th wk in skim

Fig. 26. Concentration vs. storage time for n-hexanal in skim milk.

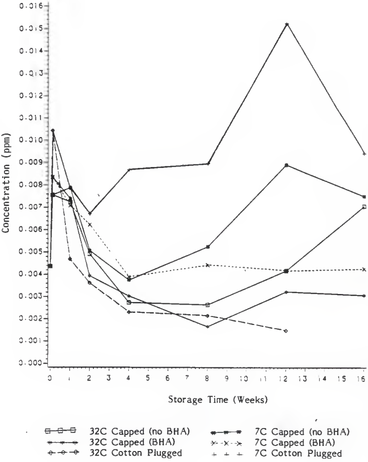


Fig. 27. Concentration vs. storage time for n-hexanal in whole milk.

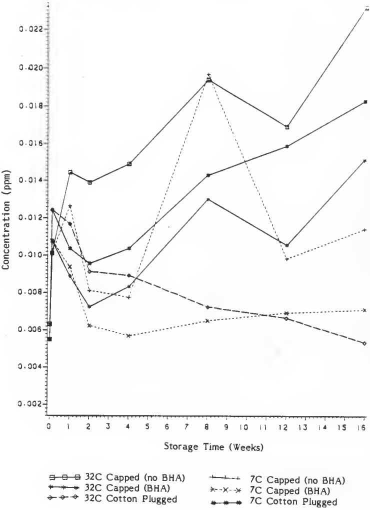


Fig. 28. Peak height vs. storage time for the peak at retention time 5.4 min in skim milk.

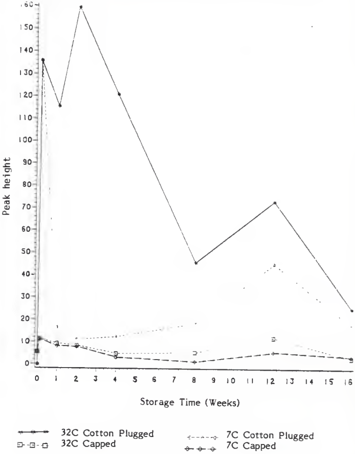


Fig. 29. Peak height vs. storage time for the peak at retention time 5.4 min in whole milk.

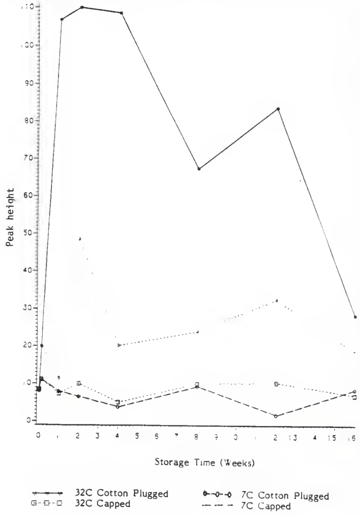


Fig. 30. Peak height vs. storage time for the peak at retention time 6.0 min in skim milk.

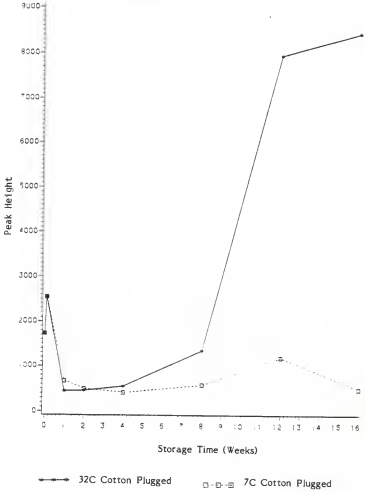


Fig. 31. Peak height vs. storage time for the peak at retention time 6.0 min in whole milk.

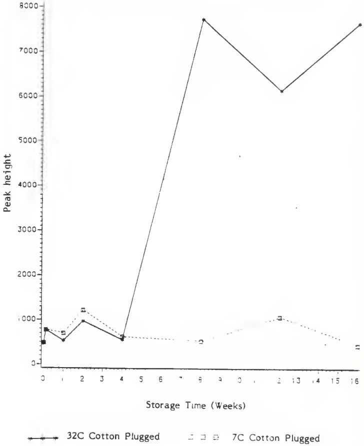


Fig. 32. Peak height vs. storage time for the peak at retention time 10.0 min in skim milk.

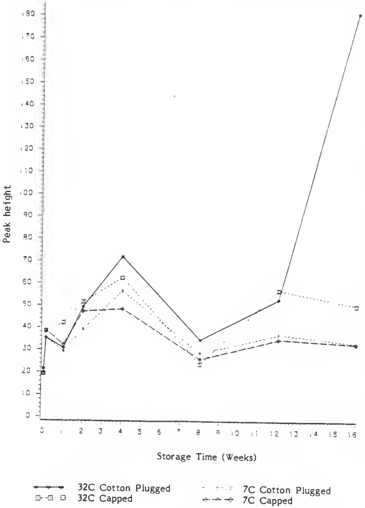
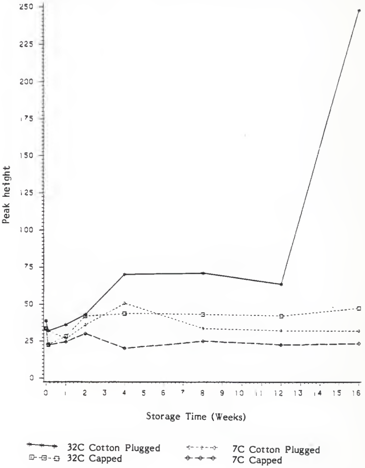


Fig. 33. Peak height vs. storage time for the peak at retention time 10.0 min in whole milk.



(Tables 27 and 28). Although I was unable to determine significance in the skim milk at the 16th wk, from Fig. 32 one can see that the peak increased considerably.

Cooked

Tables 29 and 30 shows that the cooked flavor in both the skim and whole milk increased after processing and decreased upon storage. As expected, a similar pattern occurred in the concentration of methyl sulfide because it is one of the compounds that contibutes to the cooked flavor (26). Initially the cooked flavor decreased rapidly, but after 2 wk it remained relatively constant. Although not statistically significant, the scores were lower in samples stored at 32°C. Also, samples stored at 32°C with cotton plugs received lower cooked flavor scores and showed lower methyl sulfide concentrations.

Stale

Temperature appears to be a major contributor to development of the stale flavor. Tables 31 and 32 show that the whole milk samples stored at 32°C were significantly more stale at 2-16 wks; while, skim milk at 32°C was significantly more stale at wks 2 and 12-16. The difference is not as apparent in the skim milk (Fig. 34) as in the whole mlk stored at 32°C (designated by the upper three lines in Fig. 35). Even though the intensity of stale flavor in skim was not significantly greater from 2-16 wk, it was consistently greater. Figure 35 illustrates that whole milk samples stored at the higher temperature developed a more pronounced stale flavor as early as 4 wks (cotton plugged) whereas samples stored at 7°C were only slightly stale after 16 wks storage. Both figures show that a strong stale flavor developed early in the cotton plugged samples stored at 32°C. One therefore would surmize that the concentration of acetaldehyde, propanal, n-pentanal, and n-hexanal are not directly related to stale flavor, because the 32°C cotton plugged samples usually had low concentration of these compounds in all treatments and

Fig. 34. Stale intensity vs. storage time in skim milk.

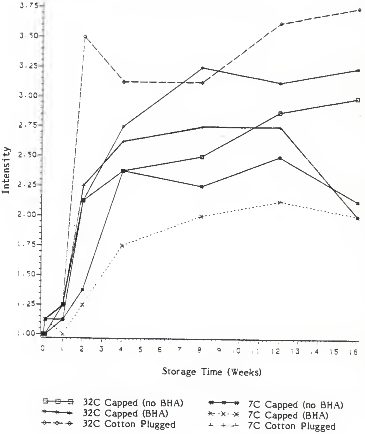
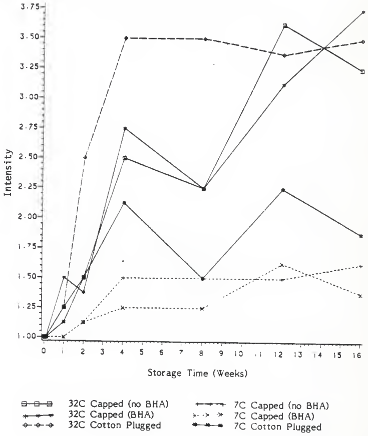


Fig. 35. Stale intensity vs. storage time in whole milk.



yet developed the stale flavor the fastest. Whereas the 32°C capped samples without antioxidant had the highest concentration of these compounds, and yet they developed approximately the same stale intensity 8 wks later in whole milk and about 6 wks later in the skim milk. An exception to this idea and supporting the role of aldehydes in staling occurred with n-pentanal and n-hexanal in skim milk. Concentrations of n-pentanal and n-hexanal in skim milk were the highest in cotton plugged samples stored at 7°C and developed a stronger stale flavor 2 wk earlier than whole milk. If one postulates that decreases in acetaldehyde, propanal, n-pentanal, and n-hexanal in the 32°C cotton plugged samples resulted from involvements in reactions that contributed to a stale flavor, this would explain the early increase in stale due to early decreases in these four compounds. However, it would not explain why the stale flavor developed during the same time that these compounds continually increased in the 32°C capped samples.

If one considers the peaks with retention times of 5.4, 6.0, and 10.0 min as having an additive effect, the relatively high concentration of a component which develops early and responsible for the 5.4 min peak (0.1 wk) plus the high concentrations that develop later in the 6.0 min and 10 min peaks (8 wk and 16 wk, respectively), could be directly related to the rapid and consistent stale flavor development in the 32°C cotton plugged samples. Furthermore, the additive pattern of increases in the compounds at retention times of 5.4, 6.0, and 10.0 min in 7°C cotton plugged samples was similar to the pattern of stale development in these samples but the concentration of these comoponents and the stale flavor was much lower in skim than in whole milk.

Flavor

Flavor scores appear to be inversely related to overall stale flavor (Fig. 36 and 37) development. A decrease in flavor acceptability paralleled an increase in the stale intensity (Fig. 34, 35, 36, and 37). At 32°C storage, flavors scores decreased proportionally to the increase in stale intensity. In samples stored at

Fig. 36. Flavor scores vs. storage time in skim milk.

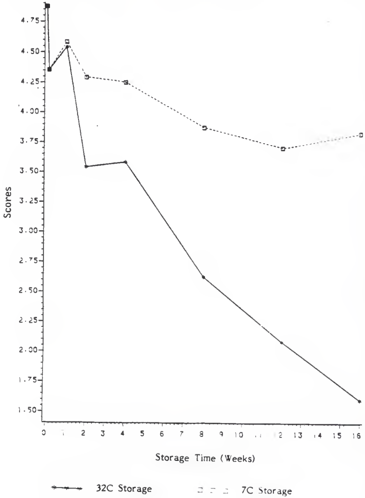
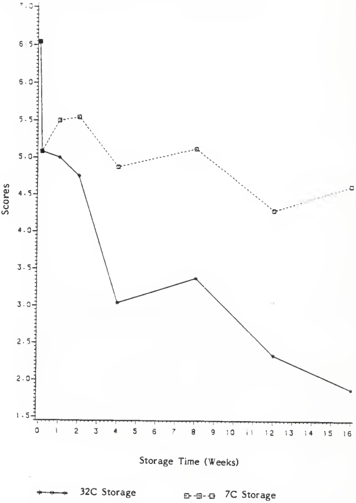


Fig. 37. Flavor scores vs. storage time in whole milk.



7°C, the flavor decreased slightly but scores were still relatively high after 16 wk of storage. The 7°C samples scored higher in acceptability and lower in stale intensity. Samples stored at 7°C had significantly higher scores than those stored at 32°C in skim milk from 4-16 wk and whole milk at 4, 12, and 16 wk (Table 33 and 34).

Flavors relating to TBA and ADV scores

TBA values increased after the 4th wk in the whole milk (Fig. 9) and remained unchanged after the 8th wk of storage in the skim milk (Fig. 10). Therefore, lipid oxidation does not appear to be major contributors to the stale flavor. Since staling apparently was not directly related to increases in acetaldehyde, propanal, n-pentanal, and n-hexanal, or TBA values--it does not appear to be lipid associated. Likewise, panelist did not detect any oxidized flavors in the samples.

ADV's likewise increased late in the study, hence, free fatty acids do not appear to be major contributors to the stale flavor. Also, in the flavor evaluation, panelist did not criticize milk for being rancid.

CONCLUSIONS

Skim and whole milk with normal and low initial dissolved oxygen concentrations were processed in a small pilot-sized "homemade" UHT system. A variety of chemical-quality tests were studied and statistically related to flavor changes in the sterile milk during storage at 7° and 32°C. The following conclusions were drawn from this study.

1. Dissolved oxygen appears to affect the rate of the stale flavor development; however, aldehydes that were measured (probably from oxidation) do not appear to be major contributors to the development of the stale flavor.
2. Decreases in volatile materials that occur in the cotton plugged samples (high oxygen concentration) at both storage temperatures most probably are caused either by loss through the cotton plug or in reactions which consumed the volatiles faster than they were formed.
3. The antioxidant Tenox 2 was only slightly effective at retarding lipid oxidation in our UHT milks.
4. Both whole and skim UHT milk stored at a higher temperature (32°C) develop a more pronounced stale flavor at a faster rate than those milks stored at 7°C.
5. Stale flavor does not come from lipid associated reactions since the time during which staling occurred does not correspond to the production of lipid oxidation products. Furthermore, the off-flavor develops more rapidly in skim milk and TBA values do not parallel its development.

ACKNOWLEDGEMENTS

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APPENDIX A

Temp = Storage temperature (7° or 32°)

Anti = Antioxidants; Y = contains antioxidants; N = no antioxidants

Sweep = Deoxygenation treatment; CP = bottle plugged with sterile cotton plug; NO = no deoxygenation; N2 = deoxygenation

Temp x Anti = Temperature-Antioxidant interaction

Temp x Sweep = Temperature-Deoxygenation interaction

Anti x Sweep = Antioxidant-Deoxygenation interaction

Week 0 = Preprocessed milk

Week 0.1 = Milk the day after processing

We were unable to determine statistical differences between samples at 0 and 0.1 wk because of the way samples were treated before and the day following processing. For example, all samples were refrigerated one day until they were placed in 7°C and 32°C storage. Even though these milks had been treated differently (antioxidant and dissolved oxygen) after one day of refrigeration, samples were essentially the same. The computer verified this and calculated no significant differences.

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*Data missing in some cases due to circumstances beyond our control.

Table 3. Least square means of the BHA values in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	181.2	181.2	161.7 a	163.4 a	163.9 a	157.8 a		
32°	181.2	181.2	162.8 a	164.5 a	165.0 a	156.1 a		
ANTI								
Y	373.2	373.2	335.3 a	338.6 a	339.8 a	324.7 a		
N	-10.8	-10.8	-10.8 b	-10.8 b	-10.8 b	-10.8 b		
SWEEP								
CP	187.8	186.2	162.0 a	162.0 a	162.0 a	146.9 a		
NO	187.8	187.8	166.1 a	163.6 a	164.5 a	167.8 a		
N2	167.8	169.5	158.5 a	166.1 a	167.0 a	156.1 a		
TEMP X ANTI								
7° Y	373.2	373.2	334.2 a	337.5 a	338.6 a	326.4 a		
7° N	-10.8	-10.8	-10.8 b	-10.8 b	-10.8 b	-10.8 b		
32° Y	373.2	373.2	336.4 a	339.8 a	340.9 a	323.1 a		
32° N	-10.8	-10.8	-10.8 b	-10.8 b	-10.8 b	-10.8 b		
TEMP X SWEEP								
7° CP	187.8	186.2	162.8 a	157.8 a	157.8 a	141.1 a		
7° NO	187.8	187.8	166.1 a	166.1 a	167.8 a	169.5 a		
7° N2	167.8	169.5	156.1 a	166.1 a	166.1 a	162.8 a		
32° CP	187.8	186.2	161.1 a	166.1 a	166.1 a	152.8 a		
32° NO	187.8	187.8	166.1 a	166.1 a	161.1 a	166.1 a		
32° N2	167.8	169.5	161.1 a	166.1 a	167.8 a	149.4 a		
ANTI X SWEEP								
Y CP	386.5	383.2	334.8 a	334.8 a	334.8 a	304.7 a		
Y NO	386.5	386.5	343.1 a	338.1 a	339.8 a	346.4 a		
Y N2	346.4	349.8	328.1 a	343.1 a	344.8 a	323.1 a		
N CP	-10.8	-10.8	-10.8 b	-10.8 b	-10.8 b	-10.8 b		
N NO	-10.8	-10.8	-10.8 b	-10.8 b	-10.8 b	-10.8 b		
N N2	-10.8	-10.8	-10.8 b	-10.8 b	-10.8 b	-10.8 b		

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination <u>at a specific week</u>, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations BHA (ppm) from the regression equation of the standard curve.

*Blankspace indicates data missing.

Table 4. Least square means of the BHA values in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	205.1	205.8	190.3 a	190.1 a	195.0 a	212.3 a	NE	205.5 a
32°	205.1	205.8	188.6 a	197.8 a	193.2 a	200.0 a	NE	196.0 a
ANTI								
Y	404.4	405.8	373.1 a	385.1 a	382.4 a	406.5 a	NE	395.8 a
N	5.8	5.8	5.8 a	5.8 b	5.8 b	5.8 b	5.8	5.8 b
SWEEP								
CP	209.7	197.6	188.0 a	200.8 a	192.3 a	214.7 a	208.8	205.0 a
NO	209.7	214.1	187.6 a	189.8 a	195.6 a	212.2 a	206.5	199.3 a
N2	195.9	205.6	192.7 a	195.7 a	194.4 a	191.5 a	NE	198.1 a
TEMP X ANTI								
7° Y	404.4	405.8	374.8 a	380.4 a	384.2 a	418.8 a	NE	405.3 a
7° N	5.8	5.8	5.8 b	5.8 b	5.8 b	5.8 b	5.8	5.8 b
32° Y	404.4	405.8	371.4 a	389.8 a	380.5 a	394.3 a	NE	386.2 a
32° N	5.8	5.8	5.8 b	5.8 b	5.8 b	5.8 b	5.8	5.8 b
TEMP X SWEEP								
7° CP	209.7	197.6	188.8 a	196.6 a	193.2 a	218.4 a	217.1	215.1 a
7° NO	209.7	214.1	188.5 a	187.5 a	197.2 a	213.4 a	206.3	198.9 a
7° N2	195.9	205.6	193.5 a	195.2 a	194.6 a	205.0 a	NE	202.6 a
32° CP	209.7	197.6	187.1 a	205.0 a	191.4 a	211.0 a	200.6	194.9 a
32° NO	209.7	214.1	186.8 a	192.2 a	193.9 a	211.0 a	206.7	199.6 a
32° N2	195.9	205.6	191.9 a	196.2 a	194.2 a	178.1 a	NE	193.5 a
ANTI X SWEEP								
Y CP	413.6	389.4	370.2	395.8 a	378.8 a	423.7 a	411.9 a	404.2 a
Y NO	413.6	422.4	369.5 a	373.9 a	385.3 a	418.7 a	407.2 a	392.8 a
Y N2	386.0	405.5	379.6 a	385.7 a	383.0 a	377.3 a		390.4 a
N CP	5.8	5.8	5.8 b	5.8 b	5.8 b	5.8 b	5.8 b	5.8 b
N NO	5.8	5.8	5.8 b	5.8 b	5.8 b	5.8 b	5.8 b	5.8 b
N N2	5.8	5.8	5.8 b	5.8 b	5.8 b	5.8 b	5.8 b	5.8 b

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations BHA (ppm) from the regression equation of the standard curve.

*Blank space indicates data missing and NE means non-estimatable.

Table 5. Least square means of the dissolved oxygen in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	0.25	2.54	3.74 a	3.68 a	4.37 a	4.62 a	4.98 a	4.48 a
32°	0.25	2.54	3.50 a	3.33 a	3.81 a	3.89 a	3.90 a	3.73 a
ANTI								
Y	0.26	2.72	3.68 a	3.79 a	3.81 a	4.14 a	4.57 a	4.38 a
N	0.23	2.35	3.57 a	3.21 a	4.37 a	4.37 a	4.30 a	3.83 a
SWEEP								
CP	0.26	3.00	6.87 a	7.08 a	7.53 a	7.68 a	8.15 a	7.78 a
NO	0.26	3.10	2.20 a	1.76 b	2.01 c	2.84 b	2.04 b	2.28 a
N2	0.22	1.51	1.79 a	1.67 b	2.72 b	2.24 b	3.12 b	2.25 a
TEMP X ANTI								
7° Y	0.26	2.72	3.66 a	4.10 a	4.00 b	4.54 a	4.85 a	4.70 c
7° N	0.23	2.35	3.82 a	3.25 a	4.74 a	4.70 a	5.10 a	4.25 a
32° Y	0.26	2.72	3.69 a	3.48 a	3.62 c	3.73 a	4.30 a	4.06 a
32° N	0.23	2.35	3.31 a	3.18 a	4.00 b	4.04 a	3.50 a	3.41 a
TEMP X SWEEP								
7° CP	0.26	3.00	6.99 a	7.28 a	7.66 a	8.27 a	8.72 a	8.22 a
7° NO	0.26	3.10	2.82 b	1.96 b	2.52 c	2.98 b	2.72 b	2.81 a
7° N2	0.22	1.51	1.42 b	1.78 b	2.92 b	2.62 b	3.48 b	2.39 a
32° CP	0.26	3.00	6.76 a	6.88 a	7.40 a	7.08 a	7.57 a	7.34 a
32° NO	0.26	3.10	1.58 b	1.56 b	1.50 d	2.72 b	1.36 b	1.74 a
32° N2	0.22	1.51	2.16 b	1.56 b	2.52 c	1.86 b	2.77 b	2.11 a
ANTI X SWEEP								
Y CP	0.29	3.25	6.82 a	7.44 a	7.76 a	7.72 a	8.02 a	7.72 a
Y NO	0.29	3.25	2.44 b	1.84 b	2.05 de	2.05 bc	1.86 b	2.59 a
Y N2	0.21	1.66	1.76 b	2.10 b	1.61 ef	2.64 bc	3.82 b	2.82 a
N CP	0.23	2.76	6.92 a	6.72 a	7.30 b	7.63 a	8.27 a	7.84 a
N NO	0.23	2.94	1.96 b	1.68 b	1.97 def	3.64 bc	2.22 b	1.96 a
N N2	0.23	1.36	1.82 b	1.24 b	3.83 c	1.84 c	2.42 b	1.68 a

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Concentrations in ppm of dissolved oxygen.

Table 6. Least square means of the dissolved oxygen in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	6.10	4.84	4.98 a	5.24 a	5.01 a	5.30 a	4.99 a	4.06 a
32°	6.10	4.84	3.75 b	3.96 a	4.85 a	4.50 b	4.40 a	3.24 a
ANTI								
Y	6.23	4.98	4.24 a	5.10 a	4.89 a	4.82 a	4.69 a	3.60 a
N	5.98	4.71	4.49 a	4.10 a	4.97 a	4.98 a	4.69 a	3.70 a
SWEEP								
CP	6.86	6.52	7.00 a	7.35 a	7.46 a	7.57 a	7.51 a	6.96 a
NO	6.86	5.83	3.31 b	3.24 b	3.92 b	4.04 b	4.08 b	2.07 b
N2	4.58	2.18	2.78 b	3.21 b	3.40 b	3.09 b	2.48 c	1.93 b
TEMP X ANTI								
7° Y	6.23	4.98	4.90 a	5.97 a	5.14 a	5.32 a	4.86 a	4.13 a
7° N	5.98	4.71	5.06 a	4.51 a	4.88 a	5.28 a	5.11 a	3.99 a
32° Y	6.23	4.98	3.58 b	4.22 a	4.65 a	4.33 a	4.52 a	3.07 a
32° N	5.98	4.71	3.91 b	3.69 a	5.05 a	4.67 a	4.27 a	3.41 a
TEMP X SWEEP								
7° CP	6.86	6.52	7.84 a	7.33 a	7.46 ab	7.58 a	8.41 a	7.36 a
7° NO	6.86	5.83	4.32 c	5.06 abc	5.10 abc	4.26 bc	3.16 d	2.56 b
7° N2	4.58	2.18	2.57 de	3.34 bcd	2.46 bc	4.05 bc	3.39 d	2.26 b
32° CP	6.86	6.52	6.14 b	7.37 ab	7.46 ab	7.56 a	6.62 b	6.55 a
32° NO	6.86	5.83	2.10 e	1.41 cd	2.74 bc	3.82 bc	5.00 c	1.58 b
32° N2	4.58	2.18	3.00 d	3.08 bcd	4.34 bc	2.13 bd	1.57 e	1.60 b
ANTI X SWEEP								
Y CP	8.27	6.93	7.29 a	7.20 ab	7.22 a	7.34 a	7.62 a	7.10 a
Y NO	8.27	5.86	3.84 b	3.55 bc	3.76 b	4.20 b	4.33 bc	1.96 b
Y N2	2.14	2.14	1.59 d	4.54 abc	3.70 b	2.94 b	2.12 d	1.73 b
N CP	5.46	6.12	6.70 a	7.50 ab	7.70 a	7.80 a	7.40 a	6.81 a
N NO	5.46	5.80	2.78 c	2.92 bc	4.09 b	3.88 b	3.83 bcd	2.17 b
N N2	7.02	2.21	3.98 b	1.88 bc	3.11 b	3.24 b	2.84 cd	2.12 b

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Concentrations in ppm of dissolved oxygen.

Table 7. Least square means of TBA values in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.025	.030	.026 a	.022 a	.027 a	.022 a		.023 a
32°	.025	.030	.027 a	.025 a	.028 a	.027 a		.025 a
ANTI								
Y	.024	.024	.026 a	.023 a	.028 a	.026 a		.025 a
N	.026	.036	.027 a	.023 a	.028 a	.023 a		.022 a
SWEEP								
CP	.024	.041	.028 a	.026 a	.030 a	.021 a		.024 a
NO	.024	.026	.028 a	.024 a	.026 a	.026 a		.024 a
N2	.027	.025	.024 a	.020 a	.027 a	.025 a		.024 a
TEMP X ANTI								
7° Y	.024	.024	.026 a	.021 a	.025 a	.023 a		.025 a
7° N	.026	.036	.026 a	.026 a	.028 a	.020 a		.021 a
32° Y	.024	.024	.026 a	.026 a	.030 a	.028 a		.026 a
32° N	.024	.036	.028 a	.024 a	.026 a	.026 a		.024 a
TEMP X SWEEP								
7° CP	.024	.041	.028 a	.024 a	.030 a	.020 a		.025 a
7° NO	.024	.026	.026 a	.022 a	.024 a	.022 a		.022 a
7° N2	.027	.025	.023 a	.020 a	.027 a	.022 a		.022 a
32° CP	.024	.041	.027 a	.029 a	.029 a	.022 a		.023 a
32° NO	.024	.026	.028 a	.026 a	.028 a	.031 a		.026 a
32° N2	.027	.025	.026 a	.019 a	.027 a	.028 a		.025 a
ANTI X SWEEP								
Y CP	.024	.027	.029 a	.030 a	.030 a	.022 a		.026 a
Y NO	.024	.024	.028 a	.023 a	.027 a	.031 a		.026 a
Y N2	.023	.022	.020 a	.017 a	.026 a	.024 a		.025 a
N CP	.024	.055	.026 a	.022 a	.029 a	.021 a		.022 a
N NO	.024	.026	.028 a	.025 a	.025 a	.022 a		.023 a
N N2	.030	.028	.029 a	.022 a	.028 a	.026 a		.022 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Original TBA unit is absorbance.

*Blankspace indicates data missing.

Table 8. Least square means of the TBA values in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.020	.024	.023 a	.026 a	.019 a		.025 a	.023 a
32°	.020	.024	.022 a	.024 a	.018 a		.027 a	.029 b
ANTI								
Y	.017	.024	.021 a	.024 a	.019 a		.025 a	.025 a
N	.022	.024	.024 a	.026 a	.019 a		.027 a	.027 b
SWEEP								
CP	.020		.023 a	.028 a	.019 a		.027 a	.027 a
NO	.020	.024	.024 a	.024 b	.018 a		.027 a	.027 a
N2	.019	.024	.020 a	.024 b	.019 a		.024 a	.024 b
TEMP X ANTI								
7° Y	.017	.024	.022 a	.025 abc	.020 a		.024 a	.022 c
7° N	.022	.024	.024 a	.027 ab	.019 a		.026 a	.023 c
32° Y	.017	.024	.020 a	.022 bc	.018 a		.026 a	.028 b
32° N	.022	.024	.024 a	.025 abc	.019 a		.029 a	.031 a
TEMP X SWEEP								
7° CP	.020		.022 a	.030 ab	.021 a		.026 a	.023 a
7° NO	.020	.024	.027 a	.024 bc	.018 a		.027 a	.023 a
7° N2	.019	.024	.020 a	.025 abc	.020 a		.023 a	.022 a
32° CP	.020		.024 a	.026 abc	.017 a		.028 a	.030 a
32° NO	.020	.024	.021 a	.023 bc	.018 a		.028 a	.031 a
32° N2	.019	.024	.020 a	.023 bc	.019 a		.026 a	.026 a
ANTI X SWEEP								
Y CP	.016		.023 a	.026 abc	.018 a		.028 a	.026 abc
Y NO	.016	.024	.022 a	.022 bc	.019 a		.024 a	.024 bc
Y N2	.018	.024	.018 a	.024 bc	.019 a		.022 a	.024 bc
N CP	.024		.023 a	.030 ab	.020 a		.025 a	.027 abc
N NO	.024	.024	.026 a	.024 bc	.017 a		.030 a	.030 ab
N N2	.019	.024	.022 a	.024 bc	.020 a		.026 a	.024 bc

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Original TBA unit is absorbance.

*Blankspace indicates data missing.

Table 9. Least square means of acid degree values (ADV) in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	0.88	0.87	0.87 a	0.87 a	0.78 b	1.01 a	1.08 b	0.90 b
32°	0.88	0.87	0.88 a	0.87 a	0.87 a	1.10 a	1.15 a	1.46 a
ANTI								
Y	0.87	0.87	0.88 a	0.87 a	0.83 a	1.05 a	1.10 a	1.17 a
N	0.88	0.87	0.87 a	0.87 a	0.81 a	1.06 a	1.12 a	1.20 a
SWEEP								
CP	0.88	0.87	0.87 a	0.87 a	0.82 a	1.06 a	1.13 ab	1.18 a
NO	0.88	0.88	0.88 a	0.87 a	0.82 a	1.07 a	1.11 abc	1.18 a
N2	0.87	0.87	0.87 a	0.87 a	0.82 a	1.04 a	1.10 bc	1.18 a
TEMP X ANTI								
7° Y	0.87	0.87	0.87 a	0.87 a	0.77 a	0.98 b	1.06 b	0.90 b
7° N	0.88	0.87	0.87 a	0.87 a	0.78 a	1.04 ab	1.09 b	0.91 b
32° Y	0.87	0.87	0.88 a	0.88 a	0.88 a	1.11 a	1.15 a	1.43 a
32° N	0.88	0.87	0.88 a	0.87 a	0.85 a	1.08 ab	1.15 a	1.48 a
TEMP X SWEEP								
7° CP	0.88	0.87	0.87 a	0.87 a	0.78 b	1.01 a	1.09 bc	0.90 b
7° NO	0.88	0.88	0.88 a	0.88 a	0.78 b	1.05 a	1.09 bcd	0.89 b
7° N2	0.87	0.87	0.87 a	0.87 a	0.78 b	0.98 a	1.05 cd	0.92 b
32° CP	0.88	0.87	0.88 a	0.87 a	0.85 a	1.10 a	1.17 a	1.47 a
32° NO	0.88	0.88	0.88 a	0.87 a	0.87 a	1.10 a	1.14 a	1.47 a
32° N2	0.87	0.87	0.88 a	0.88 a	0.87 a	1.09 a	1.14 a	1.45 a
ANTI X SWEEP								
Y CP	0.87	0.86	0.87 a	0.87 a	0.83 a	1.06 a	1.12 ab	1.18 abc
Y NO	0.87	0.88	0.88 a	0.88 a	0.83 a	1.04 a	1.11 abc	1.18 abc
Y N2	0.87	0.87	0.88 a	0.87 a	0.83 a	1.04 a	1.08 bc	1.14 bc
N CP	0.89	0.87	0.88 a	0.87 a	0.80 a	1.06 a	1.14 ab	1.18 abc
N NO	0.89	0.87	0.87 a	0.87 a	0.80 a	1.10 a	1.12 abc	1.18 abc
N N2	0.87	0.87	0.87 a	0.87 a	0.80 a	1.03 a	1.11 abc	1.22 ab

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*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

Table 10. Least square means of acetone in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	5676.0	9944.0	8928.7 a	8980.5 a	7916.7 a	7293.1 a	7810.9 a	6466.5 b
32°	5676.0	9944.0	8519.1 b	8834.9 a	6613.3 b	7798.9 a	9354.6 a	9273.5 a
ANTI								
Y	6358.0	10061.3	8928.7 a	8750.9 a	7486.7 a	7502.1 a	8392.6 a	8007.7 a
N	4994.0	9826.7	8519.1 b	9064.5 a	7043.3 b	7589.9 a	8772.9 a	7732.3 a
SWEEP								
CP	5995.0	10142.0	7498.2 c	6456.8 c	4645.0 c	2847.6 c	2666.4 b	2871.3 c
NO	5995.0	10560.0	10114.5 a	11054.4 a	9360.0 a	10617.6 a	12458.7 a	11144.5 a
N2	5038.0	9130.0	8559.1 b	9212.0 b	7790.0 b	9172.8 b	10623.1 a	9594.1 b
TEMP X ANTI								
7° Y	6358.0	10061.3	9101.4 a	8728.5 a	8346.7 a	7268.8 a	7680.9 a	6573.6 b
7° N	4994.0	9826.7	8756.0 b	9232.5 a	7486.7 b	7317.3 a	7941.0 a	6359.4 b
32° Y	6358.0	10061.3	8756.0 b	8773.3 a	6626.7 c	7735.5 a	9104.4 a	9441.8 a
32° N	4994.0	9826.7	8282.3 c	8896.5 a	6600.0 c	7862.4 c	9604.7 a	9105.2 a
TEMP X SWEEP								
7° CP	5995.0	10142.0	8515.1 c	7924.0 d	6670.0 c	4608.8 e	3956.7 e	2381.7 d
7° NO	5995.0	10560.0	9834.5 b	10550.4 bc	9120.0 a	9520.0 bcd	10734.3 bcd	9477.8 b
7° N2	5038.0	9130.0	8437.0 c	8467.2 d	7960.0 b	7750.4 cd	8741.8 cd	7539.8 c
32° CP	5995.0	10142.0	6481.2 d	4989.6 e	2620.0 d	1086.4 f	1376.1 e	3360.9 d
32° NO	5995.0	10560.0	10395.0 a	11558.4 a	9600.0 a	11715.2 ab	14183.1 ab	12811.2 a
32° N2	5038.0	92130.0	8681.2 c	9956.8 c	7620.0 b	10595.2 abc	12504.5 abc	11648.4 a
ANTI X SWEEP								
Y CP	7040.0	9900.0	7569.1 e	6372.8 d	4800.0 d	2660.0 b	2501.6 b	3162.0 d
Y NO	7040.0	10912.0	10340.0 a	10729.6 ab	9500.0 a	10729.6 a	12504.5 a	11322.0 ab
Y N2	4994.0	9372.0	8870.0 c	9150.4 bc	8160.0 b	9116.8 a	10171.9 a	9539.0 bc
N CP	4950.0	10384.0	7427.0 e	6540.8 d	4490.0 d	3035.2 b	2831.3 b	2580.6 d
N NO	4950.0	10208.0	9889.0 b	11379.2 ab	9220.0 a	10505.6 a	12412.9 a	10967.0 abc
N N2	5082.0	8888.0	8241.2 d	9273.6 c	7420.0 c	9228.8 a	11074.4 a	9649.2 bc

[•] Unable to determine significant difference for week 0 and 0.1.

[•] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[•] Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 11. Least square means of acetone in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	9056.2	11684.2	11518.5 a	10201.3 a	9189.2 a	9673.3 a	9346.7 a	8912.9 b
32°	9056.2	11684.2	11821.3 a	10454.0 a	9533.7 a	10626.7 a	10432.3 a	12203.1 a
ANTI								
Y	8885.9	11770.9	11394.9 a	9748.0 a	9195.7 a	9444.0 a	9413.7 a	9936.1 a
N	9226.4	11597.6	11944.8 a	10907.3 a	9520.9 a	10856.0 a	10365.3 a	11180.0 a
SWEEP								
CP	9427.8	12471.6	10289.8 a	8456.0 b	6695.3 c	4834.0 b	3882.5 b	3864.0 b
NO	9427.8	12207.1	13153.2 a	12120.0 a	11283.7 a	13546.0 a	13078.0 a	14347.9 a
N2	8312.9	10374.0	11566.7 a	10407.0 a	10096.0 b	12070.0 a	12708.0 a	13462.1 a
TEMP X ANTI								
7° Y	8885.9	11770.9	11224.7 a	9680.0 a	8958.0 bc	8701.3 a	8765.3 a	8542.1 bc
7° N	9226.4	11597.6	11812.3 a	10722.7 a	9407.8 abc	10645.3 a	9928.0 a	9283.7 bc
32° Y	8885.9	11770.9	11565.2 a	9816.0 a	9433.4 abc	10186.7 a	10062.0 a	11330.0 abc
32° N	9226.4	11597.6	12077.4 a	11092.0 a	9634.0 ab	11066.7 a	10802.7 a	13076.2 ab
TEMP X SWEEP								
7° CP	9427.8	12471.6	11006.2 abc	9692.0 bcd	8415.3 d	6888.0 bcd	5992.0 c	4820.4 c
7° NO	9427.8	12207.1	12452.6 abc	10880.0 abc	10253.5 c	11912.0 abc	11448.0 b	11762.6 b
7° N2	8312.9	10374.0	11096.6 abc	10032.0 bcd	8879.9 d	10220.0 abc	10600.0 b	10155.8 b
32° CP	9427.8	12471.6	9573.4 bc	7220.0 cd	4975.3 e	2780.0 cd	1773.0 d	2907.7 c
32° NO	9427.8	12207.1	13853.8 ab	13360.0 ab	12313.9 a	15180.0 a	14708.0 a	16933.2 a
32° N2	8312.9	10374.0	12036.8 abc	10782.0 abc	11312.0 b	13920.0 ab	14816.0 a	16768.4 a
ANTI X SWEEP								
Y CP	8983.2	11947.2	10079.6 a	7820.0 de	6284.2 e	4760.0 b	3725.0 a	3687.4 b
Y NO	8983.2	11600.6	12769.0 a	11452.0 abcd	11615.0 a	12872.0 a	12288.0 a	13616.6 a
Y N2	8691.4	11764.8	11336.2 a	9972.0 bcde	9687.9 c	10700.0 a	12288.0 a	12504.2 a
N CP	9872.4	12996.0	10500.0 a	9092.0 bcde	7106.4 d	4908.0 b	4040.0 a	4040.7 b
N NO	9872.4	12813.6	13537.4 a	12788.0 ab	10952.4 b	14220.0 a	13868.0 a	15079.2 a
N N2	7934.4	8983.2	11797.2 a	10842.0 abcd	10504.0 b	13440.0 a	13188.0 a	14420.0 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Peak heights relative to the peak of 1 ppm acetone - 1200 units (% full-scale deflection peak height x attenuation).

Table 12. Least square means of methyl sulfide in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	271.9	575.5	505.1 a	432.9 a	285.5 a	315.8 a	279.8 a	227.1 a
32°	271.9	575.5	438.0 a	362.5 a	202.7 b	329.5 a	270.3 a	318.8 a
ANTI								
Y	156.9	613.1	101.9 a	359.5 a	237.7 a	290.1 a	243.1 b	235.9 a
N	386.8	537.9	92.4 a	435.9 a	250.5 a	355.2 a	307.0 a	310.0 a
SWEEP								
CP	330.0	574.2	140.2 b	71.1 b	39.2 c	15.1 b	4.6 c	3.7 a
NO	330.0	614.9	677.3 a	607.0 a	300.5 b	525.8 a	453.2 a	452.4 a
N2	155.6	537.4	597.0 a	514.9 a	392.5 a	427.0 a	367.3 b	362.9 a
TEMP X ANTI								
7° Y	156.9	613.1	518.1 a	381.2 a	282.7 ab	320.7 a	253.6 abc	186.3 a
7° N	386.8	537.9	492.1 a	484.6 a	288.3 ab	311.0 a	305.9 ab	267.9 a
32° Y	156.9	613.1	403.7 a	337.9 a	192.7 bc	259.5 a	232.5 bc	285.4 a
32° N	386.8	537.9	472.3 a	387.1 a	212.7 abc	399.5 a	308.1 ab	352.2 a
TEMP X SWEEP								
7° CP	330.0	574.2	259.6 d	139.4 c	78.5 c	24.6 bc	7.6 e	2.6 a
7° NO	330.0	614.9	614.9 abc	614.9 ab	323.0 b	474.9 ab	417.5 abc	361.1 a
7° N2	155.6	537.4	640.7 abc	544.3 ab	455.0 a	448.0 ab	414.2 bc	317.7 a
32° CP	330.0	574.2	20.9 e	2.8 d	0.0 c	5.6 c	1.6 e	4.8 a
32° NO	330.0	614.9	739.8 ab	599.2 ab	278.0 b	576.8 ab	488.9 ab	543.7 a
32° N2	155.6	537.4	553.3 bc	485.5 b	330.0 b	406.0 abc	320.5 d	408.0 a
ANTI X SWEEP								
Y CP	187.0	618.2	134.8 d	34.2 b	42.5 d	10.1 bc	0.0 c	4.8 a
Y NO	187.0	687.5	712.8 ab	648.5 a	303.0 bc	563.4 ab	474.2 a	422.3 a
Y N2	96.8	533.5	535.2 bc	395.9 a	367.5 abc	296.8 abc	255.1 b	280.5 a
N CP	473.0	530.2	145.8 d	108.1 b	36.0 d	20.2 bc	9.3 c	2.6 a
N NO	473.0	542.3	641.8 abc	565.6 a	298.0 bc	488.3 ab	432.2 a	482.5 a
N N2	214.5	541.2	658.9 abc	633.9 a	417.5 ab	557.2 ab	479.6 a	445.2 a

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Peak heights relative to the peak of 1 ppm acetone - 1200 units (% full-scale deflection peak height x attenuation).

Table 13. Least square means of methyl sulfide whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	131.9	624.5	564.8 a	463.3 a	407.4 a	352.3 a	316.3 a	324.6 a
32°	131.9	624.5	494.0 a	415.8 a	400.8 a	373.2 a	285.8 a	397.8 a
ANTI								
Y	130.0	611.4	475.5 a	409.3 a	406.7 a	319.3 a	267.0 a	371.0 a
N	133.8	637.6	586.3 a	469.8 a	401.5 a	406.2 a	335.2 a	351.4 a
SWEEP								
CP	148.2	558.6	245.2 b	167.0 b	107.6 c	65.2 b	23.5 b	22.7 b
NO	148.2	705.1	685.9 a	599.8 a	537.3 b	527.8 a	446.0 a	551.6 a
N2	99.2	609.9	657.1 a	552.0 a	567.4 a	495.2 a	433.8 a	509.3 a
TEMP X ANTI								
7° Y	130.0	611.4	509.6 a	443.7 a	409.0 a	275.0 a	301.3 a	299.0 a
7° N	133.8	637.6	620.0 a	483.0 a	405.7 a	429.7 a	331.3 a	350.2 a
32° Y	130.0	611.4	441.4 a	375.0 a	404.3 a	363.7 a	232.7 a	442.9 a
32° N	133.8	637.6	546.5 a	456.7 a	397.3 a	382.7 a	339.0 a	352.6 a
TEMP X SWEEP								
7° CP	148.2	558.6	326.6 b	233.5 abc	157.6 c	89.0 abc	42.0 b	39.1 bcd
7° NO	148.2	705.1	705.1 a	598.5 ab	532.8 b	526.5 ab	467.0 a	500.6 ab
7° N2	99.2	609.9	662.7 a	558.0 abc	531.8 b	441.5 abc	440.0 a	434.1 abc
32° CP	148.2	558.6	163.8 b	100.5 bc	57.6 d	41.5 bc	5.0 b	6.2 cd
32° NO	148.2	705.1	666.7 a	601.0 ab	541.9 b	529.0 ab	425.0 a	602.6 ab
32° N2	99.2	609.9	651.4 a	546.0 ab	603.0 a	529.0 ab	427.0 a	584.5 ab
ANTI X SWEEP								
Y CP	139.1	495.9	199.4 bc	118.5 bc	128.3 d	68.5 bc	22.5 b	28.8 b
Y NO	139.1	663.5	583.1 a	563.5 ab	530.2 bc	471.5 abc	381.0 a	556.2 a
Y N2	111.7	674.9	644.1 a	546.0 abc	561.6 ab	418.0 abc	397.5 a	527.9 a
N CP	157.3	621.3	291.0 bc	215.5 abc	86.9 e	62.0 bc	24.5 b	16.5 b
N NO	157.3	746.7	788.7 a	636.0 ab	544.4 bc	584.0 ab	511.0 a	546.9 a
N N2	86.6	544.9	670.1 ab	558.0 abc	573.2 ab	572.5 ab	470.0 a	490.8 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination <u>at a specific week</u>, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 15. Least square means of acetaldehyde in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0130	.0570	.0450 a	.0440 a	.0400 a	.0360 a	.0400 b	.0360 a
32°	.0130	.0570	.4670 a	.0490 a	.0400 a	.0460 a	.0550 a	.0520 a
ANTI								
Y	.0130	.0500	.0470 a	.0460 a	.0410 a	.0410 a	.0460 a	.0450 a
N	.0130	.0630	.0450 a	.0480 a	.0390 a	.0410 a	.0490 a	.0430 a
SWEEP								
CP	.0127	.0570	.0460 a	.0460 ab	.0350 bc	.0290 b	.0270 b	.0280 bc
NO	.0127	.0550	.0510 a	.0520 a	.0460 ab	.0470 a	.0590 a	.0550 ab
N2	.0140	.0580	.0400 a	.0420 b	.0390 abc	.0470 a	.0580 a	.0490 abc
TEMP X ANTI								
7° Y	.0130	.0500	.0450 a	.0410 a	.0400 a	.0350 a	.0370 bc	.0360 a
7° N	.0130	.0630	.0450 a	.0480 a	.0410 a	.0360 a	.0430 abc	.0360 a
32° Y	.0130	.0500	.0490 a	.0510 a	.0420 a	.0460 a	.0560 ab	.0550 a
32° N	.0130	.0630	.0440 a	.0480 a	.0370 a	.0460 a	.0550 ab	.0500 a
TEMP X SWEEP								
7° CP	.0127	.0570	.0470 ab	.0440 abc	.0390 abc	.0320 cd	.0300 bcd	.0260 bc
7° NO	.0127	.0550	.0460 ab	.0510 abc	.0420 abc	.0380 bcd	.0460 bc	.0440 abc
7° N2	.0140	.0580	.0420 ab	.0380 bc	.0400 abc	.0370 cd	.0440 bc	.0370 abc
32° CP	.0127	.0570	.0460 ab	.0490 abc	.0300 bc	.0260 cd	.0230 cd	.0300 abc
32° NO	.0127	.0550	.0560 a	.0540 ab	.0500 ab	.0570 ab	.0710 a	.0650 ab
32° N2	.0140	.0580	.0380 b	.0450 abc	.0390 abc	.0560 abc	.0720 a	.0610 ab
ANTI X SWEEP								
Y CP	.0116	.0570	.0548 abc	.0540 abc	.0360 bcd	.0300 bc	.0290 b	.0330 ab
Y NO	.0116	.0550	.0545 abcd	.0550 ab	.0550 ab	.0520 ab	.0600 a	.0590 ab
Y N2	.0160	.0380	.0315 cde	.0290 cd	.0320 cd	.0410 abc	.0500 a	.0440 ab
N CP	.0138	.0580	.0380 bcde	.0390 bcd	.0340 bcd	.0280 bc	.0240 b	.0230 b
N NO	.0138	.0550	.0477 abcde	.051 abc	.0370 bcd	.0430 abc	.0570 a	.0500 ab
N N2	.0121	.0780	.0480 abcd	.0540 ab	.0460 abc	.0530 ab	.0640 a	.0550 ab

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations in ppm from the regression equation of the standard curve.

Table 16. Least square means of acetaldehyde in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0086	.0399	.0438 a	.0350 a	.0312 b	.0378 b	.0364 b	.0367 b
32°	.0086	.0399	.0441 a	.0418 a	.0432 a	.0546 a	.0536 a	.0559 a
ANTI								
Y	.0086	.0330	.0376 a	.0334 a	.0343 a	.0410 a	.0408 b	.0396 a
N	.0086	.0467	.0502 a	.0434 a	.0400 a	.0515 a	.0493 a	.0510 a
SWEEP								
CP	.0087	.0490	.0396 a	.0370 a	.0348 bc	.0373 a	.0353 b	.0306 b
NO	.0087	.0355	.0484 a	.0393 a	.0374 abc	.0521 a	.0516 a	.0557 a
N2	.0085	.0351	.0437 a	.0380 a	.0391 ab	.0494 a	.0481 a	.0496 a
TEMP X ANTI								
7° Y	.0086	.0330	.0380 a	.0297 a	.0287 d	.0306 bc	.0304 cd	.0287 bc
7° N	.0086	.0467	.0495 a	.0404 a	.0336 c	.0451 abc	.0424 bcd	.0406 abc
32° Y	.0086	.0330	.0372 a	.0371 a	.0398 b	.0513 abc	.0511 abc	.0505 ab
32° N	.0086	.0467	.0510 a	.0465 a	.0464 a	.0580 ab	.0562 ab	.0613 ab
TEMP X SWEEP								
7° CP	.0087	.0490	.0433 a	.0382 a	.0308 c	.0348 a	.0321 b	.0272 cde
7° NO	.0087	.0355	.0473 a	.0326 a	.0298 c	.0386 a	.0388 b	.0412 bcde
7° N2	.0085	.0351	.0407 a	.0343 a	.0328 c	.0401 a	.0382 b	.0356 cde
32° CP	.0087	.0490	.0359 a	.0359 a	.0389 b	.0398 a	.0385 b	.0340 cde
32° NO	.0087	.0355	.0495 a	.0459 a	.0450 a	.0655 a	.0645 a	.0701 abe
32° N2	.0085	.0351	.0468 a	.0437 a	.0455 a	.0586 a	.0580 a	.0635 abc
ANTI X SWEEP								
Y CP	.0084	.0307	.0320 bc	.0278 a	.0293 d	.0352 a	.0312 cde	.0285 cde
Y NO	.0084	.0299	.0391 abc	.0336 a	.0362 bc	.0441 a	.0453 abcde	.0461 abcde
Y N2	.0089	.0386	.0416 abc	.0388 a	.0372 bc	.0436 a	.0457 abcde	.0441 abcde
N CP	.0089	.0674	.0472 abc	.0463 a	.0404 a	.0394 a	.0394 bcde	.0327 bcde
N NO	.0089	.0412	.0576 ab	.0449 a	.0386 abc	.0600 a	.0580 abc	.0652 abc
N N2	.0080	.0316	.0459 abc	.0392 a	.0410 ab	.0550 a	.0505 abcd	.0550 abcd

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Concentrations in ppm from the regression equation of the standard curve.

Table 17. Least square means of propanal in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0101	.0122	.0109 a	.0111 a	.0113 a	.0109 a	.0112 a	.0109 a
32°	.0101	.0122	.0110 a	.0113 a	.0110 b	.0112 a	.0113 a	.0117 a
ANTI								
Y	.0101	.0119	.0109 a	.0111 a	.0112 a	.0110 a	.0112 a	.0112 a
N	.0101	.0125	.0110 a	.0112 a	.0111 b	.0112 a	.0112 a	.0115 a
SWEEP								
CP	.0101	.0144	.0112 a	.0111 a	.0110 b	.0110 a	.0110 a	.0108 a
NO	.0101	.0112	.0109 b	.0113 a	.0113 a	.0112 a	.0115 a	.0118 a
N2	.0102	.0111	.0106 c	.0110 a	.0111 b	.0111 a	.0112 a	.0114 a
TEMP X ANTI								
7° Y	.0101	.0119	.0109 a	.0108 a	.0113 a	.0108 bc	.0112 a	.0110 a
7° N	.0101	.0125	.0110 a	.0113 a	.0112 a	.0110 abc	.0112 a	.0109 a
32° Y	.0101	.0119	.0110 a	.0114 a	.0111 a	.0111 abc	.0113 a	.0114 a
32° N	.0101	.0125	.0110 a	.0112 a	.0109 b	.0115 ab	.0112 a	.0120 a
TEMP X SWEEP								
7° CP	.0101	.0144	.0112 a	.0112 a	.0115 ab	.0114 abc	.0119 abcd	.0111 a
7° NO	.0101	.0112	.0109 b	.0112 a	.0112 abc	.0107 cd	.0109 bcde	.0110 a
7° N2	.0102	.0111	.0106 c	.0107 a	.0111 bc	.0107 ad	.0108 cde	.0107 a
32° CP	.0101	.0144	.0112 a	.0110 a	.0105 d	.0105 d	.0101 de	.0104 a
32° NO	.0101	.0112	.0111 a	.0114 a	.0114 abc	.0118 abc	.0121 abc	.0127 a
32° N2	.0102	.0111	.0106 c	.0114 a	.0111 bc	.0116 abc	.0116 abcde	.0121 a
ANTI X SWEEP								
Y CP	.0101	.0139	.0113 a	.0112 a	.0110 b	.0108 a	.0110 a	.0106 a
Y NO	.0101	.0112	.0111 bc	.0112 a	.0115 a	.0113 a	.0116 a	.0118 a
Y N2	.0101	.0101	.0104 e	.0108 a	.0111 b	.0108 a	.0111 a	.0112 a
N CP	.0101	.0149	.0113 a	.0110 a	.0109 b	.0110 a	.0109 a	.0110 a
N NO	.0101	.0112	.0109 bcd	.0114 a	.0111 b	.0112 a	.0114 a	.0118 a
N N2	.0102	.0114	.0108 cd	.0112 a	.0111 b	.0114 a	.0113 a	.0116 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations in ppm from the regression equation of the standard curve.

Table 18. Least square means of propanal in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0105	.0117	.0114 a	.0117 a	.0111 b	.0115 a	.0113 a	.0113 b
32°	.0105	.0117	.0114 a	.0117 a	.0117 a	.0123 a	.0117 a	.0125 a
ANTI								
Y	.0106	.0115	.0112 b	.0115 a	.0112 b	.0116 a	.0112 a	.0118 a
N	.0105	.0120	.0115 a	.0119 a	.0116 a	.0122 a	.0118 a	.0121 a
SWEEP								
CP	.0105	.0120	.0114 a	.0122 a	.0115 a	.0118 a	.0115 a	.0111 b
NO	.0105	.0118	.0115 a	.0117 a	.0114 a	.0121 a	.0120 a	.0126 a
N2	.0106	.0114	.0113 a	.0113 a	.1120 b	.0118 a	.0111 a	.0120 a
TEMP X ANTI								
7° Y	.0106	.0115	.0112 a	.0113 a	.0110 c	.0111 a	.0111 a	.0111 b
7° N	.0105	.0120	.0115 a	.0120 a	.0112 b	.0119 a	.0115 a	.0115 b
32° Y	.0106	.0115	.0112 a	.0117 a	.0114 b	.0122 a	.0112 a	.0124 a
32° N	.0105	.0120	.0116 a	.0118 a	.0120 a	.0125 a	.0122 a	.0127 a
TEMP X SWEEP								
7° CP	.0105	.0120	.0114 a	.0125 a	.0113 bc	.0118 a	.0117 a	.0115 b
7° NO	.0105	.0118	.0114 a	.0113 a	.0111 bcd	.0113 a	.0111 a	.0115 b
7° N2	.0106	.0114	.0112 a	.0112 a	.0109 cd	.0114 a	.0110 a	.0109 b
32° CP	.0105	.0120	.0114 a	.0119 a	.0118 a	.0117 a	.0112 a	.0108 b
32° NO	.0105	.0118	.0115 a	.0120 a	.0118 a	.0130 a	.0127 a	.0136 a
32° N2	.0106	.0114	.0114 a	.0113 a	.0116 a	.0123 a	.0113 a	.0132 a
ANTI X SWEEP								
Y CP	.0105	.0115	.0112 bc	.0120 a	.0113 bc	.0116 a	.0115 a	.0110 cd
Y NO	.0105	.0116	.0112 bc	.0115 a	.0113 bc	.0118 a	.0115 a	.0121 abcd
Y N2	.0107	.0112	.0112 bc	.0111 a	.0109 e	.0115 a	.0105 a	.0121 abc
N CP	.0105	.0124	.0116 abc	.0124 a	.0118 ab	.0119 a	.0115 a	.0112 bcd
N NO	.0105	.0120	.0117 ab	.0119 a	.0116 abc	.0125 a	.0123 a	.0130 ab
N N2	.0105	.0115	.0114 abc	.0114 a	.0116 abc	.0122 a	.0118 a	.0120 bcd

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations in ppm from the regression equation of the standard curve.

Table 19. Least square means of n-pentanal in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0440	.0540	.0468 a	.0458 a	.0455 a	.0462 a	.0546 a	.0483 a
32°	.0440	.0540	.0465 a	.0455 a	.0448 b	.0458 b	.0452 b	.0461 a
ANTI								
Y	.0430	.0530	.0465 a	.0454 a	.0450 a	.0458 a	.0497 a	.0469 a
N	.0440	.0560	.0467 a	.0459 a	.0453 a	.0462 a	.0500 a	.0475 a
SWEEP								
CP	.0440	.0720	.0472 a	.0453 bc	.0446 b	.0459 a	.0558 a	.0483 a
NO	.0440	.0460	.0465 b	.0460 ab	.0454 a	.0460 a	.0471 b	.0467 a
N2	.0440	.0460	.0462 b	.0457 abc	.0454 a	.0460 a	.0468 b	.0466 a
TEMP X ANTI								
7° Y	.0430	.0530	.0468 a	.0455 abc	.0454 abc	.0460 b	.0547 a	.0483 a
7° N	.0440	.0560	.0467 a	.0462 ab	.0457 abc	.0463 a	.0546 a	.0483 a
32° Y	.0430	.0530	.0463 a	.0454 bc	.0446 cd	.0456 c	.0448 b	.0454 a
32° N	.0440	.0560	.0467 a	.0456 abc	.0450 bcd	.0461 b	.0456 b	.0467 a
TEMP X SWEEP								
7° CP	.0440	.0720	.0483 a	.0462 a	.0459 a	.0473 a	.0695 a	.0532 a
7° NO	.0440	.0460	.0461 cd	.0458 a	.0454 a	.0455 c	.0474 b	.0459 a
7° N2	.0440	.0460	.0460 cd	.0456 a	.0453 a	.0456 c	.0471 b	.0458 a
32° CP	.0440	.0720	.0462 cd	.0445 b	.0434 b	.0446 d	.0421 c	.0435 a
32° NO	.0440	.0460	.0469 bc	.0461 a	.0455 a	.0465 b	.0468 b	.0475 a
32° N2	.0440	.0460	.0464 bcd	.0459 a	.0455 a	.0465 b	.0465 b	.0473 a
ANTI X SWEEP								
Y CP	.0430	.0690	.0473 ab	.0455 bcde	.0447 bc	.0454 b	.0561 a	.0484 a
Y NO	.0430	.0460	.0465 bcd	.0460 abcd	.0454 abc	.0464 a	.0469 b	.0464 a
Y N2	.0430	.0460	.0458 e	.0448 de	.0448 bc	.0455 b	.0463 b	.0459 a
N CP	.0440	.0750	.0471 abc	.0451 cde	.0446 bc	.0464 a	.0556 a	.0482 a
N NO	.0440	.0460	.0464 cd	.0459 abcde	.0454 abc	.0456 b	.0473 b	.0470 a
N N2	.0440	.0460	.0465 bcd	.0467 abc	.0460 ab	.0465 a	.0474 b	.0472 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations in ppm from the regression equation of the standard curve.

Table 20. Least square means of n-pentanal in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0438	.0768	.0826 a	.0722 a	.0739 b	.0770 b	.0795 b	.0793 b
32°	.0438	.0768	.0854 a	.0809 a	.0823 a	.0951 a	.0960 a	.1108 a
ANTI								
Y	.0437	.0712	.0784 a	.0755 a	.0764 a	.0817 a	.0835 b	.0929 a
N	.0440	.0823	.0896 a	.0776 a	.0798 a	.0903 a	.0920 a	.0972 a
SWEEP								
CP	.0439	.0878	.0771 a	.0758 a	.0696 c	.0684 b	.0697 b	.0607 b
NO	.0439	.0738	.0890 a	.0804 a	.0793 b	.0967 a	.0956 a	.1116 a
N2	.0438	.0687	.0859 a	.0734 a	.0853 a	.0930 a	.0980 a	.1128 a
TEMP X ANTI								
7° Y	.0437	.0712	.0772 a	.0719 a	.0718 b	.0707 bc	.0751 bc	.0780 cd
7° N	.0440	.0823	.0880 a	.0724 a	.0760 b	.0832 abc	.0838 bc	.0847 bcd
32° Y	.0437	.0712	.0795 a	.0790 a	.0810 a	.0928 abc	.0919 ab	.1120 ab
32° N	.0440	.0823	.0914 a	.0828 a	.0836 a	.0974 ab	.1002 a	.1100 abc
TEMP X SWEEP								
7° CP	.0439	.0878	.0801 a	.0815 a	.0746 c	.0751 b	.0766 bc	.0671 bc
7° NO	.0439	.0738	.0867 a	.0741 a	.0718 c	.0807 b	.0814 bc	.0887 bc
7° N2	.0438	.0687	.0810 a	.0608 a	.0751 c	.0751 b	.0804 bc	.0820 bc
32° CP	.0439	.0878	.0742 a	.0701 a	.0646 d	.0617 b	.0628 cd	.0543 c
32° NO	.0439	.0738	.0913 a	.0866 a	.0868 b	.1126 a	.1098 a	.1344 a
32° N2	.0438	.0687	.0908 a	.0860 a	.0954 a	.1108 a	.1155 a	.1436 a
ANTI X SWEEP								
Y CP	.0436	.0694	.0706 a	.0706 a	.0655 e	.0670 bc	.0684 d	.0605 b
Y NO	.0436	.0698	.0803 a	.0766 a	.0791 bcd	.0905 abc	.0885 bc	.1067 a
Y N2	.0439	.0744	.0841 a	.0791 a	.0844 abc	.0877 abc	.0936 abc	.1116 a
N CP	.0441	.0106	.0836 a	.0811 a	.0738 cd	.0698 bc	.0709 d	.0610 b
N NO	.0441	.0778	.0977 a	.0841 a	.0795 bcd	.1028 ab	.1027 ab	.1165 a
N N2	.0437	.0630	.0876 a	.0677 a	.0861 ab	.0982 ab	.1023 abc	.1140 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations in ppm from the regression equation of the standard curve.

Table 21. Least square means of n-hexanal in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0044	.0088	.0076 a	.0060 a	.0054 a	.0062 a	.0094 a	.NE
32°	.0044	.0088	.0064 a	.0042 b	.0027 a	.0022 a	.0030 b	.NE
ANTI								
Y	.0045	.0090	.0068 a	.0048 a	.0037 a	.0031 a	.0049 b	.NE
N	.0043	.0085	.0073 a	.0054 a	.0044 a	.0052 a	.0076 a	.NE
SWEEP								
CP	.0044	.0104	.0063 a	.0052 a	.0055 a	.0056 a	.0084 ab	.0767 a
NO	.0044	.0084	.0079 a	.0055 a	.0033 a	.0042 a	.0058 abc	.0068 b
N2	.0044	.0076	.0069 a	.0046 a	.0035 a	.0028 a	.0045 bc	.0041 b
TEMP X ANTI								
7° Y	.0045	.0090	.0073 a	.0062 a	.0046 a	.0049 a	.0073 b	.0060 b
7° N	.0043	.0085	.0079 a	.0058 ab	.0063 a	.0076 a	.0116 a	.NE
32° Y	.0045	.0090	.0062 a	.0034 c	.0028 a	.0014 a	.0020 c	.NE
32° N	.0043	.0085	.0067 a	.0049 b	.0026 a	.0029 a	.0035 c	.0528 a
TEMP X SWEEP								
7° CP	.0044	.0104	.0079 a	.0067 ab	.0086 ab	.0090 a	.0152 a	.NE
7° NO	.0044	.0084	.0080 a	.0062 abc	.0042 abc	.0057 a	.0081 abc	.0069 a
7° N2	.0044	.0076	.0069 a	.0051 bcd	.0035 abc	.0039 a	.0050 bcd	.0048 a
32° CP	.0044	.0104	.0047 a	.0036 de	.0023 bc	.0022 a	.0015 cd	.NE
32° NO	.0044	.0084	.0077 a	.0048 cde	.0023 bc	.0026 a	.0034 cd	.0068 a
32° N2	.0044	.0076	.0069 a	.0041 cde	.0035 abc	.0017 a	.0040 bcd	.0034 a
ANTI X SWEEP								
Y CP	.0047	.0104	.0058 a	.0043 bc	.0042 a	.0033 a	.0072 abcde	.NE
Y NO	.0047	.0088	.0080 a	.0054 abc	.0032 a	.0033 a	.0034 cde	.0040 b
Y N2	.0040	.0079	.0064 a	.0048 abc	.0038 a	.0028 a	.0040 bcde	.0034 b
N CP	.0040	.0104	.0068 a	.0060 ab	.0068 a	.0078 a	.0096 abc	.NE
N NO	.0040	.0079	.0077 a	.0056 abc	.0033 a	.0051 a	.0081 abcd	.0097 a
N N2	.0047	.0072	.0074 a	.0044 bc	.0032 a	.0028 a	.0050 cde	.0048 b

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Concentrations in ppm from the regression equation of the standard curve.

*.NE = non-estimatable.

Table 22. Least square means of n-hexanal in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	.0058	.0111	.0108 a	.0080 a	.0079 a	.0135 a	.0109 a	.0123 a
32°	.0058	.0111	.0117 a	.0100 a	.0107 a	.0132 a	.0114 a	.0146 a
ANTI								
Y	.0050	.0102	.0089 a	.0070 a	.0072 b	.0096 a	.0091 a	.0110 a
N	.0065	.0120	.0135 a	.0110 a	.0115 a	.0171 a	.0132 b	.0159 a
SWEEP								
CP	.0055	.0124	.0110 a	.0093 a	.0096 a	.0108 a	.0113 a	.0118 a
NO	.0055	.0118	.0118 a	.0097 a	.0096 a	.0145 a	.0117 a	.0144 a
N2	.0063	.0091	.0108 a	.0080 a	.0087 a	.0148 a	.0105 a	.0141 a
TEMP X ANTI								
7° Y	.0050	.0102	.0094 a	.0066 a	.0065 bc	.0083 a	.0094 a	.0095 a
7° N	.0065	.0120	.0122 a	.0094 a	.0094 bc	.0187 a	.0125 a	.0151 a
32° Y	.0050	.0102	.0085 a	.0075 a	.0078 bc	.0109 a	.0088 a	.0124 a
32° N	.0065	.0120	.0148 a	.0126 a	.0136 ab	.0156 a	.0140 a	.0168 a
TEMP X SWEEP								
7° CP	.0055	.0124	.0104 a	.0090 a	.0104 abc	.0143 a	.0159 abc	.0183 a
7° NO	.0055	.0118	.0113 a	.0073 a	.0067 bc	.0109 a	.0084 bcde	.0100 a
7° N2	.0063	.0091	.0107 a	.0070 a	.0067 bc	.0154 a	.0084 bcde	.0086 a
32° CP	.0055	.0124	.0117 a	.0091 a	.0089 abc	.0072 a	.0067 cde	.0053 a
32° NO	.0055	.0118	.0123 a	.0120 a	.0126 ab	.0181 a	.0149 abcd	.0189 a
32° N2	.0063	.0091	.0110 a	.0091 a	.0107 abc	.0143 a	.0126 abcde	.0195 a
ANTI X SWEEP								
Y CP	.0042	.0091	.0086 a	.0077 a	.0074 a	.0093 a	.0097 a	.0106 a
Y NO	.0042	.0108	.0090 a	.0062 a	.0069 a	.0096 a	.0091 a	.0092 a
Y N2	.0068	.0108	.0092 a	.0072 a	.0072 a	.0100 a	.0084 a	.0131 a
N CP	.0068	.0157	.0135 a	.0110 a	.0118 a	.0123 a	.0129 a	.0131 a
N NO	.0068	.0128	.0146 a	.0132 a	.0124 a	.0194 a	.0142 a	.0197 a
N N2	.0058	.0075	.0125 a	.0088 a	.0102 a	.0197 a	.0126 a	.0150 a

Unable to determine significant difference for week 0 and 0.1.

Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

Concentrations in ppm from the regression equation of the standard curve.

Table 23. Least square means of the peak with a retention time of 5.4 min in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	3.7	52.8	11.0 b	9.0 b	6.3 b	7.1 a	18.9 b	8.5 a
32°	3.7	52.8	44.7 a	58.8 a	43.7 a	19.0 a	32.7 a	9.9 a
ANTI								
Y	0.0	47.7	27.5 a	31.0 a	513.8 a	13.4 a	26.5 a	8.6 a
N	7.3	57.9	28.2 a	36.8 a	500.0 a	12.7 a	25.1 a	9.9 a
SWEEP								
CP	0.0	135.9	66.0 a	85.4 a	66.8 a	32.2 a	59.4 a	20.8 a
NO	0.0	13.8	10.4 b	9.8 b	7.0 b	2.8 b	8.2 b	5.1 a
N2	11.0	8.8	7.2 b	6.4 b	1.3 c	4.2 b	9.8 b	1.8 a
TEMP X ANTI								
7° Y	0.0	47.7	11.0 a	8.6 b	7.7 c	5.6 a	16.7 bc	7.5 a
7° N	7.3	57.9	11.0 a	9.3 b	5.0 c	8.6 a	21.1 abc	9.5 a
32° Y	0.0	47.7	44.0 a	53.4 a	41.7 b	21.3 a	36.3 ab	9.7 a
32° N	7.3	57.9	45.5 a	64.2 a	45.7 a	16.8 a	29.1 abc	10.2 a
TEMP X SWEEP								
7° CP	0.0	135.9	16.5 bc	11.2 b	12.5 b	18.5 b	45.2 b	17.8 a
7° NO	0.0	13.8	9.9 bcd	10.1 b	6.5 b	2.8 b	8.2 c	5.1 a
7° N2	11.0	8.8	6.6 cd	5.6 b	0.0 b	0.0 b	3.3 c	2.6 a
32° CP	0.0	135.9	115.5 a	159.6 a	121.0 a	45.9 a	73.6 a	23.7 a
32° NO	0.0	13.8	11.0 bcd	9.5 b	7.5 b	2.8 b	8.2 c	5.1 a
32° N2	11.0	8.8	7.7 bcd	7.3 b	2.5 b	8.4 b	16.4 c	1.0 a
ANTI X SWEEP								
Y CP	0.0	123.2	64.4 a	78.4 a	67.5 a	29.1 abc	58.9 a	18.6 a
Y NO	0.0	11.0	9.9 b	7.3 b	6.5 b	2.8 cd	5.4 b	3.6 a
Y N2	0.0	8.8	8.2 b	7.3 b	0.0 c	8.4 bcd	15.3 b	3.6 a
N CP	0.0	148.5	67.6 a	92.4 a	66.0 a	35.3 ab	59.9 a	22.9 a
N NO	0.0	16.5	11.0 b	12.3 b	7.5 b	2.8 cd	10.9 b	6.6 a
N N2	22.0	8.8	6.0 b	5.6 b	2.5 c	0.0 cd	4.4 b	0.0 a

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[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Peak heights relative to the peak of 1 ppm acetone - 1200 units (% full-scale deflection peak height x attenuation).

Table 24. Least square means of the peak with a retention time of 5.4 min in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	8.36	14.1	9.0 b	20.5 a	9.3 b	14.2 a	12.0 b	11.8 a
32°	8.36	14.1	40.5 a	43.3 a	39.6 a	29.2 a	34.8 a	14.1 a
ANTI								
Y	10.6	13.3	26.6 a	36.0 a	25.8 a	22.2 a	25.0 a	16.0 a
N	6.1	14.8	23.0 a	27.8 a	23.1 a	21.2 a	21.8 a	10.0 a
SWEEP								
CP	8.0	20.0	59.0 a	79.2 a	64.4 a	45.8 a	58.2 a	23.4 a
NO	8.0	12.5	8.8 a	8.2 b	5.0 a	9.2 b	6.0 b	5.2 a
N2	9.1	9.7	6.5 a	8.2 b	3.8 a	10.0 b	6.0 b	10.3 a
TEMP X ANTI								
7° Y	10.6	13.3	8.7 b	20.3 a	9.4 b	11.0 bc	11.3 b	14.1 a
7° N	6.1	14.8	9.4 b	20.7 a	9.1 b	17.3 abc	12.7 b	9.6 a
32° Y	10.6	13.3	44.4 a	51.7 a	42.1 a	33.3 ab	38.7 a	17.8 a
32° N	6.1	14.8	36.5 a	35.0 a	37.0 a	25.0 abc	31.0 a	10.3 a
TEMP X SWEEP								
7° CP	8.0	20.0	11.3 bc	48.5 abc	20.2 b	24.0 b	32.5 b	18.5 a
7° NO	8.0	12.5	8.5 bc	6.5 bc	5.0 b	8.5 b	1.0 c	2.6 a
7° N2	9.1	9.7	7.3 bc	6.5 bc	2.5 b	10.0 b	2.5 c	14.4 a
32° CP	8.0	20.0	106.8 a	110.0 ab	108.6 a	67.5 a	84.0 a	28.3 a
32° NO	8.0	12.5	9.0 bc	10.0 bc	5.0 b	10.0 b	11.0 c	7.7 a
32° N2	9.1	9.7	5.6 c	10.0 bc	5.0 b	10.0 b	9.5 c	6.2 a
ANTI X SWEEP								
Y CP	10.3	17.1	65.0 a	90.0 a	67.2 a	51.5 b	60.0 a	26.3 a
Y NO	10.3	11.4	7.3 b	9.0 b	5.0 b	7.5 b	7.0 b	5.2 a
Y N2	11.4	11.4	7.3 b	9.0 b	5.0 b	7.5 b	8.0 b	16.5 a
N CP	5.7	22.8	53.1 a	68.5 a	61.6 a	40.0 a	56.5 a	20.6 a
N NO	5.7	13.7	10.2 b	7.5 b	5.0 b	11.0 b	5.0 b	5.2 a
N N2	6.8	8.0	5.6 b	7.5 b	2.5 b	12.5 b	4.0 b	4.1 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 25. Least mean squares of the peak with a retention time of 6.0 min in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	1569.0	1281.5	614.0 a	546.7 a	491.5 a	585.0 b	813.5 b	524.1 b
32°	1569.0	1281.5	945.8 a	563.5 a	522.3 a	877.7 a	3147.7 a	3195.6 a
ANTI								
Y	1118.3	1345.7	981.6 a	558.7 a	513.8 a	719.0 a	2024.5 a	1746.3 b
N	2019.6	1217.3	578.2 a	551.6 a	500.0 a	743.7 a	1953.8 a	1973.4 a
SWEEP								
CP	1732.5	2552.0	561.3 a	481.6 a	489.3 a	973.0 ab	4572.6 a	4324.7 a
NO	1732.5	647.4	630.0 a	630.8 a	551.0 a	648.9 abc	703.0 b	666.3 b
N2	1241.9	645.2	1148.4 a	553.0 a	480.5 a	536.2 bc	666.3 b	588.5 b
TEMP X ANTI								
7° Y	1118.3	1345.7	623.7 a	529.8 a	499.0 a	568.6 a	837.8 b	405.6 b
7° N	2019.6	1217.3	604.3 a	563.7 a	484.0 a	601.4 a	789.2 b	642.6 b
32° Y	1118.3	1345.7	1339.4 a	587.6 a	528.7 a	869.5 a	3212.9 a	3087.0 a
32° N	2019.6	1217.3	552.2 a	539.5 a	516.0 a	885.9 a	3082.5 a	3304.1 a
TEMP X SWEEP								
7° CP	1732.5	2552.0	669.9 a	499.5 a	418.5 a	589.1 b	1193.6 bc	494.7 b
7° NO	1732.5	647.4	617.6 a	609.8 a	546.0 a	677.6 b	666.0 cd	605.4 b
7° N2	1241.9	645.2	554.4 a	530.9 a	510.0 a	488.3 b	581.0 cd	472.3 b
32° CP	1732.5	2552.0	452.6 a	463.7 a	560.0 a	1356.9 a	7951.0 a	8154.6 a
32° NO	1732.5	647.4	642.4 a	651.8 a	556.0 a	692.2 b	740.0 bcd	727.3 b
32° N2	1241.9	645.2	1742.4 a	575.1 a	451.0 a	584.1 b	751.6 bcd	704.8 b
ANTI X SWEEP								
Y CP	1133.0	2783.0	579.2 a	537.6 a	516.5 a	910.0 abc	4760.6 ab	4028.7 b
Y NO	1133.0	651.2	639.6 a	627.8 a	575.0 a	743.1 abc	696.5 c	684.9 c
Y N2	1089.0	602.8	1725.9 a	510.7 a	450.0 a	504.0 bc	619.1 c	525.3 c
N CP	2332.0	2321.0	543.4 a	425.6 a	462.0 a	1036.0 ab	4384.5 b	4620.6 a
N NO	2332.0	643.5	620.4 a	633.9 a	527.0 a	626.6 abc	709.6 c	647.7 c
N N2	1394.0	687.5	570.9 a	595.3 a	511.0 a	568.4 bc	713.4 c	651.8 c

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 26. Least square means of the peak with a retention time of 6.0 min in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7	467.8	686.5	703.0 a	816.0 a	584.8 b	579.8 b	781.2 b	575.9 b
32°	467.8	686.5	678.2 a	777.5 a	625.9 a	3119.7 a	2594.5 a	3133.8 a
ANTI								
Y	439.3	671.8	663.9 a	879.3 a	597.4 a	2082.7 a	1575.8 b	1710.6 a
N	496.3	701.1	717.4 a	714.2 a	613.2 a	1616.8 a	1799.8 a	1999.0 a
SWEEP								
CP	482.2	771.2	617.0 bc	1095.0 a	588.8 b	4129.5 a	3608.8 a	4051.8 a
NO	482.2	711.4	774.6 ab	689.2 a	638.3 a	763.2 b	746.8 b	828.4 b
N2	438.9	576.8	680.3 abc	606.0 a	588.8 b	656.5 b	708.0 b	684.4 b
TEMP X ANTI								
7° Y	439.3	671.8	674.2 a	775.3 a	570.6 a	497.0 a	637.7 d	552.8 b
7° N	496.3	701.1	731.9 a	856.7 a	598.9 a	662.7 a	888.7 c	599.1 b
32° Y	439.3	671.8	653.5 a	983.3 a	624.2 a	3668.3 a	2478.0 b	2868.5 a
32° N	496.3	701.1	702.9 a	571.7 a	627.5 a	2571.0 a	2711.0 ab	3399.0 a
TEMP X SWEEP								
7° CP	482.2	771.2	701.2 abc	1215.0 a	620.1 abc	529.0 b	1069.5 b	440.3 b
7° NO	482.2	711.4	745.2 abc	642.5 a	595.4 bcd	666.5 b	664.0 def	688.6 b
7° N2	438.9	576.8	662.7 abc	590.5 a	538.8 cd	544.0 b	610.0 ef	598.9 b
32° CP	482.2	771.2	532.8 bc	975.0 a	557.5 cd	7730.0 a	6148.0 a	7663.2 a
32° NO	482.2	711.4	804.0 ab	736.0 a	681.2 ab	860.0 b	829.5 cd	968.2 b
32° N2	438.9	576.8	697.8 abc	621.5 a	638.8 ab	769.0 b	806.0 cde	769.9 b
ANTI X SWEEP								
Y CP	442.3	695.4	585.3 a	1396.5 a	580.2 abc	4936.5 a	3362.5 b	3841.9 a
Y NO	442.3	681.7	739.6 a	685.5 a	646.9 ab	737.5 a	693.0 c	785.4 b
Y N2	433.3	638.4	666.7 a	583.0 a	565.1 bc	574.0 a	672.0 c	504.7 b
N CP	522.1	847.0	648.6 a	793.5 a	597.4 abc	3322.5 a	3855.0 a	4261.6 a
N NO	522.1	741.0	809.6 a	720.0 a	629.7 abc	789.0 a	800.5 c	871.4 b
N N2	444.6	515.3	693.8 a	629.0 a	612.6 abc	739.0 a	744.0 c	864.2 b

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 27. Least square means of the peak with a retention time of 10.0 min in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	35.3	25.6	25.2 a	32.2 a	30.3 b	27.8 a	25.7 a	26.3 b
32°	35.3	25.6	30.9 a	42.3 a	52.5 a	52.3 a	49.2 a	114.3 a
ANTI								
Y	38.4	26.6	30.9 a	39.5 a	42.8 a	45.5 a	40.2 a	72.0 a
N	32.3	24.7	25.2 a	35.0 a	40.1 a	34.7 a	34.7 a	61.6 a
SWEEP								
CP	38.8	31.9	31.4 a	39.5 a	60.4 a	52.2 a	47.8 a	140.1 a
NO	38.8	29.1	24.0 a	39.2 a	31.3 b	34.2 a	31.8 a	36.0 b
N2	28.5	16.0	28.8 a	33.0 a	32.6 b	33.8 a	32.8 a	34.8 b
TEMP X ANTI								
7° Y	38.4	26.6	30.9 a	35.0 a	29.0 b	24.3 a	23.3 a	26.8 b
7° N	32.3	24.7	19.6 a	29.3 a	31.6 b	31.3 a	28.0 a	25.8 b
32° Y	38.4	26.6	30.9 a	44.0 a	56.6 a	66.7 a	57.0 a	131.2 a
32° N	32.3	24.7	30.9 a	40.7 a	48.5 a	38.0 a	41.3 a	97.5 b
TEMP X SWEEP								
7° CP	38.8	31.9	26.6 a	36.0 a	50.5 b	33.5 a	32.0 a	31.9 b
7° NO	38.8	29.1	18.1 a	33.5 a	19.7 c	27.5 a	23.0 a	23.7 b
7° N2	28.5	16.0	31.1 a	27.0 a	20.7 c	22.5 a	22.0 a	23.2 b
32° CP	38.8	31.9	36.2 a	43.0 a	70.2 a	71.0 a	63.5 a	248.2 a
32° NO	38.8	29.1	29.9 a	45.0 a	42.9 b	41.0 a	40.5 a	48.4 b
32° N2	28.5	16.0	26.6 a	39.0 a	44.4 b	45.0 a	43.5 a	46.4 b
ANTI X SWEEP								
Y CP	43.3	34.2	32.2 a	41.0 a	65.6 a	74.0 a	58.5 a	159.6 ab
Y NO	43.3	34.2	28.2 a	41.5 a	34.3 bc	31.0 a	29.5 a	38.6 bc
Y N2	28.5	11.4	32.2 a	36.0 a	28.3 bc	31.5 a	32.5 a	38.6 bc
N CP	34.2	29.6	30.5 a	38.0 a	55.0 ab	30.5 a	37.0 a	120.5 abc
N NO	34.2	23.9	19.8 a	37.0 a	28.3 bc	37.5 a	34.0 a	33.5 bc
N N2	28.5	20.5	25.4 a	30.0 a	36.9 bc	36.0 a	33.0 a	30.9 bc

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 28. Least square means of the peak with a retention time of 10.0 min in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	20.0	37.4	31.2 a	44.6 a	51.2 a	26.9 a	35.6 b	33.0
32°	20.0	37.4	38.3 a	50.8 a	65.7 a	27.4 a	55.6 a	97.1
ANTI								
Y	20.5	36.3	33.9 a	45.0 a	56.5 a	19.2 a	46.1 a	67.5
N	19.4	38.5	35.6 a	50.4 a	60.3 a	35.1 a	45.0 a	62.6
SWEEP								
CP	21.4	35.2	30.0 bc	44.2 a	64.2 a	31.6 a	45.0 a	111.9
NO	21.4	29.2	39.9 ab	56.3 a	53.0 a	24.6 a	47.7 a	43.6
N2	17.0	47.8	34.4 abc	42.6 a	58.0 a	25.2 a	44.1 a	39.5
TEMP X ANTI								
7° Y	20.5	36.3	31.2 a	46.3 a	49.0 a	26.5 a	35.2 b	34.7
7° N	19.4	38.5	31.2 a	42.9 a	53.3 a	27.2 a	36.0 b	31.3
32° Y	20.5	36.3	36.7 a	43.7 a	64.0 a	11.9 a	57.0 a	100.3
32° N	19.4	38.5	40.0 a	57.9 a	67.3 a	42.9 a	54.1 a	93.8
TEMP X SWEEP								
7° CP	21.4	35.2	29.2 bc	39.2 a	56.5 a	28.6 a	37.1 bcd	33.2
7° NO	21.4	29.2	33.0 bc	58.2 a	46.0 a	26.9 a	37.1 bcd	34.7
7° N2	17.0	47.8	31.4 bc	36.4 a	51.0 a	25.2 a	32.7 cd	31.1
32° CP	21.4	35.2	30.8 bc	49.3 a	72.0 a	34.7 a	52.0 abc	190.7
32° NO	21.4	29.2	46.8 ab	54.3 a	60.0 a	22.4 a	58.3 ab	52.5
32° N2	17.0	47.8	37.4 abc	48.7 a	65.0 a	25.2 a	55.6 abc	47.9
ANTI X SWEEP								
Y CP	22.0	39.6	31.9 a	38.1 a	63.5 a	29.7 a	48.5 a	120.4
Y NO	22.0	30.8	31.6 a	58.2 a	56.0 a	16.8 a	46.3 a	42.3
Y N2	17.6	38.5	30.2 a	38.6 a	50.0 a	11.2 a	43.6 a	39.8
N CP	20.9	30.5	28.0 a	50.4 a	65.0 a	33.6 a	41.4 a	103.5
N NO	20.9	27.5	40.2 a	54.3 a	50.0 a	32.5 a	49.0 a	44.9
N N2	16.5	57.2	38.5 a	46.5 a	66.0 a	39.2 a	44.7 a	39.3

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Peak heights relative to the peak of 1 ppm acetone = 1200 units (% full-scale deflection peak height x attenuation).

Table 29. Least square means of the cooked flavor in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	1.46	3.23	2.58 a	1.79 a	1.62 a	1.58 a	1.62 a	1.29 a
32°	1.46	3.23	2.25 b	1.67 a	1.46 a	1.37 a	1.50 a	1.79 a
ANTI								
Y	1.50	3.21	2.33 a	1.79 a	1.54 a	1.42 a	1.62 a	1.58 a
N	1.42	3.25	2.50 a	1.67 a	1.54 a	1.54 a	1.50 a	1.50 a
SWEEP								
CP	1.38	3.38	2.25 b	1.56 a	1.44 a	1.19 a	1.25 a	1.62 a
NO	1.38	3.25	2.44 ab	2.00 a	1.56 a	1.56 a	1.62 a	1.44 a
N2	1.62	3.06	2.56 a	1.62 a	1.62 a	1.69 a	1.81 a	1.56 a
TEMP X ANTI								
7° Y	1.50	3.21	2.42 b	1.83 a	1.75 a	1.50 a	1.83 a	1.33 a
7° N	1.42	3.25	2.75 a	1.75 a	1.50 a	1.67 a	1.42 a	1.25 a
32° Y	1.50	3.21	2.25 b	1.75 a	1.33 a	1.33 a	1.42 a	1.83 a
32° N	1.42	3.25	2.25 b	1.58 a	1.58 a	1.42 a	1.58 a	1.75 a
TEMP X SWEEP								
7° CP	1.38	3.38	2.50 bc	1.62 a	1.62 a	1.25 a	1.38 a	1.38 a
7° NO	1.38	3.25	2.25 cd	2.12 a	1.50 a	1.62 a	1.50 a	1.12 a
7° N2	1.62	3.06	3.00 a	1.62 a	1.75 a	1.88 a	2.00 a	1.38 a
32° CP	1.38	3.38	2.00 d	1.50 a	1.25 a	1.12 a	1.12 a	1.88 a
32° NO	1.38	3.25	2.62 bc	1.88 a	1.62 a	1.50 a	1.75 a	1.75 a
32° N2	1.62	3.06	2.12 d	1.62 a	1.50 a	1.50 a	1.62 a	1.75 a
ANTI X SWEEP								
Y CP	1.25	3.25	2.12 cd	1.62 a	1.62 a	1.12 a	1.38 a	1.75 a
Y NO	1.25	3.50	2.50 abc	2.12 a	1.25 a	1.62 a	1.75 a	1.38 a
Y N2	2.00	2.88	2.38 bcd	1.62 a	1.75 a	1.50 a	1.75 a	1.62 a
N CP	1.50	3.50	2.38 bcd	1.50 a	1.25 a	1.25 a	1.12 a	1.50 a
N NO	1.50	3.00	2.38 bcd	1.88 a	1.88 a	1.50 a	1.50 a	1.50 a
N N2	1.25	3.25	2.75 ab	1.62 a	1.50 a	1.88 a	1.88 a	1.50 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination <u>at a specific week</u>, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Scores are on a 4-point scale with 1 = no off-flavor, 2 - slight, 3 = pronounced, 4 - definite.

Table 30. Least square means of the cooked flavor in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	1.04	2.88	2.21 a	2.38 a	2.00 a	2.08 a	2.04 a	1.83 a
32°	1.04	2.88	2.21 a	2.00 a	1.42 a	1.33 a	1.17 a	1.38 b
ANTI								
Y	1.08	2.75	2.08 a	2.21 a	1.62 a	1.79 a	1.50 a	1.54 a
N	1.00	3.00	2.33 a	2.17 a	1.79 a	1.62 a	1.71 a	1.67 a
SWEEP								
CP	1.00	3.00	1.88 a	1.81 bc	1.50 a	1.56 a	1.31 a	1.62 a
NO	1.00	2.62	1.88 a	2.12 abc	1.69 a	1.69 a	1.81 a	1.81 a
N2	1.12	3.00	2.88 a	2.62 ab	1.94 a	1.88 a	1.69 a	1.38 b
TEMP X ANTI								
7° Y	1.08	2.75	2.08 a	2.42 a	1.92 a	2.08 a	1.92 a	1.75 a
7° N	1.00	3.00	2.33 a	2.33 a	2.08 a	2.08 a	2.17 a	1.92 a
32° Y	1.08	2.75	2.08 a	2.00 a	1.33 b	1.50 a	1.08 a	1.33 b
32° N	1.00	3.00	2.33 a	2.00 a	1.50 b	1.17 a	1.25 a	1.42 b
TEMP X SWEEP								
7° CP	1.00	3.00	1.75 a	2.00 abc	2.00 abc	2.12 a	1.62 a	1.75 bc
7° NO	1.00	2.62	2.00 a	2.38 abc	1.75 abcd	2.25 a	2.25 a	2.25 a
7° N2	1.12	3.00	2.88 a	2.75 ab	2.25 ab	1.88 a	2.25 a	1.50 bcd
32° CP	1.00	3.00	2.00 a	1.62 bc	1.00 d	1.00 a	1.00 a	1.50 bcd
32° NO	1.00	2.62	1.75 a	1.88 abc	1.62 bc	1.12 a	1.38 a	1.38 d
32° N2	1.12	3.00	2.88 a	2.50 abc	1.62 bc	1.88 a	1.12 a	1.25 d
ANTI X SWEEP								
Y CP	1.00	2.50	1.62 a	2.00 a	1.50 ab	1.50 a	1.12 a	1.62 b
Y NO	1.00	2.50	1.62 a	2.12 a	1.38 bc	1.88 a	1.88 a	1.62 b
Y N2	1.25	3.25	3.00 a	2.50 a	2.00 ab	2.00 a	1.50 a	1.38 b
N CP	1.00	3.50	3.12 a	1.62 a	1.50 abc	1.62 a	1.50 a	1.62 b
N NO	1.00	2.75	2.12 a	2.12 a	2.00 ab	1.50 a	1.75 a	2.00 a
N N2	1.00	2.75	2.75 a	2.75 a	1.88 abc	1.75 a	1.88 a	1.38 b

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Scores are on a 4-point scale with 1 = no off-flavor, 2 - slight, 3 - pronounced, 4 - definite.

Table 31. Least square means of the stale flavor in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	1.00	1.04	1.12 a	1.62 b	2.25 a	2.33 a	2.46 b	2.04 b
32°	1.00	1.04	1.21 a	2.58 a	2.75 a	2.96 a	3.21 a	3.31 a
ANTI								
Y	1.00	1.08	1.12 a	2.04 a	2.46 a	2.75 a	2.75 a	2.64 a
N	1.00	1.00	1.21 a	2.17 a	2.54 a	2.54 a	2.91 a	2.71 a
SWEEP								
CP	1.00	1.00	1.25 a	2.88 ab	2.88 a	2.94 a	3.19 a	2.84 a
NO	1.00	1.12	1.12 a	1.62 bc	2.25 a	2.69 a	2.88 a	2.62 a
N2	1.00	1.00	1.12 a	1.81 abc	2.38 a	2.31 a	2.44 a	2.56 a
TEMP X ANTI								
7° Y	1.00	1.08	1.08 a	1.58 a	2.00 a	2.25 a	2.17 b	1.92 c
7° N	1.00	1.00	1.17 a	1.67 a	2.50 a	2.42 a	2.75 ab	2.17 ac
32° Y	1.00	1.08	1.17 a	2.50 a	2.92 a	3.25 a	3.33 a	3.38 abc
32° N	1.00	1.00	1.25 a	2.67 a	2.58 a	2.67 a	3.08 a	3.25 ab
TEMP X SWEEP								
7° CP	1.00	1.00	1.25 a	2.25 abc	2.62 a	2.75 a	2.75 bcd	2.00 ac
7° NO	1.00	1.12	1.00 a	1.12 bc	2.12 a	2.38 a	2.62 bcd	2.12 abc
7° N2	1.00	1.00	1.12 a	1.50 bc	2.00 a	1.88 a	2.00 cd	2.00 ac
32° CP	1.00	1.00	1.25 a	3.50 ab	3.12 a	3.12 a	3.62 ab	3.69 abc
32° NO	1.00	1.12	1.25 a	2.12 abc	2.38 a	3.00 a	3.12 abc	3.12 ab
32° N2	1.00	1.00	1.12 a	2.12 abc	2.75 a	2.75 a	2.88 abc	3.12 ab
ANTI X SWEEP								
Y CP	1.00	1.00	1.25 a	2.75 a	2.88 a	3.00 a	3.00 abc	2.69 a
Y NO	1.00	1.25	1.12 a	1.62 a	2.25 a	3.00 a	2.88 abc	2.88 a
Y N2	1.00	1.00	1.00 a	1.75 a	2.25 a	2.25 a	2.38 bc	2.38 a
N CP	1.00	1.00	1.25 a	3.00 a	2.88 a	2.88 a	3.38 ab	3.00 a
N NO	1.00	1.00	1.12 a	1.62 a	2.25 a	2.38 a	2.88 abc	2.38 a
N N2	1.00	1.00	1.25 a	1.88 a	2.50 a	2.38 a	2.50 bc	2.75 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination <u>at a specific week</u>, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Scores are on a 4-point scale with 1 - no off-flavor, 2 - slight, 3 = pronounced, 4 = definite.

Table 32. Least square means of the stale flavor in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	1.00	1.00	1.04 a	1.25 b	1.62 b	1.42 b	1.79 b	1.62 b
32°	1.00	1.00	1.33 a	1.79 a	2.92 a	2.67 a	3.38 a	3.50 a
ANTI								
Y	1.00	1.00	1.17 a	1.42 b	2.25 a	2.00 a	2.50 a	2.58 a
N	1.00	1.00	1.21 a	1.62 a	2.29 a	2.08 a	2.67 a	2.54 a
SWEEP								
CP	1.00	1.00	1.19 a	2.00 a	2.81 a	2.50 a	2.81 a	2.69 a
NO	1.00	1.00	1.25 a	1.31 b	2.19 a	2.19 a	2.50 a	2.44 a
N2	1.00	1.00	1.12 a	1.25 b	1.81 a	1.44 a	2.44 a	2.56 a
TEMP X ANTI								
7° Y	1.00	1.00	1.00 a	1.17 c	1.50 bc	1.42 a	1.83 b	1.50 b
7° N	1.00	1.00	1.08 a	1.33 c	1.75 abc	1.42 a	1.75 b	1.75 b
32° Y	1.00	1.00	1.33 a	1.67 b	3.00 ab	2.58 a	3.17 a	3.67 a
32° N	1.00	1.00	1.33 a	1.92 ab	2.83 abc	2.75 a	3.58 a	3.33 a
TEMP X SWEEP								
7° CP	1.00	1.00	1.12 a	1.50 bc	2.12 abc	1.50 a	2.25 ab	1.88 b
7° NO	1.00	1.00	1.00 a	1.12 cd	1.62 bc	1.62 a	1.50 b	1.50 b
7° N2	1.00	1.00	1.00 a	1.12 cd	1.12 bc	1.12 a	1.62 b	1.50 b
32° CP	1.00	1.00	1.25 a	2.50 a	3.50 ab	3.50 a	3.38 a	3.50 a
32° NO	1.00	1.00	1.50 a	1.50 bc	2.75 abc	2.75 a	3.50 a	3.38 a
32° N2	1.00	1.00	1.25 a	1.38 bcd	2.50 abc	1.75 a	3.25 a	3.62 a
ANTI X SWEEP								
Y CP	1.00	1.00	1.00 a	1.75 bc	2.75 a	1.50 bc	2.75 a	2.62 a
Y NO	1.00	1.00	1.25 a	1.12 de	2.12 a	1.62 abc	2.25 a	2.50 a
Y N2	1.00	1.00	1.25 a	1.38 cde	1.88 a	1.12 bc	2.50 a	2.62 a
N CP	1.00	1.00	1.38 a	2.25 a	2.88 a	3.50 ab	2.88 a	2.75 a
N NO	1.00	1.00	1.25 a	1.50 bcd	2.25 a	2.75 abc	2.75 a	2.38 a
N N2	1.00	1.00	1.00 a	1.12 de	1.75 a	1.75 abc	2.38 a	2.50 a

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Scores are on a 4-point scale with 1 = no off-flavor, 2 = slight, 3 = pronounced, 4 = definite.

Table 33. Least square means of the flavor scores in skim milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	4.88	4.35	4.58 a	4.29 a	4.25 a	3.88 a	3.71 a	3.83 a
32°	4.88	4.35	4.54 a	3.54 b	3.58 b	2.62 b	2.08 b	1.52 b
ANTI								
Y	5.67	4.46	4.62 a	3.88 a	4.00 a	3.17 a	2.92 a	2.77 a
N	4.08	4.25	4.50 a	3.96 a	3.83 a	3.33 a	2.88 a	2.58 a
SWEEP								
CP	4.50	4.00	4.38 a	2.94 b	3.31 b	2.75 a	2.50 a	1.97 bc
NO	4.50	4.62	4.75 a	4.38 a	4.19 a	3.44 a	2.75 a	2.94 abc
N2	5.62	4.44	4.56 a	4.44 a	4.25 a	3.56 a	3.44 a	3.12 ab
TEMP X ANTI								
7° Y	5.67	4.46	4.58 a	4.17 a	4.50 a	3.92 ab	3.92 a	4.08 a
7° N	4.08	4.25	4.58 a	4.42 a	4.00 ab	3.83 ab	3.50 a	3.58 ab
32° Y	5.67	4.46	4.67 a	3.58 b	3.50 b	2.42 bc	1.92 a	1.46 b
32° N	4.08	4.25	4.42 a	3.50 b	3.70 b	2.83 abc	2.25 a	1.58 b
TEMP X SWEEP								
7° CP	4.50	4.00	4.12 abc	3.38 c	3.62 cdef	3.25 abc	3.12 a	2.88 b
7° NO	4.50	4.62	5.50 ab	4.75 a	4.62 abc	4.25 ab	3.62 a	4.25 a
7° N2	5.60	4.44	4.12 abc	4.75 a	4.50 abcd	4.12 ab	4.38 a	4.38 a
32° CP	4.50	4.00	4.62 abc	2.50 d	3.00 def	2.25 bc	1.88 a	1.06 cd
32° NO	4.50	4.62	4.00 bc	4.00 b	3.75 bcdef	2.62 bc	1.88 a	1.62 cd
32° N2	5.60	4.44	5.00 abc	4.12 b	4.00 abcde	3.00 abc	2.50 a	1.88 cd
ANTI X SWEEP								
Y CP	5.75	4.25	4.00 a	2.88 c	3.25 cde	2.75 a	2.38 a	2.19 abcd
Y NO	5.75	4.25	4.75 a	4.00 b	4.25 abc	3.12 a	2.88 a	2.75 abcd
Y N2	5.50	4.88	5.12 a	4.75 a	4.50 abc	3.62 a	3.50 a	3.38 abc
N CP	3.25	3.75	4.75 a	3.00 c	3.38 bcde	2.75 a	2.62 a	1.75 cd
N NO	3.25	5.00	4.75 a	4.75 a	4.12 abcd	3.75 a	2.62 a	3.12 bc
N N2	5.75	4.00	4.00 a	4.12 b	4.00 abcde	3.50 a	3.38 a	2.88 bc

[*] Unable to determine significant difference for week 0 and 0.1.

[*] Within each treatment combination at a specific week, least square means that are not significantly different ($P>0.05$) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

[*] Scores are on a 7-point hedonic scale with 7 - like very much, 6 - like moderately, 5 - like slightly, 4 - neither like nor dislike, 3 - dislike slightly, 2 - dislike moderately, 1 - dislike very much.

Table 34. Least square means of the flavor scores in whole milk.

TREATMENT	Week 0	Week 0.1	Week 1	Week 2	Week 4	Week 8	Week 12	Week 16
TEMP								
7°	6.54	5.08	5.50 a	5.54 a	4.88 a	5.12 a	4.29 a	4.62 a
32°	6.54	5.08	5.00 a	4.75 a	3.04 b	3.38 a	2.33 b	1.88 b
ANTI								
Y	6.50	5.00	5.29 a	5.33 a	3.92 a	4.46 a	3.54 a	3.29 a
N	6.58	5.17	5.21 a	4.96 a	4.00 a	4.04 a	3.08 a	3.21 a
SWEEP								
CP7	6.62	4.62	5.06 a	4.75 a	3.56 bc	3.56 a	2.94 a	2.62 b
NO	6.62	5.38	5.88 a	5.44 a	3.94 abc	4.18 a	3.25 a	3.50 a
N2	6.38	5.25	4.81 a	5.25 a	4.38 ab	5.00 a	3.75 a	3.62 a
TEMP X ANTI								
7° Y	6.50	5.00	5.58 a	5.67 a	4.92 a	5.25 a	4.42 a	4.83 a
7° N	6.58	5.17	5.41 a	5.42 a	4.83 a	5.00 a	4.17 a	4.42 a
32° Y	6.50	5.00	5.00 a	5.00 a	2.92 b	3.67 a	2.67 b	1.75 b
32° N	6.58	5.17	5.00 a	4.50 a	3.17 b	3.08 a	2.00 b	2.00 b
TEMP X SWEEP								
7° CP	6.62	4.62	5.38 a	5.38 a	4.50 b	4.50 a	3.88 a	4.00 b
7° NO	6.62	5.38	6.12 a	5.88 a	4.75 ab	5.00 a	4.25 a	4.75 a
7° N2	6.38	5.25	5.00 a	5.38 a	5.38 a	5.88 a	4.75 a	5.12 a
32° CP	6.62	4.62	4.75 a	4.12 a	2.62 c	2.62 a	2.00 b	1.25 d
32° NO	6.62	5.38	5.62 a	5.00 a	3.12 c	3.38 a	2.25 b	2.25 c
32° N2	6.38	5.25	4.62 a	5.12 a	3.38 c	4.12 a	2.75 b	2.12 c
ANTI X SWEEP								
Y CP	6.50	4.75	5.12 a	4.88 a	3.50 bc	3.62 a	3.00 a	2.88 bcd
Y NO	6.50	5.50	6.12 a	5.88 a	4.00 abc	4.38 a	3.88 a	3.62 ab
Y N2	6.50	4.75	4.62 a	5.25 a	4.25 abc	5.38 a	3.75 a	3.38 abc
N CP	6.75	4.50	5.00 a	4.62 a	3.62 bc	3.50 a	2.88 a	2.38 cd
N NO	6.75	5.25	5.62 a	5.00 a	3.88 abc	4.00 a	2.62 a	3.38 abc
N N2	6.25	5.75	5.00 a	5.25 a	4.50 ab	4.62 a	3.75 a	3.88 ab

*Unable to determine significant difference for week 0 and 0.1.

*Within each treatment combination at a specific week, least square means that are not significantly different (P>0.05) are indicated by a common letter of the alphabet (a, b, c, d, e, f).

*Scores are on a 7-point hedonic scale with 7 - like very much, 6 - like moderately, 5 - like slightly, 4 neither like nor dislike, 3 - dislike slightly, 2 = dislike moderately, 1 - dislike very much.

EFFECT OF OXYGEN ON THE DEVELOPMENT OF OFF-FLAVORS IN UHT MILK

by

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The objective of this study was to investigate the role of dissolved oxygen as a contributor to flavor deterioration in sterile milk during storage. To accomplish this, an indirect UHT system was constructed in which raw skim and pasteurized-homogenized whole milk were sterilized.

A combination of nitrogen sweeping, sonication, and vacuum treatment reduced oxygen levels to less than 2 parts per million (ppm) dissolved oxygen in the preprocessed milk. Whole pasteurized and raw skim milk with normal (8 ppm) and low (2 ppm) levels of oxygen, as well as milks with and without antioxidants, were sterilized at 135°C for 5 s in the UHT system. Sterilized milk was aseptically collected in 250 ml amber colored bottles which were stoppered with either teflon-lined caps or sterile cotton plugs. Capped bottles maintained relatively low (~ 2 ppm) dissolved oxygen concentrations where as cotton plugged samples had relatively high (~ 8 ppm) oxygen concentrations. These samples were stored at 7 and 32°C for four months and subjected to the following tests: sterility, proximate analysis, antioxidant level, dissolved oxygen, volatile material, TBA, ADV, and organoleptic analysis.

Statistical analysis relating various quality tests to organoleptic properties of the sterile milks during storage yielded the following results.

Dissolved oxygen affected the rate of stale flavor development. Sterile milk in bottles with cotton plugs that had relatively high concentrations of dissolved oxygen during storage developed stale flavors sooner and more intense than milks with low levels of oxygen. However, acetaldehyde, propanal, n-pentanal, and n-hexanal (measured by GLC) that are most likely products of lipid oxidation do not appear to be principal contributors to staling in sterile milk during storage.

A decrease in the concentration of several of the volatile materials through out the storage period appears to be caused by dissipation of the volatile material thru the cotton plug or by their interaction with other compounds in the milk.

Both skim and whole milk developed a more pronounced stale flavor and at a faster rate at 32°C than at 7°C.

Several tests suggest that lipid oxidation is not the principal cause of staling in UHT sterile milk. Stale flavor development did not parallel changes in TBA values. Furthermore, the off-flavor developed faster in skim milk than it did in whole milk. Although the antioxidant did retard lipid oxidation slightly, it did not control staling.

Acid Degree Values (ADV) did increase in sterile milk stored at 32°C during the later stages of storage but changes in ADV did not parallel development of the stale flavor.

Further studies of this problem might focus on three GLC peaks that were shown to change in concentration as stale intensities increased. These peaks with retention times of 5.4, 6.0, and 10.0 min at the operating conditions employed may be flavor or flavor precursor compounds of the staling defect.